

Current Trends in Quality Science

Quality management and safety of food and non-food products

EDITOR

Maria Sielicka-Różyńska

POZNAŃ 2023

THE POZNAŃ SOCIETY FOR THE ADVANCEMENT OF ARTS AND SCIENCES

Institute of Quality Science

Poznań University of Economics and Business

Current Trends in Quality Science. Quality management and safety of food and non-food products

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DOI: 10.56091/CTQS.Qual

Cover design: Izabela Jasiczak

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Published by Wydawnictwo Poznańskiego Towarzystwa Przyjaciół Nauk

e-ISBN: 978-83-7654-536-3

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Preface

A growing demand for safe and high quality products is effecting in extensive scientific research. Several factors may influence the quality and safety of food and non-food products and the control of them seems to be of great importance. The papers included in the monograph entitled ***Current Trends in Quality Science. Quality management and safety of food and non-food products*** cover the area of managing the quality and safety of products from different perspectives. In total, the collection of thirty one papers written by the authors representing Polish and foreign academic institutions gives a new perspective on current issues in the field of Management and Quality Sciences.

In the Part I ***Quality and safety of food products*** the presented researches focus on shaping the physicochemical properties and assurance of quality in food products. Moreover, the safety issues are described basing on the analysis of RASFF (Rapid Alert System for Food and Feed) notifications. In the Part II ***Quality and safety of non-food products*** the attention is paid to evaluation and testing different substances and materials applied to design industrial products, heavy duty cleaners and cosmetic products of high quality. Part III ***Consumer perspective & quality issues*** consists of papers devoted to the consumers' behaviour and their acceptance of different products and mobile applications. The collection of manuscripts is wrapped up by the studies highlighting current quality issues and concerns.

Maria Sielicka-Różyńska

Part I

Quality and safety of food products

THE INFLUENCE OF SELECTED FACTORS ON SHAPING THE QUALITY OF THE STRAWBERRY

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DOI: 10.56091/CTQS.Qual-1

Abstract

Strawberries are one of the favourite seasonal fruits in Poland and across Europe. These fruits are valued especially for their exceptional palatability. They are characterized by low calorific value, low glycemic index and other health benefits. Strawberries owe these unique health and sensory properties to numerous bioactive compounds, such as flavonoids, vitamins and dietary fibre. The article is a review of scientific literature and the authors' research and focuses on the factors shaping the quality of strawberries. Their quality is influenced by factors such as variety, climatic, soil and cultivation conditions, as well as agrotechnical treatments. In the light of the current research results, Polish strawberries are characterized by a higher content of vitamin C, dry matter and palatability compared to strawberries from other growing regions.

Keywords: Kashubian strawberries, quality determining factors, varieties, cultivation conditions, climatic and soil factors, harvest period and conditions

Introduction

Globalisation has largely led to significant changes in consumers' lifestyles and eating behaviours. However, nowadays, an increase in demand for traditional, regional and organic food has been observed, which is mainly caused by the increase in environmental awareness of the society (Doba et al., 2019). Food hazards and safety caused by, among other things, long supply chains, have also contributed to behavioural change. In order for food to reach the consumer, it often has to cross many national and even continental borders (Gizaw, 2019). Consumers are looking for products with unique properties and remarkable health and sensory values. Such distinguished products include strawberries, which are appreciated almost all over the world for their exceptional tastiness. Strawberries are also one of the favourite fruits in Poland, and the region where, among others, strawberries are grown in Poland is the Pomeranian Voivodeship, specifically its region called Kashubia.

Geographically the area is known as Kashubian Lake District. (Drzewiecka, 2021). The cultivation of strawberries in Kashubia began in the first half of the 19th century. The unique taste and popularity of the crop resulted in a significant increase in the acreage of Kashubian strawberries plantations in the region. In 2009, the Kashubian strawberry was entered as Kaszëbskô malëna into the European system of names and designations as a Protected Geographical Indication (Commission Regulation (EC) No 1155/2009; Drzewiecka & Śmiechowska, 2016).

The aim of this article is to present the properties and benefits of strawberry fruit, including the Kashubian strawberry as a regional and traditional product. Based on the literature review and the results of authors' research, the factors influencing the quality of strawberry fruit were characterized, such as variety, climatic, soil and cultivation conditions, as well as agrotechnical treatments.

Cultivation of Kashubian strawberry

Among the many varieties of strawberries, three varieties are very popular and these are Senga Sengana, Elsanta and Honeoye (Figure 1). Each of these varieties is characterized by different properties and purpose. Honeoye and Elsanta fruits are intended for direct consumption, while Senga Sengana fruits are used mainly for processing (Drzewiecka & Śmiechowska, 2016).

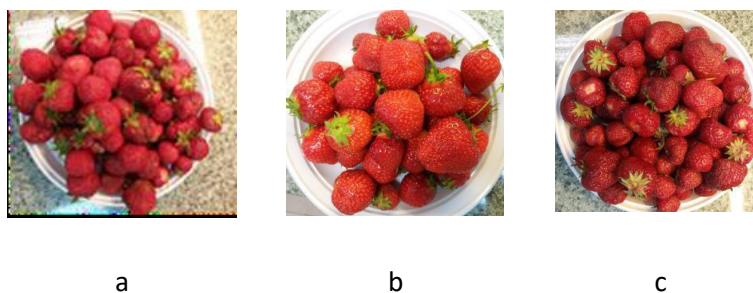


Fig. 1. Kashubian strawberry varieties: a. Honeoye, b. Elsanta, c. Senga Sengana
Source: own research.

The Honeoye strawberry is an American variety cultivated since 1979, obtained from the crossing of Holiday and Vibrant varieties. Its fruits are intended for direct consumption, with a colour ranging from intense red to dark red, uniform over the entire surface with a strong gloss. It is a very early and fertile variety (Solarska & Potocka, 2014).

The Elsanta variety is a medium-early, Dutch variety created from the crossing of Holiday and Gorella varieties cultivated since 1981. This variety is distinguished by a high content of total acids and a low content of total sugars and this differs it from other varieties of strawberries (Voća, 2007). It is the

most important variety of strawberries in Central and Northern Europe, of great commercial importance. It is characterized by excellent durability, good fruit consistency and high yield (Gössinger et al., 2009). The last, mid-late variety is Senga Sengana, otherwise known as the Favourite (Jakubiec & Nelken, 1995; Rebandel, 1982). It is a German variety, whose fruits are distinguished by their colour from intense red to dark red, aligned over the entire surface, varying in size, while the shape is broad-conical with a truncated apex, heart-shaped, heart-spherical and spherical-kidney-shaped (Gaj, 1985; Solarska & Potocka, 2014). They are characterized by aromatic, sweet, harmonized taste and smell reminiscent of wild strawberry. The fruits of this variety tolerate transportation well (Cegłowski et al., 1981).

Factors determining the quality of strawberries

The quality of strawberry fruits depends on many factors. The most important include: genetic factors (variety), climate, cultivation conditions (fertilization, soil factors, yield), harvest-related factors (harvest period, plantation lifetime), degree of maturity and post-harvesting factors (Sprogis et al., 2017) (Fig. 2).

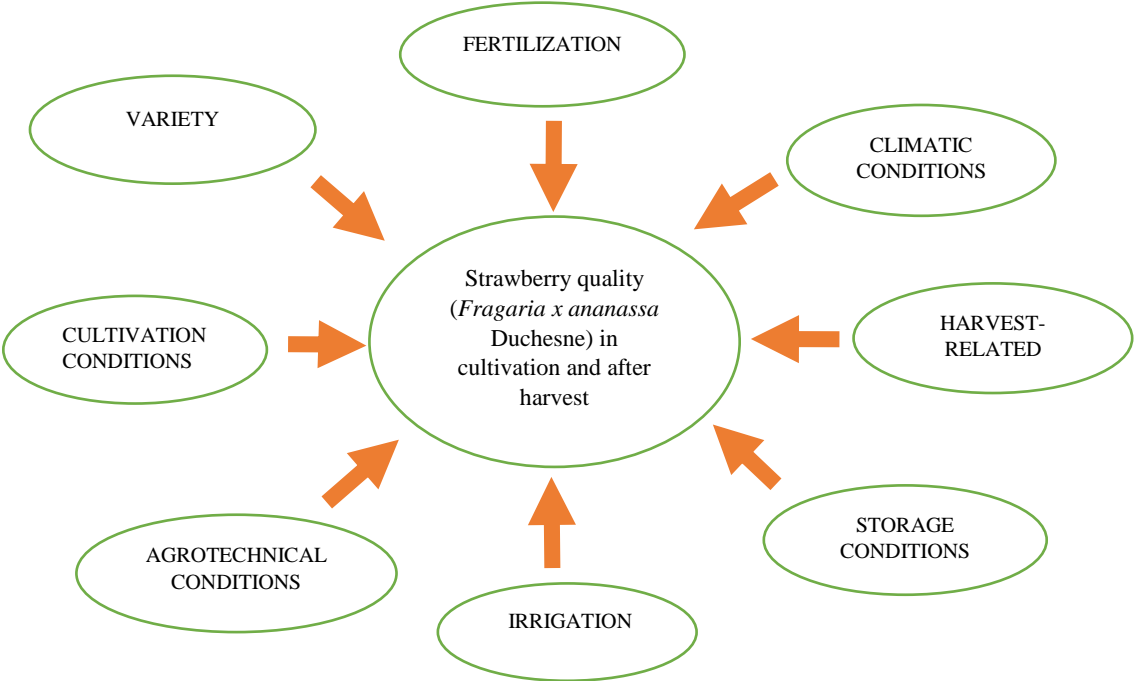


Fig. 2. Main factors affecting the quality of strawberries (*Fragaria x ananassa* Duchesne)
Source: own research.

The influence of variety on the quality of strawberry fruits

The variety and genetic characteristics have a significant impact on the quality of strawberry fruits. Each variety is characterized by specific sensory characteristics, as well as quantitative and qualitative composition. This is confirmed by studies by Aaby et al. (2012), Bojarska et al. (2011), Häkkinen & Törrönen (2000), Kopytowski et al. (2006), Skupień (2003) and Skupień & Oszmiański (2004). Research programs focus, among other things, on the selection of strawberry varieties with improved taste and higher levels of health-promoting compounds (Vandendriessche et al., 2013). Akhatou & Recamales (2014) showed that among many factors that can affect the sensory and nutritional quality of strawberry fruit, the variety is of particular importance, but the growing conditions also have an impact.

The influence of agrotechnical conditions on the quality of strawberry fruit

The content of nutrients of strawberries is influenced by growing conditions (Fan et al., 2021). Numerous studies have shown the impact of different strawberry cultivation methods on the content of bioactive substances, palatability and other quality parameters (Akhatou & Recamales, 2014; Clark & Mousavi-Avval, 2022, Laugale et al., 2014; Paszko et al., 2014; Roussos et al., 2022). Irrigation and fertilization have a decisive impact on the quality of crops and the chemical composition of strawberry fruits. The type of fertilizers used affects the yield, quality, and chemical composition of strawberry fruits, which was confirmed in research conducted by Ochmian et al. (2007), Sprogis et al. (2017) and Wysocki et al. (2012).

Research carried out in Poland and Finland shows that geographical origin influences the content of phenolic compounds in strawberries of the Senga Sengana variety (Häkkinen & Törrönen, 2000). This is also confirmed by the research of Aaba et al. (2012) on the content of phenolic compounds in strawberries grown in Norway. The researchers found that the content of all phenolic compounds was influenced by the variety. The influence of the growing region on the vitamin C content in strawberries was demonstrated by scientists from various countries (Ikegaya et al., 2021b; Munoz et al., 2023; Pukszta & Platta, 2017). Strawberries grown in Poland contained significantly more anthocyanins and vitamin C than strawberries grown in Spain (Pukszta & Platta, 2017).

The influence of strawberry harvest period and storage conditions on fruit quality

The chemical composition of strawberry fruits, primarily the content of flavonoids, phenolic acids, vitamin C, polyphenols, dry matter and anthocyanins, is influenced by the harvesting period (Hallmann et al., 2016, Voća et al., 2007). Typically, fruit is harvested three times during the growing

season and strawberries from the third harvest are characterized by the highest content of dry matter, vitamin C, polyphenolic compounds, flavonoids and phenolic acids.

Many studies have shown that the content of bioactive substances in strawberry fruits at the time of harvest depends on the degree of their ripeness (Kafkas et al., 2007; Mazur et al., 2014; Nunes et al., 2006; Pineli et al., 2011; Shin et al., 2008; Tulipani et al., 2011). However, vitamin C content declines most rapidly after harvest. Post-harvest temperature management is the most important factor in maintaining vitamin C in fruits and vegetables; losses are accelerated at higher temperatures and longer storage periods (Pavlovska et al., 2014).

The influence of the cultivation system on the quality of strawberries

The quality of strawberry fruit is also influenced by the cultivation system. The latest research shows that the crops from organic farming often contain more bioactive compounds, including antioxidants (Aninowski et al., 2020; Fijoł-Adach et al., 2016).

Organic and conventional plant cultivation form can affect the colour, quantitative and qualitative composition of strawberries (Fan et al., 2021). It is believed that strawberries from organic farming, despite lower yield, are distinguished by a higher dry matter content, higher content of bioactive substances and minerals, which makes them significantly richer in valuable ingredients for human health, than fruits from conventional cultivation (Solarska & Potocka, 2014).

Conclusions

Strawberries are a fruit with unique values. Interest in this fruit is growing not only because of its sensory value, but also because of its nutritional and health benefits (Newerli-Guz et al., 2023). The article presents the most important factors affecting the quality of strawberries, such as: variety, cultivation system, agrotechnical conditions, the impact of fertilization and irrigation, climatic and weather conditions, harvesting period, as well as storage and transport conditions. Research results indicate that Polish strawberries stand out in terms of vitamin C content, dry matter and palatability (Muñoz et al., 2023).

Prospects for further research include, among others, the impact of climate change and prolonged drought periods in strawberry-growing regions around the world. Varieties that are more resistant to long-term drought are being actively sought (Klamkowski et al., 2013). Another innovative direction of research is covering strawberry fruits with protective coatings and searching for new packaging methods that will protect this delicate and sensitive fruit during transport (Ali et al., 2022; Ikegaya et al., 2021a; Khodaei et al., 2021).

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EFFECT OF BLENDING ON STABILITY AND NUTRITIONAL QUALITY OF OILS

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DOI: 10.56091/CTQS.Qual-2

Abstract

Most vegetable oils have limited application in their original forms because of their different properties. The major problem affecting edible oils is lipid oxidation, changing their chemical, sensory and nutritional properties. One of the possible solutions to increase stability and improve quality is designing the composition of oils by blending. Blending is one of the simplest and cost-effective, physical method to create new and specific product with desired composition and characteristic.

The purpose of this study was to determine the effect of blending on physicochemical and nutritional values of selected vegetable oils (coconut, sunflower and rapeseed) during heating and accelerated storage.

Blending of rapeseed and sunflower oils with coconut oil is an efficient method to improve the nutritional value of analysed blends. Blending changed and improved the fatty acids composition. Coconut oil provides blends with medium chain fatty acids and change the proportion of saturated: monounsaturated: polyunsaturated fatty acid to 1:1.79:1.62, and 1:9.84 for n-3:n-6 essential fatty acids which is close to recommended by World Health Organization. During heating the peroxide value increased in rapeseed/sunflower oil blend over 26.8 times, whereas after coconut oil addition only 4.3 times, what suggest significant decrease of peroxide formation.

Keywords: blending, stability, vegetable oils, quality, nutritional values

Introduction

Vegetable oils are an integral part of our daily diet because of their documented positive effect on humans. They are used in almost all types of food preparation like cooking, frying or baking, but also as fresh just for dressing. The nutritional value and health benefits of vegetable oils can be attributed mainly to their specific constituents such as fatty acids composition, especially the ratio of saturated and mono- and polyunsaturated fatty acids, vitamins and natural antioxidants (Zhao et al., 2021). However, most cooking oils offer limited application in their natural state. Consumers often do not have enough knowledge on proper application and storage conditions. The same factors which are recognized as beneficial, unfortunately are also responsible for lowering stability and deterioration of oils. Composition of fatty acids in oils, mainly the degree of unsaturation affects the oxidation sensi-

tivity of the product. The polyunsaturated fatty acids, which are characteristic for vegetable and marine oils and fats, are most easily oxidized (Harvolson & Blomhoff, 2011). Additionally, Nadeem, Chen & Abdullah (2015) reported oxidation rates for stearic acid, oleic acid, linoleic acid and linolenic acid as 1:1000:1200:2500. Therefore, oils with a high proportion of unsaturated fatty acids show poor oxidation stability (Zhu et al., 2022). Lipid oxidation as a series of different reactions gives a negative impact on oils, fats and food quality. It results in loss of nutrients, reduction of product shelf-life and cause huge economic losses (Barden & Decker, 2016). When unsaturated fatty acids react with oxygen, firstly lipid peroxides are formed and then are degraded to volatile aldehydes and ketones. Volatile compounds induced the off-flavor and thus lowering the consumer acceptance of cooking oils and different food. But also influence the safety of such products (Barden and Decker, 2016). These secondary oxidation products can accumulate, leading to potential toxic and carcinogenic effects (Shahidi, 2010; Min, 2002).

Therefore, the oxidative deterioration and methods to enhance stability and nutritional quality of oils are of the great importance in oil production, marketing and consumption. Tremendous efforts have been made to reduce lipid oxidation during the processing and storage of oil (Grosshagauer, Steinschaden, & Pignitter, 2019). To enhance commercial application vegetable oils are often modified with physical or chemical process, and the four major modification methods are hydrogenation, interestrification, fractionation and blending (Lee & Wang, 2022). Unfortunately, oil modification methods have several drawbacks, and therefore their application is limited. Hydrogenation results in conversion of liquid oils into solid or semisolid more stable product. During hydrogenation *trans* isomers of fatty acids are formed, which are known to have a negative effect on human health (Skeaff & Miller, 2009). Interestrification is an alternative method to hydrogenation, in which the composition of the acids does not change, except for their arrangement in triacylglycerols structure. The advantage is that this process does not result in the production of *trans* fatty acids neither positional isomers of fatty acids. But it needs special equipment and therefore is more expensive (Senanayake & Shahidi, 2020). Fractionation is probably the oldest modification method, it is a thermo-mechanical separation process in which oils or fats are separated into fractions with different properties. Fractionation is however applicable only to several oils and fats, popular to obtain solid and semisolid fats (Kellens & Calliauw, 2013; Timms, 2005). Among these methods blending seems to be the best choice, it is a common acceptable practice in many countries, and new products are available on the market. Blending of vegetable oils and fats with different properties is one of the simplest and cost-effective methods to create new products with desired properties. Blending of oils and fats lead to changes in sensory attributes, physicochemical and nutritional properties (O'Brien, 2004). For example, Ravi, Prakash & Bhat (2005) showed that oil blending can change the

odour profiles of oils during deep fat frying. Aladedunye and Przybylski (2013) reported, that mixing vegetable oils can mostly influence fatty acids composition and give higher levels of natural oxidants and bioactive lipids in prepared blends and therefore improve the nutritional value and stability of mixed oils. However, in some cases, blending did not change the main fatty acid composition but improve the stability of blends by increasing the level of antioxidants, i.e. mixing black cumin cold pressed oil with sunflower oil and increased level of thymoquinone and tocopherols (Kiralan et al., 2016). One of the very stable against oxidation and hence not prone to peroxide formation cooking oil is a coconut oil, rich in saturated fatty acids (over 93%), of which 60% are medium chain fatty acids, especially lauric acid (Bhatnagar et al., 2009). Coconut oil could be incorporated into oils which are susceptible to oxidation, to increase their stability in prepared blends.

Therefore, the hypothesis of this work is that blending oils characterised by high saturated fatty acids content will improve stability and prolong the shelf-life of highly unsaturated oils, but at the same time will not lower their nutritional values. The objective of this study was to determine quality changes in oils (coconut, sunflower and rapeseed) and their blends during thermal processing and storage.

Materials and methods

Oil samples and reagents

The refined sunflower (SUO), rapeseed (RAO), coconut (CNrO) oils and the unrefined coconut oil (CNUO) were purchased in the local markets. The combinations of rapeseed with sunflower oil (B1) was prepared in equal proportion 50/50 (% w/w) and the coconut oil, rapeseed oil and sunflower oil (B2) in a proportion 20/40/40 (% w/w/w). To prepare blend 2 refined coconut oil was used as the one with the minimal coconut aroma. Oils and blends samples were stored in the cold condition (4°C) until the experiments were done.

Sodium methoxide, n-hexane, sodium thiosulfate, acetic acid, chloroform, potassium iodide and starch were purchased from Chempur (Piekary Śląskie, Poland) or from Merck, Sigma-Aldrich. All chemicals were of analytical grade. All standard compounds used in this study were purchased from Supelco, Sigma-Aldrich.

The Fatty acids composition

The gas chromatography-flame ionization detection (GC-FID) was used to separate and determine the fatty acids composition of the samples. Prior the separation, transesterification method was used for the preparation of fatty acid methyl esters (FAME) (PN-ISO 12966-2011). FAME were analyzed

applying the Agilent 7820A gas chromatograph, equipped with a capillary column BPX-70 (60m x 0.25mm x 0.25µm) dedicated for FAME analysis. Temperature gradient was from 140°C (5min) to 240°C (20 min), with the temperature rate 6°C. The carrier gas was helium with constant flow 0.8 ml/min. Injection port was set to 250°C, with the split ratio 50:1, and the detector was at 270°C. The identification of fatty acids methyl esters was done by comparison of retention times of peaks in a sample with those of standard pure compounds. The quantification was based on relative percentage basis.

The calculated oxidizability (CoxV) and iodine (CIV) values

The calculated oxidation values of oils and their blends was obtained by applying the equation proposed by Fatemi and Hammond (1975):

$$\text{CoxV}_1 = (\text{C18:1} + 10.3 \times \text{C18:2} + 21.6 \times \text{C18:3})/100$$

And Cosgrove, Church & Pryor (1987):

$$\text{CoxV}_2 = (0.02 \times \text{C18:1} + \text{C18:2} + 2 \times \text{C18:3})/100$$

The calculated iodine value was also calculated from fatty acid composition according to AOCS Official Method Cd 1 c-85:

$$\text{CIV} = 0.950 \times \text{C16:1} + 0.860 \times \text{C18:1} + 1.732 \times \text{C18:2} + 2.616 \times \text{C18:3} + 0.785 \times \text{C20:1} + 0.723 \times \text{C22:1}$$

where all fatty acids are expressed as a percentage of total fatty acids.

The calculated lipid quality indexes

The atherogenic index (AI) and thrombogenic index (TI) were calculated from the fatty acids composition using equations by Ulbricht and Southgate (1991):

$$\text{AI} = [\text{C12:0} + 4 \times (\text{C14:0}) + \text{C16:0}] / [\Sigma \text{MUFA} + \Sigma \text{n-3 PUFA} + \Sigma \text{n-6 PUFA}]$$

$$\text{TI} = [\text{C14:0} + \text{C16:0} + \text{C18:0}] / [0.5 \times \Sigma \text{MUFA} + 0.5 \times \Sigma \text{n-6 PUFA} + 3 \times \Sigma \text{n-3 PUFA} + (\Sigma \text{n-3 PUFA} / \Sigma \text{n-6 PUFA})]$$

where: ΣMUFA – sum of monounsaturated fatty acids excluding TFA, all fatty acids (lauric, myristic, palmitic, stearic) and sums are expressed as a percentage of total fatty acids.

Accelerated oxidation test

Shaal Oven Test

A modified Schaal oven test was conducted by placing oils and their blends in loosely sealed glass flasks in a dark. Samples were incubated at 60°C for 8 days and the peroxide value was measured.

Heating and oil sampling

The pure and blend oil samples were heated to about 160°C, in a thin layer on a kitchen pan to imitate domestic frying procedure. Samples were heated for 60 min, and collected for further analysis at intervals of 0, 10, 20, 30, 40, 50 and 60 min.

The Peroxide value

Peroxide value (PV) was determined by incubating a mixture of oil and chloroform/acetic acid (10:15, v/v) with a solution of potassium iodide in the dark for 5min (AOCS Official Method Cd 8b-90(1)). Then, water and titration indicator were added and the released iodine was titrated with sodium thiosulfate (0.01N). Obtained results were expressed as milliequivalents of active oxygen per kilogram of oil (meqO₂/kg).

Results and discussion

Fatty acids composition and nutritional parameters

Fatty acids composition of unrefined and refined coconut oils, refined rapeseed and sunflower oils and two prepared blends is given in Table 1.

The predominant fatty acids in both types of coconut oil is lauric acid (C12:0) 48.82 and 45.20% in unrefined and refined oil, respectively. Additionally, in both studied samples myristic acid (C14:0) was observed at the level 19.99 and 18.57%. The coconut oils were dominated by saturated fatty acids (Table 2), refined oil was characterized by 89.15% and unrefined by 93.90%. CNO1 and CNO2 were also deficient in MUFA (5.32 and 8.68%) and PUFA (0.78 and 2.16%).

The unsaturated fatty acids observed in analysed coconut oil samples were oleic acid (C18:1, n9) and linoleic acid (C18:2, n6). A similar profile of fatty acids composition has been reported previously (Bhatangar et al., 2009; Dorni et al., 2018; Sabahannur & Alimmudin, 2020). In opposite to CNO samples, refined rapeseed and sunflower oil were rich in unsaturated fatty acids, both mono- (65.58 and 28.92%, respectively) and polyunsaturated fatty acids (27.40 and 59.87%, respectively). On the basis of the fatty acid profile these oils are recognized as a good source of essential fatty acids, including linoleic acid (C18:2, n6) and alpha-linolenic acid (C18:3, n3). Our results are in agreement with the average of the rapeseed and sunflower oil fatty acid compositions given by the Codex Alimentarius standard for named vegetable oils (Codex Alimentarius).

Tab. 1. Fatty acids composition, Cox and CIV of oils and their blends

FA %	Oils and blends samples					
	CNuO	CnrO	RAO	SUO	B1	B2
Saturated fatty acids (SFA)						
C6:0	0.45±0.03	0.41±0.01	nd	nd	nd	nd
C8:0	6.47±0.01	6.04±0.01	nd	nd	nd	0.94±0.01
C10:0	5.52±0.02	5.16±0.02	nd	nd	nd	0.81±0.01
C12:0	48.82±0.03	45.20±0.03	nd	nd	nd	7.75±0.05
C14:0	19.99±0.06	18.57±0.04	nd	nd	nd	3.22±0.03
C16:0	9.24±0.03	10.62±0.01	4.41±0.05	6.48±0.01	5.48±0.00	6.38±0.01
C18:0	3.41±0.01	3.15±0.01	1.64±0.01	3.51±0.01	2.66±0.01*	2.67±0.01*
C20:0	nd	nd	0.76±0.01	0.24±0.00	0.63±0.00	0.43±0.01
C22:0	nd	nd	0.31±0.00	0.73±0.01	0.53±0.00	0.44±0.00
C24:0	nd	nd	nd	0.23±0.00	0.18±0.00	0.15±0.01
Monounsaturated fatty acids (MUFA)						
C18:1 n9	5.32±0.03	8.68±0.07	60.90±0.05	28.10±0.02	44.79±0.01	38.38±0.08
C18:1 n7	nd	nd	3.01±0.01	0.67±0.01	1.94±0.00	1.57±0.07
C20:1	nd	nd	1.29±0.00	0.14±0.00	0.71±0.01	0.60±0.00
C24:1	nd	nd	0.14±0.00	nd	nd	nd
Polyunsaturated fatty acids (PUFA)						
C18:2 n6	0.78±0.01	2.16±0.02	18.92±0.02	59.89±0.04	39.08±0.01	33.36±0.1
C18:3 n6	nd	nd	0.23±0.01*	nd	0.23±0.01*	nd
C18:3 n3	nd	nd	8.25±0.01	nd	3.77±0.01	3.39±0.01

All data are expressed as mean ± standard deviation. nd – not detected, CNuO – coconut oil unrefined, CnrO – coconut oil refined, RAO – rapeseed oil refined, SUO – sunflower oil refined, B1 – rapeseed + sunflower oil 50/50 (% w, w), B2 – coconut, rapeseed and sunflower oil 20/40/40 (% w/w/w). *mean values in the rows do not differ statistically significantly (Tukey test, p>0.05)

Source: own study.

As could be expected, mixing rapeseed and sunflower oil in equal proportion affect the fatty acids composition of the blend. The data indicated that the percentage of oleic acid (18:1, n9) decreased comparing to rapeseed oil, while increased comparing to sunflower oil. The percentage of linoleic acid (C18:2, n6) changed inversely. The MUFA and PUFA in blend 2 were more similar then in individual oils (Table 2). Blending improved the profile of the n-3 fatty acids, the percentage of alpha-linolenic acid (C18:3, n3) in the blend was 3.77%, whereas in sunflower oil it was not observed (Table 1). Omega 3 fatty acids have an extremely important role in normal growth and prevention of different diseases, these fatty acids also improve immune functions (Simopoulos, 2016). Additionally,

incorporating 20% of refined coconut oil into the blend changed especially the saturated fatty acids. The blend was enriched in medium chain fatty acids, caprylic acid (C8:0) 0.94%, capric acid (C10:0) 0.81% and lauric (C12:0) 7.75%, which are characteristic for CNO. The medium chain fatty acids may be considered as a potential tool in the prevention of weight gain and obesity, they are burnt for the energy rather than stored in the body (Kiyasu, Bloom, Chaikoff, 1952; St-Onge et al., 2003). The fatty acids composition itself affects dietary factors (Table 2).

Tab. 2. Nutritional parameters of oils and their blends

Fatty acids	Oils and blends samples					
	CNuO	CnrO	RAO	SUO	B1	B2
Σ SFA	93.90	89.15	7.11	11.19	9.48	22.69
Σ MCFA	61.26	56.81	-	-	-	9.50
Σ MUFA	5.32	8.68	65.58	28.92	47.44	40.55
Σ PUFA	0.78	2.16	27.40	59.89	43.07	36.75
n-6/n-3	-	-	2.29	-	10.37	9.84
Σ SFA/ Σ MUFA/ Σ PUFA*	1/0.06/0.01	1/0.1/0.02	1/9.22/3.85	1/2.58/5.35	1/5.00/4.54	1/1.79/1.62
Σ UFA/ Σ SFA	0.06	0.12	13.08	7.93	9.55	3.41
Σ PUFA/ (Σ SFA + Σ MUFA)	0.01	0.02	0.38	1.49	0.76	0.58
AI	22.64	12.00	0.05	0.07	0.06	0.35
TI	10.71	5.97	0.09	0.22	0.15	0.26

*WHO recommendation on nutritional evaluation of oils, SFA – saturated fatty acids, MCFA – medium chain fatty acids, MUFA – monounsaturated fatty acids, PUFA – polyunsaturated fatty acids, n-6 – omega-6 fatty acids, n-3 – omega-3 fatty acids, AI – atherogenic index, TI – thrombogenic index.

Source: own study.

Among the World Health Organization recommendations on nutritional evaluation of oils are the ratio of saturated, mono- and polyunsaturated fatty acids (1:1,5:1); essential fatty acids ratio (1:5–10, alpha-linolenic acid (C18:3, n-3) : linoleic acid (C18:2, n-6)); and presence of natural antioxidants. Unfortunately, none of the analysed oils samples met the requirements and had the ideal fatty acid profile. But the prepared blends with refined coconut oil had the proportion close to recommended, 1:1.79:1.62 for SFA:MUFA:PUFA and 1:9.84 for n-3:n-6 essential fatty acids. It is well known that the daily diet rich in SFA is associated with cardiovascular heart disease and high level of cholesterol in the blood (Ulbricht & Southgate, 1991). On the basis on fatty acids composition nutritional quality indices were calculated: AI – atherogenic index, TI – thrombogenic index. These indices are more useful than the fatty acids composition of the different oils. The lowest AI and TI were calculated for rapeseed and sunflower oils and their blends, whereas the highest for unrefined and refined coconut

oils (Table 2). Ulbricht and Southgate showed that AI of coconut and sunflower oil was 13.63 and 0.07, respectively, whereas their TI was 6.18 and 0.28, respectively. Introducing coconut oil into the blend (B2) affect the AI and TI indices of the obtained composition. The AI of blend B2 is 7 and 5 times higher than in RAO and SUO respectively. Additionally, the TI is almost 3 times higher than in RAO, but similar to SUO. In recent years, a negative campaign against saturated fatty acids and tropical oils from no sustainable plantations resulted in limited application in food preparation. However, its popularity is growing especially due to MCFA concentration and composition.

Oxidative stability

Oxidative stability of oil depends on their chemical composition, including natural antioxidants and fatty acids composition, especially the degree of unsaturation. Although PUFA play an important role in human health, it also makes the oil more susceptible to oxidation. Kapich et al. (2010) reported the oxidation rate for oleic acid: linoleic acid: linolenic acid as 1:22:77, the more polyunsaturated fatty acids in oils the more prone to oxidation. Based on fatty acids composition oxidation and iodine values were calculated. Because CNUO and CNrO had the highest amount of SFA and lowest amount of MUFA and PUFA, their COX values 0.01 and 0.02 (Table 3) were considerably lower than for other oils, 0.37 and 0.60, for RAO and SUO, respectively.

Tab. 3. Calculated oxidation and iodine values in oils and blends

COX/CIV	Oils and blends samples					
	CNUO	CNrO	RAO	SUO	B1	B2
Cox 1	0.01	0.02	0.37	0.60	0.48	0.41
Cox 2	0.12	0.31	4.42	6.46	5.36	4.56
CIV	5.92	11.21	111.22	128.92	119.12	101.49

Cox – calculated oxidation value, CIV – calculated iodine value

Source: own study.

Coconut oil addition let to lower the COX value in blend from 0.48 to 0.41. Similar results were observed for calculated iodine value, much lower calculated for coconut oils based on low rate of unsaturation. Schal oven test confirmed the calculated oxidation values (Figure 1). During accelerated storage the most stable oils were coconut oils, but there were significant differences among them.

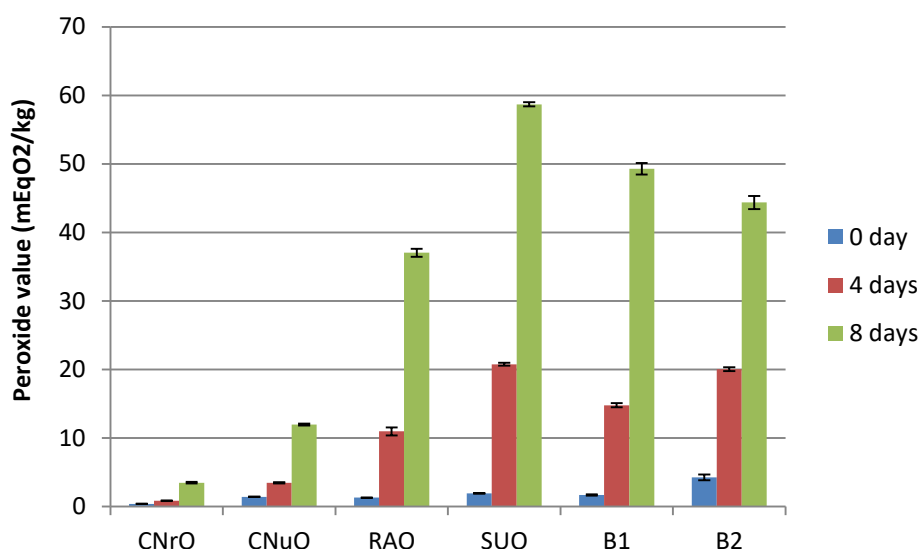


Fig. 1. Oxidative stability of oils and blends – accelerated storage

Mean values differ statistically significantly (Tukey test, $p > 0.05$)

Source: own study.

In order to evaluate the degree of oxidative degradation in oils and blends, peroxide value is commonly used (Akil et al., 2015). The refined oil samples after 12 days of storage at 60°C reached peroxide values at 5.7 mEq O₂/kg, whereas unrefined 13.9 mEq O₂/kg (data not shown). The high oxidative stability of coconut oil was also reported by Rohman et al. (2011). Rapeseed and sunflower oils and samples and their blend exceeded the 10 mEq O₂/kg after only 4 days of storage. According to Bhatnagar et al. (2009) if the PUFA content of oil is reduced through blending with MUFA, MCFA and SFA, the stability of the blend should increase. Unfortunately, incorporating more stable CNrO did not secure the blends from oxidation, after 4 days the PV exceeded 20 mEq O₂/kg. Fresh oils should have the PVs below approximately 10 mEq O₂/kg, whereas PVs over 30 mEq O₂/kg indicate that oil is spoiled and became rancid (Gordon, 2001). In our study, the rate of peroxide formation was in the order of CNrO < CNuO < RAO < B1 < SUO < B2. It suggest that probably other factors affect also the peroxide formation in samples during accelerated storage.

Oils are integral cooking medium of different food preparation. Domestic use of rapeseed and sunflower oil includes pen frying. Heating of oil at high temperature 160°C imitated domestic application and last for an hour. In all analysed samples the increase in PV was observed (Figure 2).

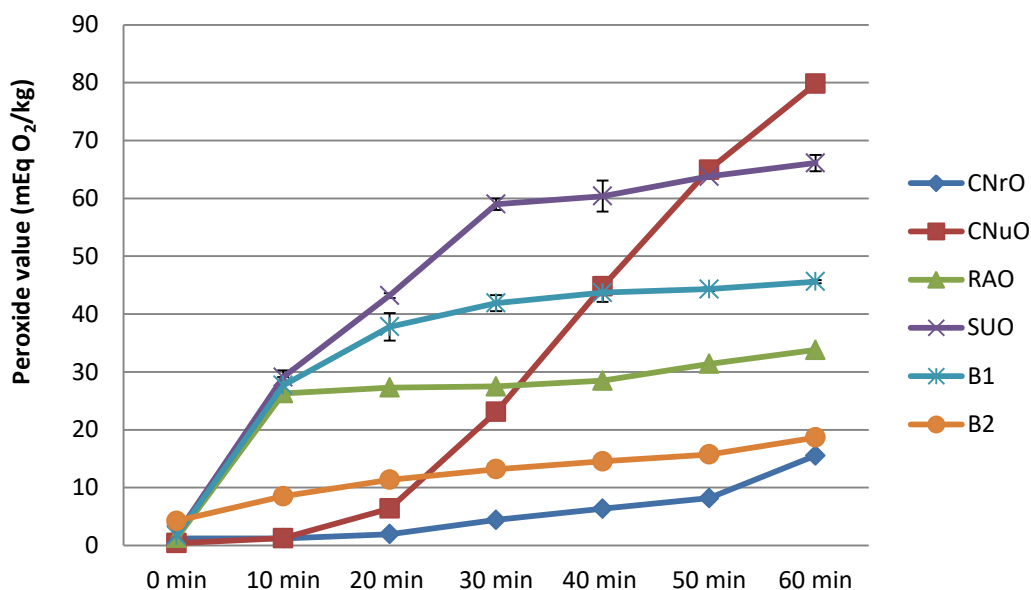


Fig. 2. Oxidative stability of oils and blends during heating 160°C

Source: own study.

Long time and continuous exposure to air, light and heat affect the peroxide formation. However, the lower PV was reported in CNrO 15.5 mEq O₂/kg and the higher surprisingly in CNuO 79.8 mEq O₂/kg. In rapeseed, sunflower and their blend samples the higher increase was observed during first 10 min of heating, from 1.3 to 26.3 mEq O₂/kg in RAO, 1.9 to 29.1 mEq O₂/kg in SUO, and 1.7 to 27.7 mEq O₂/kg in blend. But in contrast to accelerated storage test, 20% addition of refined coconut oil to blend improved significantly oxidation stability of the prepared samples. The PV increased in rapeseed/sunflower oil blend over 26.8 times, whereas after coconut oil addition only 4.3 times. Therefore, it can be concluded that during heating the presence of refined coconut oil in the oil blends was responsible for the decrease of peroxide formation.

Conclusions

Based on our results, blending of highly unsaturated vegetable oil with refined coconut oils improve the nutritional value and oxidation stability during heating. Proper choice of oils origin and proportions is important to obtain blends with a balanced composition of fatty acids, desired ratio of saturated, mono-unsaturated and polyunsaturated fatty acids, and ratio of omega-6/omega-3. It was concluded that addition of coconut oil provided medium chain fatty acids to the prepared blends, and on the other hand coconut oil was enriched with mono- and polyunsaturated fatty acids. Moreover, presence of refined coconut oil in the oil blends was responsible for the decrease of peroxide formation in the blends. Therefore, blending oils with coconut oil could be considered as an economic approach to modify chemical and nutritional properties of oils.

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THE EFFECT OF THE CULTIVATION METHODS ON THE CHEMICAL COMPOSITION OF GARLIC

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DOI: 10.56091/CTQS.Qual-3

Abstract

Knowledge of the chemical composition of vegetables and its continuous analysis is extremely important due to the constant introduction of changes that optimize the crop, primarily in terms of fertilization and protection measures used. Garlic is characterized by a rich chemical composition. It contains numerous micro- and macroelements (potassium, phosphorus, magnesium, iron, calcium, zinc, copper, nickel, cobalt, chromium, selenium, boron and germanium) and vitamins (C, A, PP, B vitamins). Garlic can be grown by several methods, differing in size and place of cultivation and method of fertilization. Taking this into account, in the submitted work, tests were carried out to verify the composition of garlic from home cultivation, allotment cultivation (with natural and mineral fertilization) and from industrial cultivation. The content of vitamins, light metals, total phosphorus and microelements was determined. The host was also interviewed as a reliable source of supplementary information. On the basis of the obtained research results, it was found that organic cultivation of garlic produces a crop with the richest chemical composition, compared to other crops, including in particular industrial cultivation. Garlic samples from organic farming were distinguished by the highest content of vitamin C, B1, potassium and phosphorus.

Keywords: garlic, cultivation, chemical composition, safe food, quality

Introduction

Common garlic (Latin: *Allium sativum*) is a perennial herb, cultivated as an annual or biennial bulb. The native region from which it comes is Central Asia. Garlic is one of the oldest cultivated species. It has been accompanying people as a vegetable, spice and medicinal plant for a long time. It is classified as a plant that reproduces vegetatively. Its chemical composition was only recognized in the 1940s, even though it was known already in Neolithic times. Garlic contains approximately 2,000 biologically active substances that have biocatalytic, nutritional, flavor and healing properties (Najda et al., 2016). This plant can be used for over 150 different health problems (Kumar et al., 2010). For

many years, there has been an increase in demand for garlic around the world, which is caused by the increasing popularity of various ethnic cuisines and the culinary search of consumers. The reason for the growing interest in garlic is also its taste and health benefits.

Garlic has various pharmacological effects due to the various biologically active chemicals it contains. It has been proven that this plant has numerous medicinal properties, including antioxidant properties (Najman et al., 2020). Allicin, present in garlic, has antifungal and antibacterial properties. Studies have shown that garlic increases the level of beneficial lipoproteins in the blood (Kumar et al., 2010). It also has anti-inflammatory properties and protects endothelial functions. Garlic inhibits cyclooxygenase activity, which reduces platelet aggregation (Ribeiro et al., 2021). Research has also shown that this vegetable has anti-cancer properties (Saastamoinen et al., 2019). Thanks to its action, the plant produces bile, thus improving intestinal function. Garlic supports the functioning of the immune system, thanks to which the body has increased defensive properties. It also helps reduce blood cholesterol levels, has anti-atherosclerotic properties, prevents thrombosis and helps balance blood pressure (Parreño et al., 2023).

Numerous varieties of garlic allow it to be grown in various climatic zones. Garlic, depending on the physiology of a given variety, thrives in the temperate and intertropical zones. In Poland, there are favorable conditions for growing winter and spring garlic. The plant roots well and produces green mass when the day is short and the temperature is low. Garlic needs a lot of light to develop properly and requires fertile soil that retains moisture well (Martins et al., 2016). Soil requirements are relatively high because the plant has a root system that is poorly developed. Garlic grows best in soil with a pH of 6.0 to 7.5 and a high humus content. Growing conditions can significantly affect the chemical composition of garlic (Sałata et al., 2020). Cultivation in selected areas can be used to manipulate the content of bioactive compounds and, consequently, to obtain the appropriate quality of the final product. Garlic cultivation is demanding in terms of nutrient demand, therefore an appropriate fertilization regime must be maintained. The quality and chemical composition of the crop is also influenced by the date of harvest and, later, the method of storage (Etana et al. 2018). Proper irrigation also has a significant impact on achieving maximum yield potential.

When considering soil fertilization for growing garlic, it should be noted that it has a low ability to use nutrients present in the soil. This is due to its shallow and underdeveloped root system. This feature of garlic requires enriching the soil intended for cultivation with nutrients (Pączka et al., 2021). Garlic cultivation uses natural fertilization, which improves the biological, physical and chemical properties of the soil (Khade et al., 2017) and is most often used on organic farms. Natural fertilizers used in garlic cultivation are: manure, straw, compost and green manures produced as a result of plowing

selected plant species. In addition to natural fertilization, mineral fertilization is also used to supplement the plant's nutritional requirements. In integrated garlic cultivation, they are used to provide mineral nutrients to the soil (Nguyen et al., 2022). Mineral fertilization includes fertilization with: potassium, nitrogen, magnesium, phosphorus, microelements and liming. Both the type of garlic fertilization used and the scale of cultivation provide the basis for dividing the methods of its cultivation into: home cultivation (in pots), plot cultivation with natural or mineral fertilization and industrial – using chemical protection products in permissible doses. However, according to literature data and practice, the division of garlic cultivation methods is as follows: cultivation from cloves, cultivation from air bulbs, accelerated cultivation using foil tunnels, as well as autumn and spring cultivation (Ko et al., 2023).

The main aim of this work was to initially verify the chemical composition of garlic obtained from home cultivation, allotment cultivation with natural fertilization. and artificial and industrial (in this case – field) and comparison of the results obtained. Garlic was obtained from own home cultivation and own allotment cultivation (with natural and mineral fertilization) – spring garlic, Jarus variety. Samples from the so-called industrial cultivation was obtained from the producer – winter garlic, Harnaś variety. Garlic samples collected from each type of crop were analyzed for the content of macro- and microelements, including: sodium, potassium, calcium, magnesium, phosphorus, iron, zinc, copper, manganese, selenium and iodine, and vitamin C (ascorbic acid), B1 (thiamine). Moreover, in order to deepen the information about the specificity of garlic cultivation using selected methods, an interview was conducted with the farmer. The topics discussed in the interview concerned among others: soil preparation for cultivation, types of fertilizers, methods of combating weeds.

Materials and methods

Garlic cultivation

Garlic was grown using home and allotment methods in the Dopiewo commune (Poznań district), where brown soil occurs (Fig. 1 a, b). The plot was cultivated using natural fertilizer in the form of fresh cattle manure, delivered from a farmer and also purchased in granulated form (“Doktor O'grodnik – granulated cattle manure natural fertilizer”, Fertigo, Suchy Las, Poland) – in organic cultivation. In allotment cultivation, separate mineral fertilization was also used – a preparation (“Florovit BIO fertilizer for vegetables and herbs”, Grupa INCO S.A., Góra Kalwaria, Poland) with the following composition: nitrogen (2%), phosphorus (0.3%), potassium (5.5%), calcium (0.6%), organic matter. In the case of garlic grown with artificial fertilization, an ecological plant protection product was oc-

asionally used (“Agrocover Spray”, Target, Kartoszyn, Poland), the active substance of which was saffron oil.

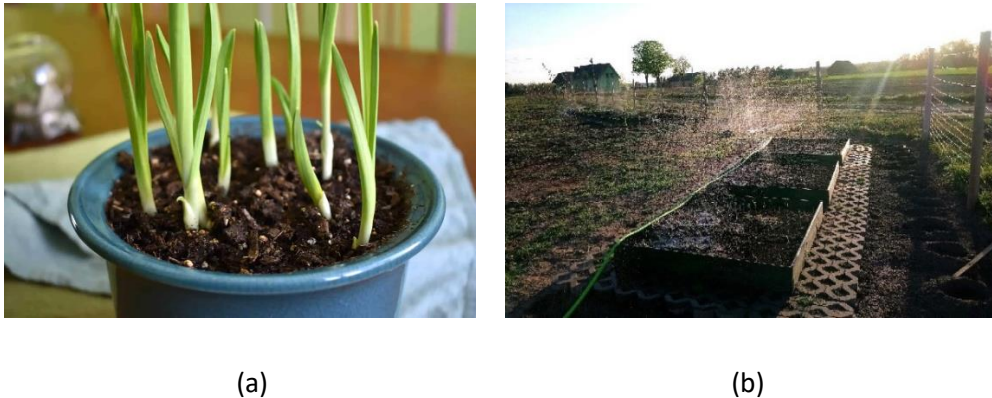


Fig. 1. View of (a) home cultivation and (b) the beds in allotment cultivation – after planting, original material

Source: own research.

Garlic cultivation in the plot was started in March 2023. Garlic was harvested at the turn of August and September of the same year. Before planting, the soil was loosened and enriched with natural fertilizer (organic beds; fresh manure) and mineral fertilizer (first batch before planting). Subsequent batches of mineral fertilizer were applied according to the manufacturer's recommendations. In the case of beds with mineral fertilization, granulated manure was used in the following months of cultivation. Basically, the garlic was 'fed' once a month until June, trying to choose cloudy but windless days for this activity. Spring garlic of the Jarus variety was purchased for planting, coming from a reliable source. Healthy, large and undamaged heads were selected. The heads, divided into cloves, were placed directly into a depth of about 5 cm, with the heel down, then covered with soil and pressed lightly. On average, the distance between plants in a row was 10-15 cm. After planting, the garlic was watered regularly (dripwise and traditionally), which is important during the germination period. Other maintenance procedures, such as weeding, were performed manually and often due to the rapid overgrowth of free spaces. The procedure was identical in both types of fertilization. The effects of garlic cultivation with mineral fertilization (end of May) are shown in Fig. 2. Morphological differences of plants growing on soils fertilized with natural and mineral fertilizers were not observable.



Fig. 2. Effects of garlic cultivation with natural fertilization, original material

Source: own research.

In order to analyze the chemical composition of garlic from industrial cultivation, garlic from a well-known producer (from field cultivation), the autumn-winter variety Harnaś, was used. It is an early variety, wintering in the ground, with a large number of cloves (8–13). The weight of the head of this variety is significant and amounts to 60–80 g, the scale on the cloves is purple and closely adheres to the cloves, while the scale on the head is gray. The Harnaś variety produces high yields, harvesting takes place already in July, and is recommended for direct consumption and for use in the processing and pharmaceutical industries. For comparison, garlic bulbs planted in spring are smaller and do not produce inflorescence shoots, which was confirmed by the yields of home cultivation (see Fig. 3). Garlic bulbs from naturally fertilized beds and artificial, did not differ in size. The yield from all beds was also comparable.



(a)

(b)

Fig. 3. Garlic harvest from allotment cultivation (September 2023), (a) natural fertilization and (b) mineral fertilization, original material

Source: own research.

Home cultivation began at the beginning of April 2023. A 20 cm deep pot was filled with soil, leaving approximately 1 cm from the upper rim of the pot. Garlic cloves (Jarus variety) were placed in the pot at a depth of 10 cm, with the sharpened end upwards. The pot with planted garlic was placed next to the window in the sunniest place. The garlic was watered in moderation to avoid rotting the planted cloves. The cultivation lasted approximately 7 months. When the garlic leaves turned brown, the garlic was dug out.

Analysis of the chemical composition of garlic

Determination of vitamin C (ascorbic acid) content

The analysis was performed using the Tillmans titration method, which is based on the reducing properties of L-ascorbic acid in relation to 2,6-dichlorophenolindophenol, DCIP (Tillmans reagent) – a dye that also serves as an indicator, discoloring after reduction, according to the standard PN-Standard A-04019:1998. In the first stage, the determination of vitamin C involved its extraction with oxalic acid from the sample and then the oxidation of ascorbic acid to dehydroascorbic acid in an acidic environment using the standard blue DCIP dye. The blue dye used during the determination turns pink in an acidic environment in the oxidized form, while in the reduced form it is colorless. The titration of the DCIP solution was made using the titration solution, which was sodium thiosulfate.

Determination of vitamin B1 (thiamine) content

The content of vitamin B1 in garlic samples was determined using the thiochrome method high-performance liquid chromatography (HPLC), after prior extraction of thiamine using enzymatic and acid hydrolysis. Thiamine contained in the extracts was converted into a thio-chromium derivative using 1% potassium ferricyanide and immediately quantitatively determined by chromatography using a fluorescence detector.

Determination of light metals (Na, K, Ca, Mg)

In the determination of light metals (alkali metals and alkaline earth metals), including sodium, potassium, calcium and magnesium, the Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) technique was used by performing analysis using a spectrometer: JY 2000 2 ICP-OES Spectrometer, Hitachi, Tokyo, Japan, according to a specific standard (PN-EN ISO 11885:2009). In the ICP-OES technique, high-frequency radio waves are used for atomization and excitation, which enable the creation of a high-temperature plasma (approx. 7000 K). This allows for disintegration of chemical compounds into atoms, which are excited, and then emit the absorbed energy in the form of electromagnetic radiation, characteristic of a given element. The analysis included preparation

(mineralization) of samples, preparation of the apparatus for operation and its calibration, and measurement of the sodium, potassium and magnesium content in the prepared (mineralized) garlic samples.

Determination of total phosphorus (P)

The UV-VIS spectrophotometry method was used with the addition of ammonium vanadomolybdate as a coloring reagent, after prior ashing of the sample according to the MCMiAŻ/PB-06 research procedure, used in food products, except for meat and meat products, as well as oils and fats. Spectrometry is an analytical technique that uses the phenomenon of absorption of light waves in a medium of different density and structure, in accordance with the Lambert-Beer law. After the beam passes through the tested substance, the obtained UV-VIS spectrum, compared with the standard curve, allows us to determine both the presence and concentration of a given substance in the sample. The analysis was performed using a UV-VIS SP8001 spectrophotometer from Meter-tech, Taipei, Taiwan.

Determination of microelement content (Fe, Cu, Zn, Mn, J, Se)

To determine the content of microelements in garlic samples, the Atomic Absorption Spectrometry (ASA or AAS) technique was used, which involves measuring the absorption of light energy by free atoms of elements located in the measurement space. The whole process, as mentioned in the previous point, is based on the practical application of the Lambert-Beer law in mathematical calculations of measurement. The result of the analysis is quantitative – the amount of light absorbed is directly proportional to the concentration of a given element in the sample.

In the absorption spectrometer used in the study (ASA Spectrometer by PG Instruments, Lutworth, Great Britain), model AA500, flame atomization – a nebulizer was used to produce an aerosol (Flame Atomic Absorption Spectrometry, FAAS). The analyzed material is directed towards the burner. There it is mixed with a flammable gas (usually acetylene and air or acetylene and nitrous oxide) and burned in a flame. The actual atomization takes place in the flame due to high temperature.

Interview with a farmer

An interview is a research method based on a focused conversation involving at least two people. The aim of the interview conducted in this study was to supplement information on the methods of cultivating (planting, fertilizing, caring for), harvesting and storing garlic. The thematic conversation was conducted directly with the farmer. The article presents selected questions and answers from an interview (conducted with the same farmer).

Results and discussion

Comparison of the content of selected vitamins in tested garlic samples

The content of vitamin C in garlic from different types of crops varied (see Table 1). The highest amount of vitamin C was recorded in the garlic sample from organic farming. As shown by the data presented in the above chart (Fig. 4), the home-grown garlic sample contained the least amount of vitamin. The results are generally similar to the data presented by other authors (Borlinghaus et al., 2014). Based on the obtained results (Table 1), it was found that garlic from organic gardening has the highest vitamin B1 content, while home-grown garlic had the lowest vitamin B1 content.

Tab. 1. Content of macro- and microelements in samples of garlic cultivated using various methods

Type of vitamin	Unit mg/100 g	Home cultivation (\pm MU)	Ecological – natural fertilizers (\pm MU)	Mineral fertilizers (\pm MU)	Industrial cultivation (\pm MU)
Vitamin C	mg	18.0 \pm 0.028	42.0 \pm 0.065	29.0 \pm 0.045	24.0 \pm 0.037
Vitamin B1	mg	0.14 \pm 0.0002	0.19 \pm 0.0003	0.16 \pm 0.0002	0.15 \pm 0.0002

MU – measurement uncertainty

Source: own research.

Comparison of the content of macro- and microelements in tested garlic samples

The results presented in Table 2 show that the highest content of sodium, potassium, calcium, magnesium, phosphorus, iron, zinc, manganese, iodine and selenium characterized samples from organic farming, while the lowest – in most cases – samples from home cultivation. In many cases, samples fertilized with minerals and those originating from industrial cultivation had similar contents of the analyzed elements. In the case of copper, the lowest amount was recorded in home-grown garlic. Garlic samples from other crops contained a similar amount of copper, but, interestingly, the greatest amount of this microelement was found in industrially grown garlic. The obtained results are consistent with literature data (Mardomi, 2017; Yusuf et al., 2018). The results obtained by Yusuf et al. (2018) indicated high values in K, Ca, P and Fe compared to magnesium Mg, Zn and Mn which were found in trace forms. Garlic contained 10.19, 26.30, 10.19, 5.29, 0.001, 0.34 and 0.001 mg/100g of potassium, calcium, phosphorus, iron, magnesium, zinc and manganese respectively while lead and cobalt were below detection level. Low ash is an indication of low inorganic mineral content but garlic contained appreciable amounts of mineral elements which makes garlic a potential regulator of blood pressure, fluid balance, anti-hypertension, anti-cardiac arrhythmias, anti-ischemic heart disease, anti-atherogenesis, anti-sudden cardiac death and anti-diabetic.

The obtained results show that the type of method of growing plants, including garlic, affects their chemical composition – the content of macro- and microelements, as well as vitamins. The poorest composition of home-grown garlic is probably due to the quality of the soil in which the plant grew, including the availability of nutrients (Bloem et al., 2010). Fertilizing the soil with natural fertilizers is the most effective. This method enriches the soil with easily assimilable forms of mineral substances that determine plant metabolism. Plants, as living organisms, are capable of growth, development and – most importantly from the producer's point of view – high yields, thanks to the constant supply of matter and energy that enable them to perform basic life functions.

Tab. 2. Content of macro- and microelements in samples of garlic cultivated using various methods

Type of element	Unit mg/100 g	Home cultivation (±MU)	Ecological – natural fertilizers (±MU)	Mineral fertilizers (±MU)	Industrial cultivation (±MU)
Na	mg	13.0±0.035	19.0±0.05	14.0±0.04	16.0±0.04
K	mg	351.0±0.932	423.0±1.12	361.0±0.96	369.0±0.98
Ca	mg	38.0±0.101	44.0±0.12	36.0±0.10	42.0±0.11
Mg	mg	21.0±0.056	26.0±0.07	23.0±0.06	22.0±0.06
P	mg	131.0±0.348	158.0±0.42	149.0±0.40	151.0±0.40
Fe	mg	1.10±0.003	1.80±0.00	1.40±0.00	1.60±0.00
Cu	mg	0.18±0.000	0.24±0.00	0.25±0.00	0.28±0.00
Zn	mg	0.80±0.002	1.10±0.00	0.90±0.00	0.90±0.00
Mn	mg	0.32±0.001	0.49±0.00	0.41±0.00	0.38±0.00
J	µg	1.90±0.005	2.60±0.01	2.10±0.01	2.30±0.01
Se	µg	9.20±0.024	13.60±0.04	10.10±0.03	11.20±0.03

MU – measurement uncertainty

Source: own research.

Interview with the farmer – selected questions and answers

Below is the author's interview questionnaire along with the answers provided by the farmer.

1. How long have you been growing garlic?

I have been growing garlic for about 10 years.

2. What variety of garlic do you most often choose for planting?

I most often choose the Harnaś variety for planting.

3. Why is this variety chosen most often by you?

The Harnaś variety produces large heads with quite a lot of teeth. Personally, I think that this variety is not difficult to grow and the yield is always satisfactory.

4. Do you use organic fertilization?

Yes, I use it.

5. What organic fertilizer do you most often use to fertilize the soil for cultivation? Please justify your answer.

I fertilize the soil with manure. My neighbor has cows, so I don't have a problem with obtaining fertilizer. I have been using manure since I started growing garlic and the yield has always been good, so I will continue to do so.

6. Do you use mineral fertilization in your crops?

Yes, I use it.

7. Why do you think it is worth using mineral fertilization?

Of course, it is worth using such fertilization, but only in appropriate amounts. Such fertilization will help enrich the soil, which will result in better yields.

8. How do you prepare the soil for growing garlic?

I loosen the soil myself to a depth of approx. 5–7 cm. Then I remove larger stones and remaining plants. The last step is to level the soil surface. I don't loosen the soil when it's too wet.

9. Do you use soil mulching? Please justify your answer.

I don't use it. I've heard that it reduces weeds, but so far I've managed without mulching. My crop is small, so I pull out weeds by hand on a regular basis.

10. What is your position on the use of plant protection products? Please justify your answer.

I believe they are necessary, but in reasonable, permitted amounts. Sometimes I'm forced to use them. Most often, I ask agricultural advisors what preparations to use and in what quantities. There are many ecological protection products on the market now, and I try to use them most often.

11. How do you irrigate the crop? Please justify your answer.

When there is little rain, I water the crops using a stationary sprinkler. Sometimes it happens that I water the garlic with a watering can. I have time and willingness for this.

12. How do you deal with garlic diseases?

When I notice that the garlic is attacked by a disease, I use appropriate protection measures recommended by the agricultural advisor, and whenever possible, I try to choose ecological protection measures.

13. Do you follow the principle of taking turns?

Yes. Only after 3–4 years do I plant garlic in the same place. This protects against garlic diseases, pests that attack it and weeds.

14. What does your garlic harvest look like?

I always choose a day when it doesn't rain. I place the dug garlic in a shaded place for drying.

15. How do you store garlic?

Garlic is stored evenly distributed in plastic boxes in a "cell" in the garden.

16. Has your farming method changed over time and why?

Over the years I have had to use more protective measures as illnesses occur more frequently. When it comes to cultivation, it always looks the same for me.

According to the interview, the farmer uses the efficient Harnaś variety when growing garlic. He considers natural soil fertilization using cattle manure as basic. The soil is additionally enriched with mineral fertilizers (in limited quantities). The farmer has noticed that over the years the need to use plant protection products has increased, but he only uses them when necessary. The most frequently used and available one, as confirmed by the above interview and literature data, is cattle manure. Fresh manure consists of excrements of farm animals – cows, horses, pigs and poultry, as well as straw, which is their bedding. Fresh manure has high nitrogen content in the ammonium form. Additionally, it contains calcium, magnesium, boron and iron. Manure, like compost, effectively improves the quality of the soil and enriches it with micro and macro elements, as well as microorganisms that decompose organic matter from manure and create valuable humus from it. The main advantage of manure is that it decomposes slowly. Thanks to this, it supplies the soil with nutrients (Eleduma et al., 2020). Their full use takes place within three or four years. In addition, manure affects the structure of the soil – it improves permeability and the ability to store water. However, fresh manure is quite problematic when it comes to its use in practice. Due to its unpleasant smell and quantity, it is difficult to store, especially on small farms. It is also not easy to use, because it must be spread and then dug in with the top layer of soil. Otherwise it loses a lot of nitrogen. Manure in the form of

granules is often an alternative for small farms – it is convenient to use. It has a neutral pH, making it a universal product. It does not emit any unpleasant odor during fertilization.

The intensification of agriculture is always associated with an increase in the use of fertilizers minerals and increasing their pollution in the aquatic environment ingredients. The hallmark of sustainable agriculture is striving to reduce threats to the natural environment, and the development of agriculture must take into account environmental protection requirements (Constantin et al., 2010). Natural fertilization gives the best results in terms of chemical composition, but it will not match the yields resulting from intensive mineral fertilization. Combining, as the farmer noted in the interview, two types of fertilization: natural and mineral, gives optimal results. Taking into account the guidelines of the European Biodiversity Strategy (2020), the challenge for modern horticulture/agriculture is to maintain plant production at a high level. It is worth mentioning that scientists and producers of modern, effective and environmentally friendly biopreparations are currently meeting new challenges (Kubiak et al., 2022, 2023).

Conclusions

1. The tested garlic samples, cultivated using various methods, were characterized by a high content of vitamin C. Organic plot cultivation had the highest content of vitamin C and B1. The results obtained by analyzing the vitamin B1 content in garlic samples from different cultivation methods were similar.
2. Of all the light metals determined, the highest amount of potassium was recorded in 'eco' garlic samples. Analysis of the chemical composition of garlic from various crops also showed a high phosphorus content, especially in the sample from organic farming. The tested garlic samples were characterized by a low content of microelements.
3. In the conducted research, it was observed that garlic from organic plot cultivation has a richer chemical composition, compared to garlic obtained from other cultivation methods used in the research. As a result of the experiment, it was also found that garlic samples from home cultivation have the poorest chemical composition compared to the others.
4. Taking into account the above conclusions and the fact that the chemical composition of cultivation with mineral fertilization and industrial cultivation is very similar, the type of variety used in the experiment (Harnaś – industrial cultivation; Jarus – other crops) is insignificant.
5. The interview confirmed that natural fertilizer in the form of manure works well when growing garlic and that the principle of rotation is used even when cultivating on a very small scale. Ecological plant protection products work slower than chemical ones, but in most cases equally effectively, which is important information from the point of view of food quality and safety.

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BIOLOGICAL ACTIVITY OF NATURAL HOMEMADE MIXTURES

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DOI: 10.56091/CTQS.Qual-4

Abstract

Nowadays, an increasing trend to benefit from traditional medicine together with seeking out natural remedies for infections is observed. Despite various recipes for natural antimicrobial mixtures, there is a lack of studies unequivocally confirming these declarations, as the literature data focuses on the individual components' properties. The most popular recipes use herbs, fruits, and vegetables, among others, garlic, onion, turmeric, or lemon. Meanwhile, the composition of the mixture may be crucial for its biological activity.

The studies aimed to evaluate the antimicrobial activity of 14 self-prepared mixtures based on recipes from websites and social media with indications of antibacterial, antifungal, and antiviral properties. The antimicrobial activity was determined by the well-diffusion assay towards Gram-positive and Gram-negative bacteria: *Micrococcus luteus*, *Staphylococcus aureus*, *Bacillus subtilis*, *Enterococcus faecalis*, *Listeria monocytogenes*, *Escherichia coli*, *Pseudomonas aeruginosa*, *Salmonella enterica* ser. Enteritidis, *Proteus vulgaris*, and fungi: *Candida albicans* and *Rhodotorula mucilaginosa*. The effect of the individual components on the antimicrobial properties of the mixture was also tested on the example of two mixes with the strongest activity.

The majority of the tested mixtures exhibited antimicrobial activity against indicator microorganisms. The strongest antimicrobial effect was observed by *Natural Antibiotics 1-4* (samples 7–10), inhibiting the growth of all indicator microorganisms tested. However, the properties of the tested mixtures depended strongly on their composition as well as on the sensitivity of the microorganisms used in the research.

Keywords: antimicrobial activity, biological activity, homemade remedies, functional foods, medicinal plants, traditional medicine

Introduction

In recent years, an increasing trend among consumers to reach for natural ways to prevent and treat infections together with a growing interest in traditional medicine has been observed. Worldwide there are many different remedies recipes available, that, according to the authors' statements, may inhibit the growth of bacteria and fungi or inactivate viruses. In general, such homemade remedies are mostly in the form of different mixtures including beverages, infusions, shots, blends, pastes,

filtrates, and shakes. Primarily, homemade natural mixtures are prepared from different morphological plant parts including fruits, leaves, stems, twigs, roots, bark, flowers, and buds. Such natural remedies in the form of mixtures are mostly minimally processed products. The abundance of natural bioactive compounds such as phenolic acids, flavonoids, capsaicinoids, carotenoids, coumarins, alkaloids, polyacetylenes, saponins, terpenoids, vitamins and minerals, and many others determines their high biological activity (Chandrasekara & Shahidi, 2018). Plant-based foods rich in secondary metabolites pose valuable biological properties including antioxidant, antiaging, antibacterial, antifungal, antiviral, anti-inflammatory, and many others. However, no studies unequivocally confirm specific properties regarding varied complex compositions of natural remedies. In the literature, studies focus on individual components' biological activity and properties, including the most popular used for natural remedies: garlic, onion (Corzo-Martínez et al., 2007), turmeric (Verma et al., 2018), and lemon (Mohanapriya et.al., 2013). The studies do not confirm specific sophisticated mixtures properties, furthermore, the interaction between ingredients should also be explored. Meanwhile, researchers indicate that the composition of the mixture may be crucial for its antimicrobial impact.

The presented study aimed to evaluate the antimicrobial properties of 14 self-prepared mixtures based on recipes found in various portals or social media and indicated as mixtures with antibacterial, antifungal, and even antiviral properties, towards 11 different indicator microorganisms. Furthermore, two mixes with the highest activity were used as examples to assess how each component affected the mixture's antibacterial qualities. The samples included mixtures: 'for the cold' (samples 1 and 6), *Anti-flu mixtures* (samples 2-5), *Natural antibiotics 1–4* (samples 7–10), together with *White infusions* (samples 11–12), and *Golden milks* (samples 13–14), which had within the group similar compositions or claimed health-promoting properties.

Materials and methods

Materials

Natural mixtures preparation

The tested mixtures were prepared based on the recipes from both websites and social media profiles. The samples differed in composition and health-promoting properties (including antibacterial and antifungal activity) depending on the declaration made by the author's recipe (Tab. 1). Ingredients including among others: dried spices, fresh fruits and vegetables, and honey, used for the studies were purchased from a popular retail chain store in Poland. Samples were prepared under conditions that simulated domestic settings. Lemon, grapefruit, and orange juices were pressed freshly before preparing the samples.

Tab. 1. Composition and preparation description of the tested mixtures

No.	Name of the mixtures	Ingredient	Amount of ingredient	Mixtures preparation
1	Firecracker for the cold	Star anise	3.5 pieces	All the ingredients, except honey, were poured over boiling water and steamed covered until cooled, next honey was added (Rzezińska, 2022)
		Cloves	1 teaspoon	
		Grated ginger root	20 g	
		Ground turmeric	0.5 teaspoon	
		Lemon juice	Half a lemon	
		Honey	1 teaspoon	
		Boiling water	350 mL	
2	Anti-flu mixture	Water	0.5 cup	Fruit juice, garlic, ginger, and honey were blended, then water and turmeric were added, and the whole mixture was blended again (Szulc-Górska, 2022)
		Lemon juice	Half a lemon	
		Grapefruit juice	Half a grapefruit	
		Orange juice	Half an orange	
		Grated garlic	1 clove	
		Ginger root	1.5 slices	
		Honey	1.5 tablespoons	
Ground turmeric	0.25 teaspoon			
3	Anti-flu mixture with lemon	Lemon juice	2 lemons	The juice of the respective citrus was blended with garlic, honey, turmeric, ginger, and water. The sample was set aside in a dark place for 8 hours (Lokalny Rolnik, 2019)
		Grated garlic	10 cloves	
		Honey	2 tablespoons	
		Ground turmeric	1 teaspoon	
		Ground ginger	1 teaspoon	
		Water	1 glass	
4	Anti-flu mixture with orange	Orange juice	2 oranges	
		Grated garlic	10 cloves	
		Honey	2 tablespoons	
		Ground turmeric	1 teaspoon	
		Ground ginger	1 teaspoon	
		Water	1 glass	
5	Anti-flu mixture with grapefruit	Grapefruit juice	2 grapefruits	
		Grated garlic	10 cloves	
		Honey	2 tablespoons	
		Ground turmeric	1 teaspoon	
		Ground ginger	1 teaspoon	
		Water	1 glass	
6	Bomb for the cold	Grated ginger	15 g	All ingredients were mixed and poured over warm, but not boiling, water. The covered sample was set aside for 5-10 minutes (Węgrzyn, 2022)
		Lemon juice	1 lemon	
		Honey	1 teaspoon	
		Cloves	6 pieces	
		Ground turmeric	1 teaspoon	
		Olive oil	1 teaspoon	
7	Natural antibiotic 1	Grated garlic	2 cloves	All ingredients were mixed, and set aside at room temperature for 24 hours (Stachura, 2021).
		Grated ginger root	2 teaspoons	
		Honey	3 tablespoons	
		Lemon juice	3 tablespoons	
8	Natural antibiotic 2	Lemon juice	0.25 glass	The ingredients were mixed (Mucha, 2014).
		Honey	1 teaspoon	
		Grated garlic	0.5 cloves	

		Grated ginger root	0.25 teaspoon	
		Cayenne pepper	0.25 teaspoon	
		Ground cinnamon	0.5 teaspoon	
9	Natural antibiotic 3	Lemon juice	half a lemon	The ingredients were blended and pressed through a sieve to make syrup (Maciąg, 2015).
		Honey	3 tablespoons	
		Grated garlic	5 cloves	
		Grated ginger root	3 cm	
		Red onion	1 onion	
		Cloves	2 pieces	
		Star aniseed	1 piece	
10	Natural antibiotic 4	Grated garlic	1 clove	Grated garlic was left for 10 minutes. Next, it was added to lemon juice with ginger, chili, and cinnamon, and mixed. Next, honey was added, mixed again, and the sample was left at room temperature for 3 hours (Fizia, 2016).
		Honey	2 tablespoons	
		Ground ginger	2 tablespoons	
		Ground chilli peppers	0.5 teaspoon	
		Ground cinnamon	0.5 teaspoon	
		Lemon juice	Half a lemon	
11	White infusion	Cinnamon bark	3 cm	The ingredients (excluding honey) were poured over boiling water, and steeped for 15 minutes covered. Once cooled, honey was added (Korczyk, 2022).
		Ginger root	3 cm	
		Cloves	8 pieces	
		Saffron	Two threads	
		Honey	1 teaspoon	
12	Modified white infusion	Ginger root	5 cm	The ginger root was sliced and poured over boiling water with the other ingredients. The sample was brewed for 15 minutes covered (Pstrąg-Jaworska, 2022).
		Cinnamon bark	1 piece	
		Cloves	5 pieces	
		Cardamom	4 grains	
		Star anise	1 piece	
		Honey	1 teaspoon	
		Lemon juice	Half a lemon	
		Orange	2 slices	
13	Golden milk	Almond beverage	1 glass	Ingredients excluding honey were heated for 10 minutes without bringing to a boil. Honey was added at the end (Sejbuk, 2023).
		Ground turmeric	1 teaspoon	
		Black pepper	Pinch	
		Clarified butter	1 teaspoon	
		Honey	1 teaspoon	
		Ground cinnamon	1 teaspoon	
		Grated ginger root	2 cm	
14	Modified golden milk	Almond beverage	1 glass	Milk, turmeric, and pepper were heated for 10 minutes without bringing them to a boil. Coconut oil was added and when the drink cooled down, honey and cinnamon were added (Aura Herbals, b.d.).
		Unrefined coconut oil	1 teaspoon	
		Ground turmeric	1 teaspoon	
		Ground cinnamon	1 teaspoon	
		Black pepper	Pinch	

Source: own research.

Microorganisms

In the experiment, eleven indicator microorganisms were chosen for the studies among which were Gram-positive bacteria including: *Micrococcus luteus* ATCC® 4698™, *Bacillus subtilis* ATCC® 11774™, *Listeria monocytogenes* ATCC® 19115™, *Staphylococcus aureus* ATCC® 33862™, *Enterococcus faecalis*

ATCC® 19433™, Gram-negative bacteria: *Escherichia coli* ATCC® 8739™, *Salmonella enterica* ser. Enteritidis ATCC® 13076™, *Pseudomonas aeruginosa* ATCC® 9027, *Proteus vulgaris* PCM 542, as well as yeasts: *Rhodotorula mucilaginosa* DKK 040, and *Candida albicans* ATCC® 10231™. Indicator microorganisms were obtained from the American Type Culture Collection (ATCC), Polish Collection of Microorganisms (PCM), and the Poznań University of Economics and Business Department of Natural Science and Quality Assurance Collection (DKK). All indicator strains used for the experiments were stored using Microbank® cryogenic beads (BIOMAXIMA, Lublin, Poland) at –22°C. Each time before the studies, microorganisms were subcultured into the broth medium and after incubation re-subcultured onto agar medium appropriate for a specific strain. The incubation conditions, used media as well as the time of incubation are listed in Table 2.

Tab. 2. Indicator microorganisms, incubation conditions, and the microbial media used for the studies

No.	Microorganism	Incubation time	Incubation time	Microbial media
Gram-positive bacteria				
1	<i>Micrococcus luteus</i>	30°C	24-48 h	Trypticasein Soy Broth (TSB) and Agar (TSA)
2	<i>Bacillus subtilis</i>	37°C	24 h	Nutrient Broth (NB) and Nutrient Agar (NA)
3	<i>Listeria monocytogenes</i>	37°C	24 h	Brain Heart Infusion (BHI) broth and agar
4	<i>Staphylococcus aureus</i>	37°C	24 h	Nutrient Broth (NB) and Nutrient Agar (NA)
5	<i>Enterococcus faecalis</i>	37°C	24 h	Brain Heart Infusion (BHI) broth and agar
Gram-negative bacteria				
6	<i>Escherichia coli</i>	37°C	24 h	Nutrient Broth (NB) and Nutrient Agar (NA)
7	<i>Salmonella enterica</i> ser. Enteritidis	37°C	24 h	Brain Heart Infusion (BHI) broth and agar
8	<i>Proteus vulgaris</i>	37°C	24 h	Brain Heart Infusion (BHI) broth and agar
9	<i>Pseudomonas aeruginosa</i>	37°C	24 h	Nutrient Broth (NB) and Nutrient Agar (NA)
Yeasts				
10	<i>Rhodotorula mucilaginosa</i>	30°C	24–48 h	Sabouraud Dextrose Broth and Agar with Chloramphenicol
11	<i>Candida albicans</i>	30°C	24–48 h	Sabouraud Dextrose Broth and Agar with Chloramphenicol

Source: own research.

Microbial media for the tests were purchased from BIOMAXIMA, and were selected according to the microorganisms' nutritional requirements.

Methods

Antimicrobial assay

The antimicrobial properties of the prepared mixtures were examined by the well-diffusion method. From fresh cultures of indicator bacteria, suspensions were prepared in sterile saline with an optical density set at 0.5 on the McFarland scale. The cultures were made by the plating method, and after solidifying the agar the wells were cut out using a sterile cork bor with a diameter of 10 mm. Then undiluted mixtures in the amount of 100 μ L were introduced into the wells. Samples were incubated followed by the condition presented in Table 2. The diameter of growth inhibition including the well (10 mm) was measured to obtain the results. As the control, 100 μ L of water was used. The study was conducted in 3 parallel repetitions. The mean and standard deviation values were calculated for the obtained results (Microsoft Excel®).

Tab. 3. Composition of samples 8 ‘Natural antibiotic 2’ and 9 ‘Natural antibiotic 3’ variations in the individual component removal tests

No.	Lemon juice	Honey	Garlic	Ginger root	Cayenne pepper	Cinnamon	Red onion	Cloves	Star aniseed
8.0	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>			
8.1	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>			
8.2	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>			
8.3	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>			
8.4	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>			
8.5	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>			
8.6	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>			
9.0	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>			<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>
9.1	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>			<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>
9.2	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>			<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>
9.3	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>			<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>
9.4	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>			<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>
9.5	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>			<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>
9.6	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>			<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>
9.7	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>			<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>

Source: own research.

Furthermore, two samples (8 and 9) were selected for the second stage of the studies, as these mixtures inhibited the growth of all tested indicator microorganisms. Antimicrobial activity of mixtures prepared without individual ingredients was also performed, using the well-diffusion method. The approach was implemented to determine which ingredient had the greatest effect on the antimicro-

bial activity and the correlation between the ingredients. The positive control samples consisted of mixtures containing all the ingredients according to the initial recipe (Table 1), as the negative control, water was used. The test samples (8.1–8.6 and 9.1–9.7) were mixtures in which a specific ingredient had been omitted (Table 3), while the rest of the ingredients were used in unchanged quantities. The performed experiments included undiluted mixtures and were carried out in three parallel replicates. The mean and standard deviation values were calculated for the obtained results (Microsoft Excel®).

Results and discussion

In the first step of research, samples of fourteen mixtures were analysed to determine their ability to inhibit the growth of eleven indicator microorganisms. The obtained results were divided into three groups in terms of the tested microorganisms groups and presented in Figures 1-3. The majority of tested mixtures showed antimicrobial activity towards Gram-positive bacteria (Figure 1), with the best results demonstrated by *Natural antibiotic 1-4*. These mixtures inhibited the growth of all tested Gram-positive bacteria with the average inhibition zones from 19.00 to 37.50 mm, depending on the mixture and microorganism. Also, *Bomb for the cold* inhibited the growth of all Gram-positive indicator bacteria, however, the inhibition zones were lower and reached from 13.33 to 26.67 mm. *Anti-flu mixtures* (samples 2–5) also demonstrated high antibacterial activity but didn't influence the growth of *E. faecalis*. The mixture called *Firecracker for the cold* inhibited the growth of three of five Gram-positive indicator bacteria: *M. luteus*, *B. subtilis*, and *S. aureus*, while *Modified white infusion* was active only towards *B. subtilis* and *S. aureus*. Three mixtures: *White infusion*, *Golden milk*, and *Modified golden milk* showed no antimicrobial activity.

In Figure 2 the results of the antibacterial activity of handmade mixtures towards Gram-negative bacteria are presented. Compared with the results obtained for Gram-positive bacteria it can be stated that Gram-negative bacteria were less sensitive to prepared mixtures and inhibition zones didn't exceed 30 mm diameter. The best results showed *Natural antibiotic 1-4* as well *Anti-flu mixture with lemon* (sample 3), inhibiting the growth of all tested bacteria belonging to the Gram-negative group. The *Anti-flu mixtures with orange and grapefruit* (samples 4 and 5) as well as *Bomb for the cold* inhibited the growth of three tested microorganisms including *E. coli*, *P. aeruginosa*, and *P. vulgaris*. *Anti-flu mixture* (sample 2) and *Modified white infusion* (sample 12) only inhibited the growth of *E. coli*, while *Firecracker for the cold*, *White infusion*, *Golden milk*, and *Modified golden milk* didn't show antibacterial activity towards Gram-negative bacteria. Comparing the sensitivity of the tested strains to the mixtures, it can be seen that *E. coli* was the most sensitive Gram-negative bacteria.

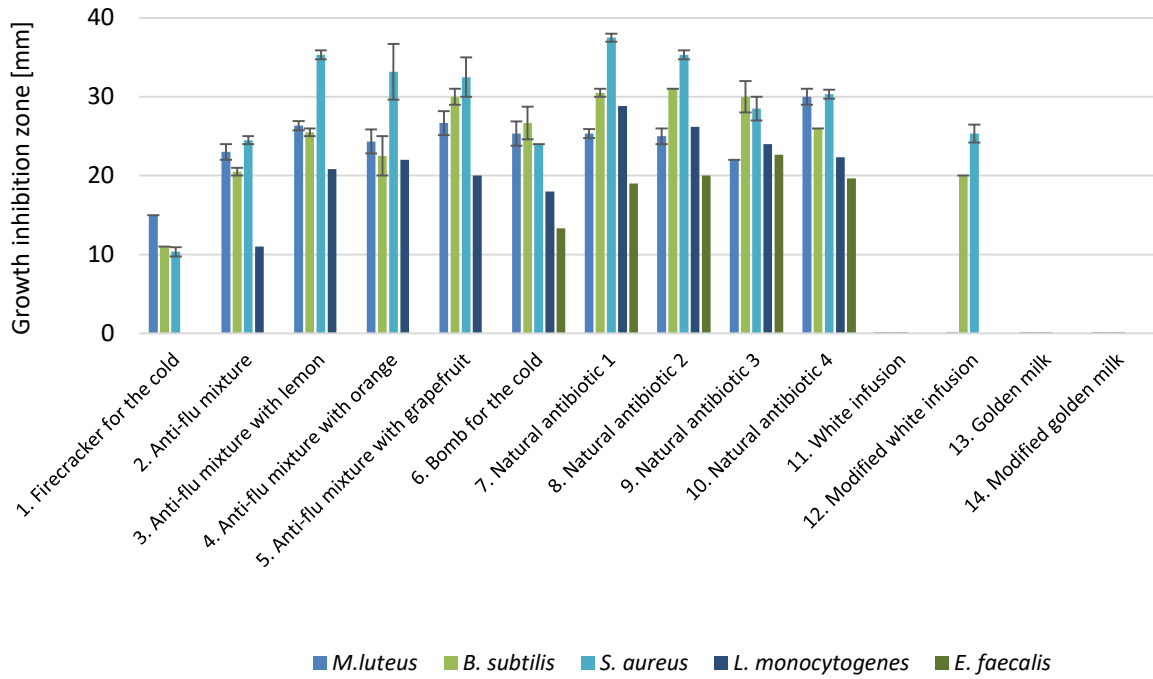


Fig. 1. Antibacterial activity of the tested natural mixes towards Gram-positive¹

¹The result values include the 10 mm well diameter

Source: own research.

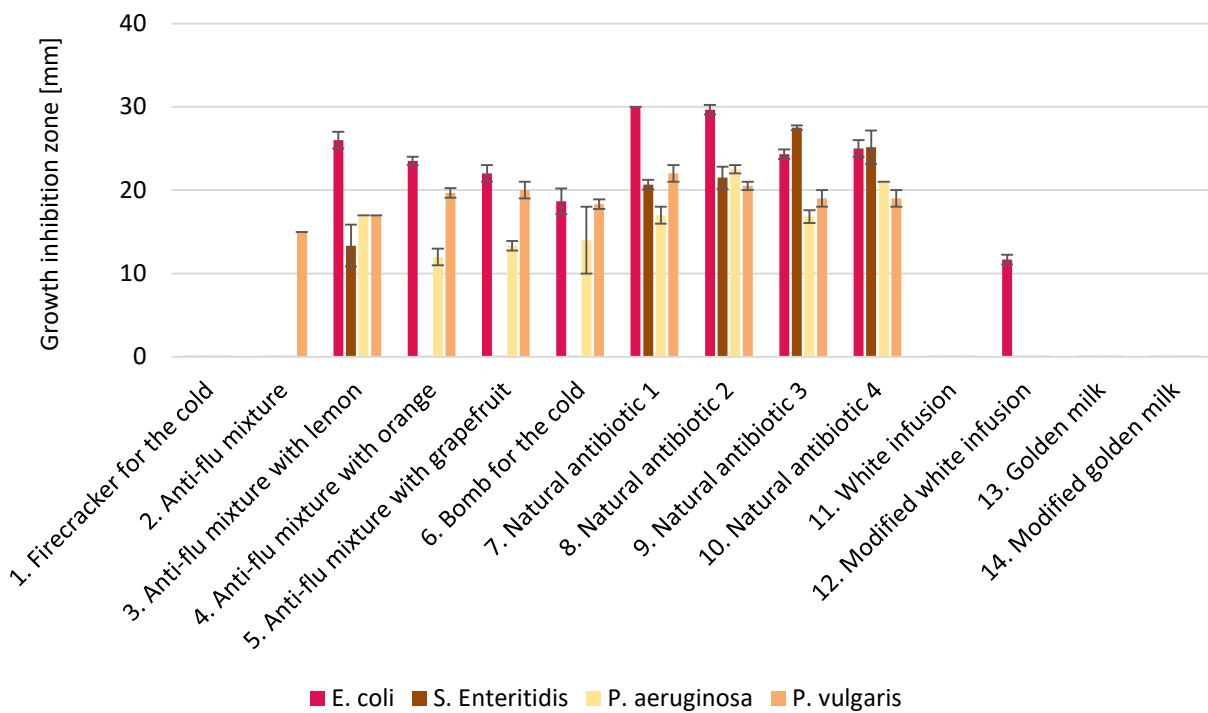


Fig. 2. Antibacterial activity of the tested natural mixes towards Gram-negative bacteria¹

¹The result values include the 10 mm well diameter

Source: own research.

The handmade mixtures were also tested for their activity towards fungi including *R. mucilaginosa* and *C. albicans* (Fig. 3). Both microorganisms were susceptible to the *Natural antibiotic 1–4* (samples 7–10) as well as *Anti-flu mixtures with lemon, orange, and grapefruit* (samples 3–5). The other mixtures didn't show activity towards examined yeasts. The sensitivity of fungi was similar and the growth inhibition zones reached from 21 to 41 mm, depending on the mixture.

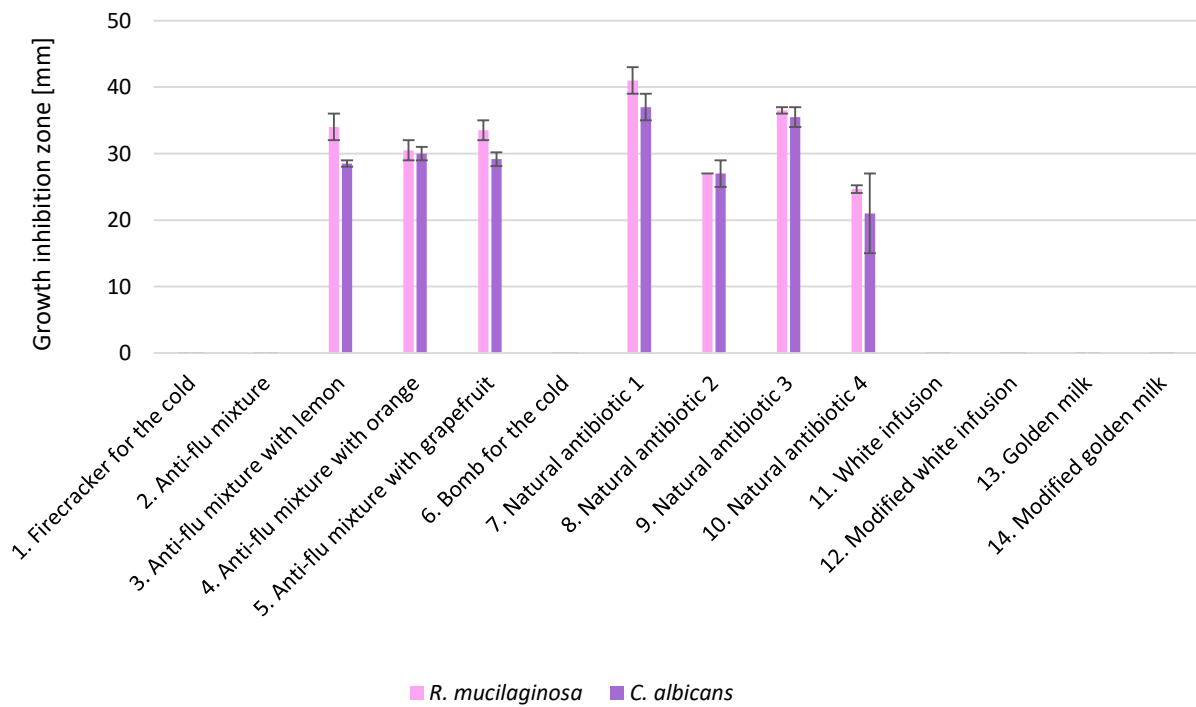


Fig. 3. Antifungal activity of the tested natural mixes towards yeasts¹

¹The result values include the 10 mm well diameter

Source: own research.

In the next step of experiments, the effect of mixture composition on the antimicrobial properties was assessed by the removal of the individual components and the determination of the activity of the preparation without this ingredient. Two mixtures with the highest activity have been chosen: *Natural antibiotics 2 and 3* (samples 8 and 9), while the list of indicator microorganisms has been limited to 8. The results are presented in Figures 4–5 and on the Photos 1 A–E.

Natural antibiotic 2 (sample 8) used in the full formula inhibited the growth of all microorganisms, while the composition changes affected the range of its activity. The most significant impact on the results was the lack of lemon juice, eliminating the activity of the mixture for *L. monocytogenes*, *S. Enteritidis*, and *P. aeruginosa*. The lack of this citrus juice also decreased the antimicrobial properties of the mixture towards *B. subtilis*, *S. aureus*, *E. faecalis*, *E. coli* and *C. albicans*, however, the sensitivity of yeasts decreased the lowest. On the other hand, the lack of cinnamon and cayenne pepper

increased the growth inhibition of *L. monocytogenes* and *S. Enteritidis*. It could be due to the antagonistic effect of the substances included in the mixture.

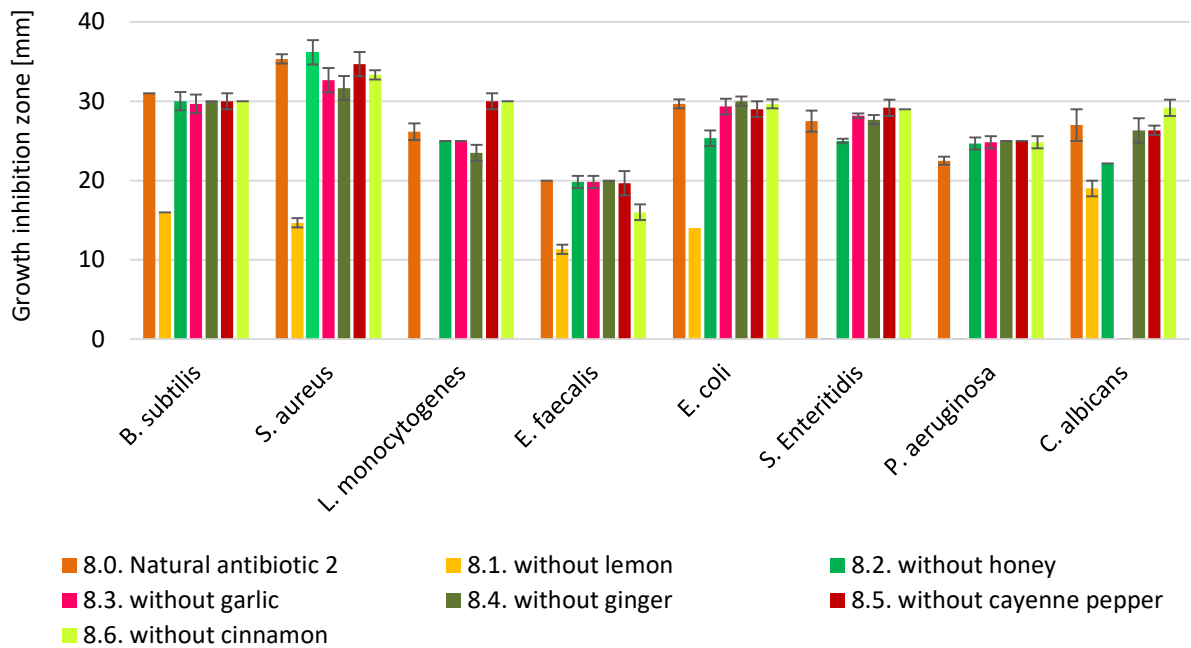


Fig. 4. Antimicrobial activity of *Natural antibiotic 2* samples variations

¹The result values include the 10 mm well diameter

Source: own research.

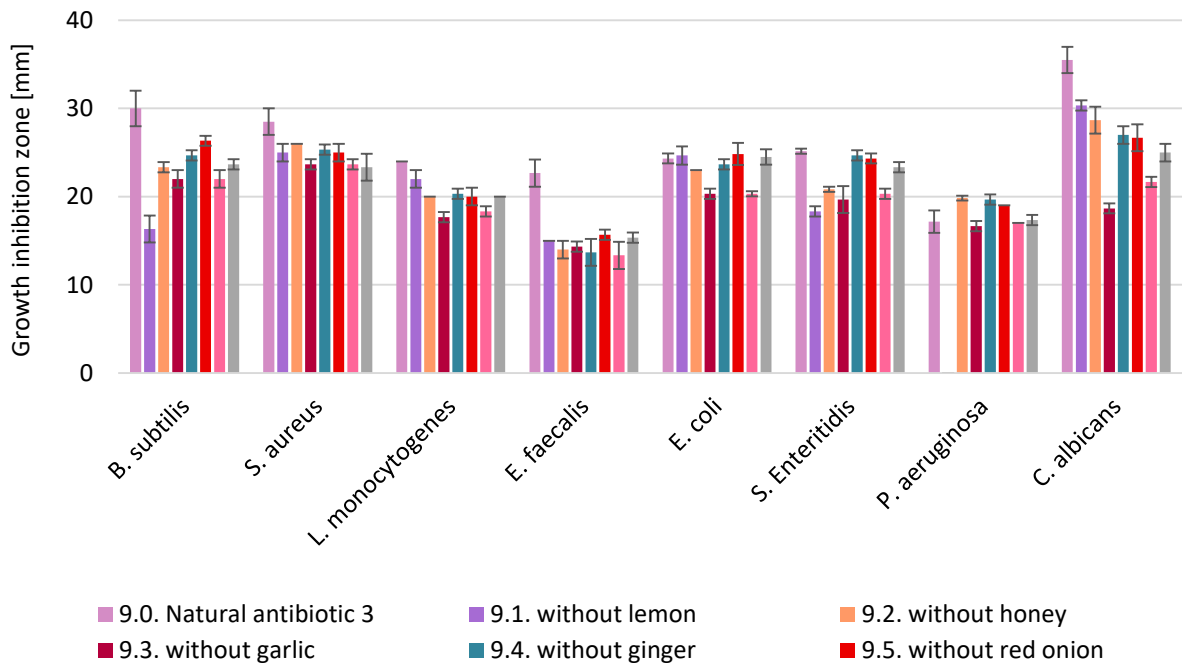


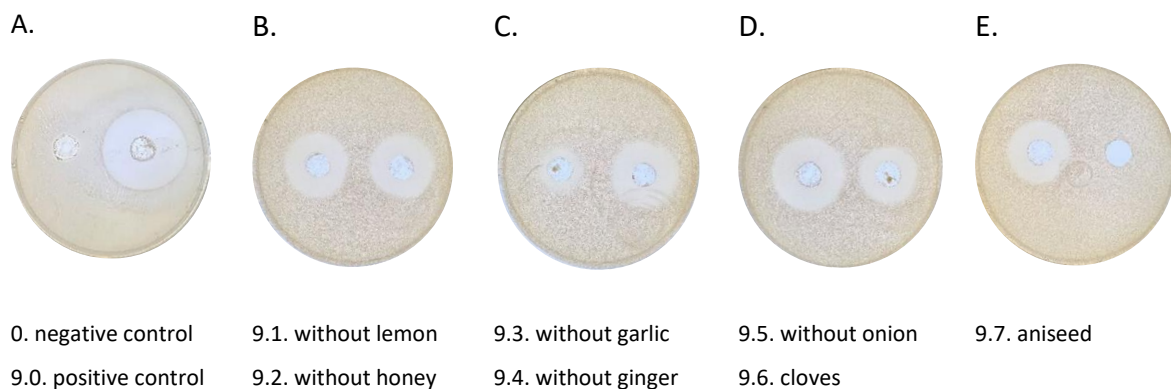
Fig. 5. Antimicrobial activity of *Natural antibiotic 3* samples variations

¹The result values include the 10 mm well diameter

Source: own research.

Natural antibiotic 3 (sample 9) variations effectively inhibited the growth of all microorganisms (Fig. 5). The indicator bacteria and yeast growth inhibition capacity weren't increased by removing any of the primarily used ingredients. The exclusion of individual components of the original composition resulted in obtaining the same or lower antimicrobial activity of the tested samples in comparison to the positive control (*Natural antibiotic 3*).

Different diameters of inhibition zones, shown in Photos 1. A–E demonstrates changes in the antimicrobial activity of sample 9 variations towards *C. albicans*. The sample prepared according to the original recipe displays the highest antimicrobial activity (Photo 1. A). The abundance of ginger (sample 9.3) decreased the diameter of the inhibition zone at the highest level (from 35.50 to 18.67, respectively). Furthermore, omitting cloves (sample 9.6) also lowered significantly the biological activity of the mixture (from 35.50 to 21.00). Whereas the lack of other used ingredients indicated a similarly declining trend in activity, but the reduction was not as high as for samples 9.3 and 9.6 (Photos 1. A–E).



Photos 1. A–E. Antimicrobial properties of *Natural antibiotic 3* samples towards *C. albicans*

Source: own compilation.

Summing up, tested natural homemade mixtures demonstrated different antimicrobial activity towards chosen indicator microorganisms. *Firecracker for the cold* (sample 1) had a low inhibiting effect only towards 3 out of 11 tested indicator microorganisms i.e. *M. luteus*, *B. subtilis*, and *S. aureus*. *Anti-flu mixtures* (samples 2–5) showed the tendency to inhibit the growth of Gram-positive bacteria and fungi stronger than Gram-negative bacteria. Except for *E. faecalis*, which was resistant to these samples. *Bomb for the cold* (sample 6), showed mild antibacterial properties towards 8 indicator bacteria, with no activity against fungi. In the group of *Natural antibiotics 1–4* (samples 7–10), all mixtures effectively decreased the growth of all tested microorganisms. The results are much more similar to each other within the group, in comparison to the other groups. *White infusion* did not pose any antimicrobial activity towards tested microorganisms, whereas the *Modified white infusion* inhibited the growth of 3 tested bacteria (samples 11 and 12, respectively). The obtained results were

insufficient to confirm the antimicrobial effectiveness of *Golden milks* (samples 13–14), casting doubt on the claimed properties of these two beverages.

It is worth underlining that most mixtures were rich in compounds such as garlic, honey, onion, or lemon juice known for their strong antimicrobial properties. Garlic is an ingredient used in folk and traditional medicine due to its antibacterial, antifungal, antiviral, anti-inflammatory, and even anticancer properties. The main antibacterial compound is allicin, forming from a precursor alliin. Literature data confirm garlic antibacterial activity in different forms: crude, fresh extract or garlic paste (Bhatwalkar et al., 2021; Rivlin 2001; Roshan et al., 2017). Onion contains organic sulphur compounds and allinase enzyme as well as is rich in flavanoids, therefore, it exhibits wide biological activity including, among others, antioxidant, antibacterial, and antifungal properties (Shon et al., 2004). It is worth emphasizing that red onion has more flavanols compared with white onion (Benmalek et al., 2013). The antimicrobial properties of honey are strongly dependent on its botanical origin, season and harvest conditions, processing, and storage (Molan & Cooper, 2000), however, some types of honey are known for their antibacterial activity, due to the presence of hydrogen peroxide or non-peroxide components as well as low water activity causing osmosis (Viuda-Martos et al., 2018, Kumar et al., 2010). In turn, citrus fruits are also known to contain bioactive compounds such as phenolics, flavonoids, vitamins, and essential oils affecting their antimicrobial properties (Arouma et al., 2012; Karimi et al., 2012). Among the ingredients frequently used to compose natural remedies is ginger, known for its biological activity such as antibacterial, antifungal, antiviral, antioxidant or antitumor (Freire et al., 2017; Shukla & Singh, 2007; Singh et al., 2008).

The results of the studies' second stage – individual component removal tests, mostly confirmed that the originally tested compositions of samples 8 and 9 showed the highest antimicrobial activity towards selected bacteria and yeasts. *Natural antibiotic 2* in its original composition and several variations posed similar inhibitory effects. The elimination of lemon juice (sample 8.1) resulted in the highest decrease in antimicrobial properties of the *Natural antibiotic 2*, to the extent that 3 bacteria were not affected at all. *Natural Antibiotic 3* was the most effective in decreasing the growth of the tested microorganisms when all ingredients from the recipe were used. Exclusion of any ingredient led to the reduction of microorganisms' inhibition. Nevertheless, even with the exclusion of each ingredient all of the sample 8 variants still inhibited all the microorganisms. It can be concluded that self-prepared mixtures show antimicrobial activity, however, the composition had a crucial effect on their effectiveness in the inhibition of microbial growth.

Conclusions

The majority of tested natural homemade remedies exhibited antimicrobial properties towards tested indicator bacteria and yeasts. The results highly depended on the composition, indicating that homemade mixtures prepared according to recipes from websites or social media do not always meet the expectations. Despite the author's declaration, some of them did not have antimicrobial properties. Moreover, the main limitation of any natural remedies treatments is the lack of standardization. Hence the reproducibility of the results obtained from each batch of the natural remedies might be different. The quality and the composition of plant products may differ due to various factors such as the natural genetic variation of plant species, or their origin, so the same product may exhibit different properties. It is one of the reasons for the low credibility regarding the efficacy of medicinal plants. Plant-based remedies limitations also are associated with the availability of plant species in particular geographical areas.

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THE EFFECT OF OZONATION ON THE MICROBIOTA OF EDIBLE FLOWERS

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DOI: 10.56091/CTQS.Qual-5

Abstract

Edible flowers are extremely perishable products. Storing edible flowers may lead to significant growth of microbiota, making them unsuitable for safe consumption. Due to the lack of possibility of interfering in the process of flower cultivation, our work focused only on the processes from harvesting to the delivery to the consumer. The aim of this study was to evaluate the effect of ozonation on the microbial quality of edible flowers including marigolds, pansies, and daisies.

Unpackaged flowers and flowers in packages (PET boxes) immediately after harvesting were ozonated with ozone at a concentration of 1 and 3 ppm. The control attempt was the flowers not called ozonation. The presence of the following microorganisms were determined on the flowers: the number of yeasts and moulds, the number of *Staphylococcus aureus* (*S. aureus*) and the number of *Escherichia coli* (*E. coli*).

The results showed that all flowers were contaminated with yeast and moulds at counts 2.7–6.03 log CFU /g and 2.48–4.18 log CFU/g respectively, while only pansies and daisies were contaminated with *Escherichia coli* at counts ranging from 1.5 to 3 log CFU/g. All the tested flowers also contained *Staphylococcus aureus* at numbers ranging from 2.08 to 3.07 log CFU/g. The differences in the counts of moulds and *S. aureus* were statistically significant depending on the type of flower. Ozonation did not significantly affect the degree of microbiological contamination.

Keywords: edible flowers, microbial quality, ozonation

Introduction

In recent years, a change in eating behaviour can be observed. More and more often, consumers are looking for new, unknown products. Such foodstuffs include, among others, edible flowers, which not only decorate various types of dishes, but also give them a unique taste and aroma. In the literature, edible flowers have been defined as non-toxic, harmless, with health benefits flowers that are safe to consumer. However, it should be emphasized that although most of the flowers have been classified as safe for consumption, official lists of edible and inedible flowers have not yet been developed (Mlcek and Rop, 2011; Lu et al., 2016; Purohit et al., 2021). Flowers have been used for hun-

dreds of years for the production of teas, wine, as well as an addition to marinades and jams. Historical sources say that edible flowers were present in Middle Eastern, Indian and Roman cuisine (Pinakin et al., 2020). Nowadays they are becoming more and more popular dietary ingredient all over the world. The most popular edible flowers include: chrysanthemum, lilac, mint, nasturtium, pansy, rose, tulip and violet.

Previous researches show that edible flowers are natural sources of antioxidants, including phenolic acids, flavonoids, carotenoids and anthocyanins directly related to colour. In addition, they are a source of natural preservatives, colouring and flavouring agents. They also show strong antimicrobial activity. Edible flowers support the treatment of many civilization diseases including diabetes, coronary heart disease, cancer, which are caused by oxidative stress (Demasi et al., 2021; Devecchi et al., 2021; Kaisoon et al., 2011; Lu et al., 2016; Navarro-González et al., 2015; Pinakin et al., 2020; Skrajda-Brdak et al., 2020; Zhao et al., 2021).

Flowers are extremely perishable products. As our previous research has shown, most edible flowers can be contaminated with pathogenic bacteria as well as yeasts and moulds. The sources of this pollution are primarily pre-harvest factors. The usual processes, such as packaging or refrigerated storage, are not able to reduce this contamination, and heat treatment is rather not used, making edible flowers unsuitable for safe consumption (Wilczyńska et al., 2021; Wilczyńska et al., 2023).

One of the methods of decontamination of food products of plant origin may be ozonation. Ozone is an antibacterial substance, has a high oxidation-reduction potential, to which gram-positive and gram-negative bacteria, viruses, fungi and bacterial spores are susceptible, including: *Listeria monocytogenes*, *Staphylococcus aureus*; *Escherichia coli*, *Salmonella Typhimrium*; *Candida parapsilosis* and *Bacillus cereus*. Microorganisms show varying sensitivity to ozone. Bacteria are more sensitive than fungi, and gram-positive bacteria more than gram-negative. In turn, bacterial spores are more resistant than vegetative cells (Brodowska et al., 2014; Dyas et al., 1983; Pascual et al., 2007; Póljanowska et al., 2007; Young and Setlow, 2005).

It should be emphasized that no guidelines have been developed so far, including standards regarding both the collection and storage of edible flowers. As these are products that are commonly consumed raw without any washing or prior thermal treatment, to preserve aesthetic value, consumers do not have the opportunity to eliminate harmful microflora. That is why in our work we focused on the possibility of reducing microbiological contamination by ozonizing ready-to-eat edible flowers. We assumed that ozonation reduces number of microorganisms.

Materials and methods

The research material consisted of selected edible flowers obtained directly from Ogródnictwo Lawenda, Gdańsk: marigolds (*Tagetes* L.), pansies (*Viola × wittrockiana* Gams), and daisies (*Bellis* L.). Immediately after harvesting, the flowers were ozonated with two doses of ozone at a concentration of 1 and 3 ppm, in the packages (PET boxes) and without the packages, for 15 minutes. In all flower samples – the control samples (non-ozonated) and the ozonized samples, the presence of the following microorganisms were determined: the number of fungi on the Merck Yeast Extract Glucose Chloramphenicol agar (incubation at 25°C for 120 h), the number of *Staphylococcus aureus* on Merck's Baird Parker + Rabbit Plasma Fibrinogen medium (incubation at 37°C for 48 h) and the number of *Escherichia coli* on a selective Coli ID medium by bioMerieux (incubation at 37°C for 48 h) (Wilczyńska et al., 2021; Wilczyńska et al., 2023). The entire experiment was repeated three times (research cycles I–III) in one growing season. A total of 45 samples of edible flowers were analysed.

From the obtained results, the mean and standard deviation were calculated using Statistica v. 13.3 software (StatSoft, Tulsa, Oklahoma, USA). One-way ANOVA analysis of variance was conducted to investigate the effect of ozonation on the microbial quality of edible flowers.

Results and discussion

Edible flowers, like other low-processed food products of plant origin, retain their physiological activity until they are consumed. They are not subjected to thermal preservation, therefore they are easily spoiled by microorganisms. Therefore, the selection of raw material, production hygiene and maintaining appropriate conditions in production and trade are of particular importance. The degree of flower contamination immediately after harvesting is presented in Table 1. None of the flowers tested in the first and second cycles were contaminated with *E. coli*. Contamination with this bacterium appeared in the third cycle of research, in pansies and daisies, at 3.29 log CFU/g and 1.85 log CFU/g, respectively. Although the main source of *E. coli* infection are animal products, also plant materials, especially lettuce, can be contaminated with this bacterium, which can be pathogenic to humans (Szczech et al., 2014). As our previous studies have shown, some edible flowers, especially those in contact with organic fertilizers, may contain *E. coli* in varying amounts (Wilczyńska et al., 2021; Wilczyńska et al., 2023). According to Fernandes et al. (2020) in most of the edible flowers counts for total coliforms and *E. coli* were less than 1 CFU/g. No legal acts specify the acceptable levels of microbiological contamination in edible flowers, but in accordance with Commission Regulation (EC) No 2073/2005 of 15 November 2005 on microbiological criteria for foodstuffs ready-to-eat fruits and vegetables should contain no more than 3 log CFU *E. coli*/g. In the light of these requirements, the level of contamination of the tested edible flowers with *E. coli* can be considered as high.

Tab. 1. Contamination of tested samples with *E. coli*, *S. aureus*, moulds and yeasts [log CFU/g]

Type of flower	Cycle of research	<i>E. coli</i>	<i>S. aureus</i>	moulds	yeasts
marigolds	I	nd	2.08	2.48	2.70
pansies	I	nd	3.07	3.04	3.75
daisies	I	nd	2.73	4.18	6.03
marigolds	II	nd	1.30	2.30	2.30
pansies	II	nd	1.85	3.83	4.75
daisies	II	nd	1.60	3.62	2.79
marigolds	III	nd	1.78	4.32	4.76
pansies	III	3.29	2.15	4.08	5.51
daisies	III	1.85	2.59	4.95	5.04

Source: own elaboration.

All tested flowers were contaminated with *S. aureus* at levels 1.3–3.07 log CFU/g, marigolds were the least contaminated and pansies were the most contaminated in all three series of tests. *S. aureus* are relatively common in the human environment. It is estimated that 10 to 50% of the human population are permanently or periodically carriers of these bacteria without the occurrence of disease symptoms. Unfortunately, these bacteria are increasingly found in plant-based foods. Sources of these bacteria in edible flowers may be production practices, growth conditions and the location of the edible part during growth, as well as all post-harvest operations (Beuchat, 2002; Elmacioglu et al., 2010, Wetzel et al., 2010). Like *E. coli* contamination, the *S. aureus* presence is an indicator of the poor hygienic conditions, especially during post-harvest operations. Staphylococci do not produce spores, but produce enterotoxin in the infected food product. Staphylococcal infection can cause purulent infections of the skin, subcutaneous tissues and soft tissues, systemic infections, infections or poisoning associated with the production of toxins.

Edible flowers are exposed to microbial contamination from soil, water and air during cultivation, harvesting and distribution. They can also be infected by insects and humans. Due to the high content of nutrients and water, they are an excellent breeding ground for microorganisms, especially moulds and yeasts. Microbiological guidelines for minimally processed vegetables and fruits (Ragaert et al., 2010) specify that the maximum number of yeasts and moulds after production may not exceed 3 log CFU / g, and on the last day of shelf life, 5 log CFU/g for yeasts and 4 log CFU/g for moulds. Considering these requirements, moulds contamination of the tested edible flowers, ranging from 2.3 to 4.95 CFU/g, can be considered moderate, while yeast contamination (from 2.3 to slightly more

than 6 CFU/g) – too high. The occurrence of yeasts and moulds at similar levels was found by researchers evaluating the microbiological quality of ready-to-eat salads and selected herbs (Adeyemi et al., 2019; Mohammad et al., 2012; Wójcik-Stopczyńska et al., 2010).

Statistical analysis showed that individual flower species differed significantly in contamination by *S. aureus* ($F=7.75$, $p=0.001$) and moulds ($F=4.51$, $p=0.01$) (Figure 1). There are few articles in the world literature on microbiological contamination of edible flowers, so it is difficult to say why some flower species are less contaminated than others. They may be more resistant to microbiological inoculation due to the content of biologically active compounds.

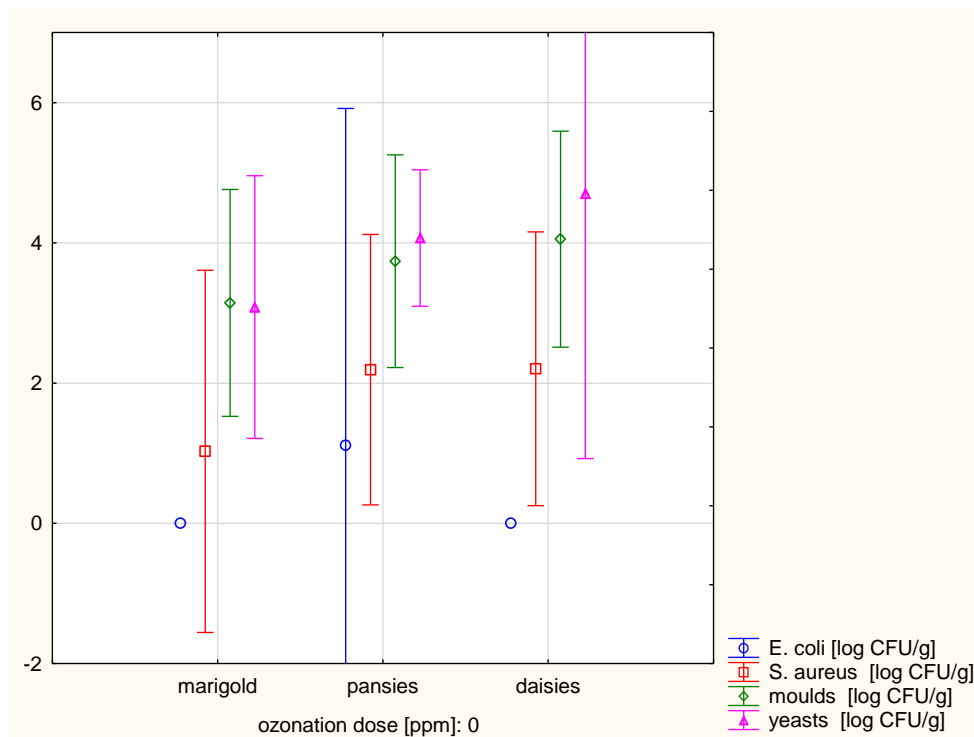


Fig. 1. Average contamination of particular flower species by bacteria, yeasts and moulds
Source: own elaboration.

As mentioned in the introduction, subjecting flowers to ozonation was expected to reduce their degree of microbiological contamination. Unfortunately, ozonation did not bring the expected effect and did not allow to reduce or eliminate contamination with bacteria, moulds or yeasts, regardless of whether they were ozonated in packaging or not (Table 2).

Tab. 2. Average contamination ozonated flower species with *E. coli*, *S. aureus*, moulds and yeasts

Flower's species	Ozonation dose [ppm]	<i>E. coli</i> [log CFU/g]		<i>S. aureus</i> [log CFU/g]		Moulds [log CFU/g]		Yeasts [log CFU/g]	
		PET	Not packed	PET	Not packed	PET	Not packed	PET	Not packed
marigolds	0 (control)	nd		1.26		3.62		4.37	
pansies	0 (control)	1.16		2.13		3.93		4.29	
daisies	0 (control)	0.65		2.05		4.17		4.41	
marigolds	1	nd	nd	1.36	1.69	3.89	3.93	4.56	4.33
pansies	1	1.09	2.39	1.98	2.12	4.05	4.16	5.00	5.53
daisies	1	1.10	1.70	2.09	2.88	4.30	4.97	4.28	5.61
marigolds	3	nd	nd	1.65	1.45	3.87	3.52	3.59	3.72
pansies	3	0.61	1.44	2.06	2.01	4.30	3.87	4.96	5.33
daisies	3	nd	0.73	2.02	2.09	4.56	4.20	4.27	4.35

Source: own elaboration.

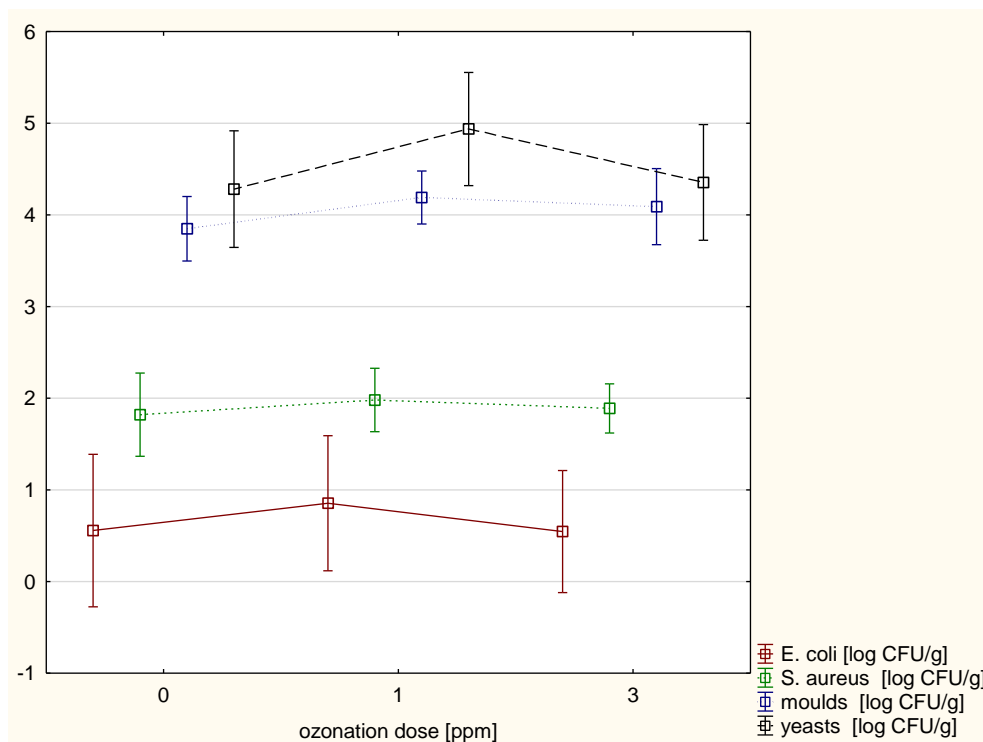


Fig. 2. Changes in the number of microorganisms in flowers subjected to ozonation

Source: own elaboration.

A slight increase in the number of all determined microorganisms was observed in edible flowers subjected to ozonation with ozone at a concentration of 1 ppm, but these changes were not statistically significant at $p > 0.05$ (Table 2, Figure 2).

In many countries ozone is used as an agent that effectively inhibits the growth of most microorganisms. The sensitivity of microorganisms to ozonation depends on many factors, including: temperature, humidity, content of various substances (e.g. acids, surfactants and sugars). Furthermore, the degree of inactivation is greater in systems without ozone requirement than when the medium contains oxidizable organic substances (Kim et al., 1999; Manousaridis et al., 2005). The effectiveness of disinfection by ozonation is assessed by determining the degree of reduction of microorganisms. In the case of bacteria, the reduction should be at least 3 logs, and the minimum level of mould and yeast reduction should be at least 1 log, which means a reduction in at least 90%. The time of ozonation is also important – the longer, the more effective disinfection is (Nowakowicz-Dębek et al., 2017).

The present study focused on the determining the effect of ozonation on the number of microorganisms in edible flowers. We assumed that ozone would reduce this number. Unfortunately the ozonation of edible flowers turned out to be an ineffective method of reducing unwanted microflora. Probably in our experience we subjected edible flowers to ozonation too briefly (15 minutes), which is why it did not bring the expected results.

Conclusions

According to the results of this study analysed edible flowers may contain pathogenic bacteria. Food pathogens can survive in flowers, and thereby represent a risk to the consumers in the home regard to food borne disease. Edible flowers are extremely perishable food products that require preservation methods that do not cause sensory changes. Ozone can be used as a safe and effective antimicrobial agent in many food applications, exposure to ozone extends the shelf life of certain products such as fruits and vegetables while preserving its sensory attributes. However the ozonation of edible flowers turned out to be an ineffective method of reducing unwanted microflora. Further studies should focus on investigating methods to reduce microbial populations in edible flowers.

Acknowledgments

We would like to thank the Gdynia Maritime University for providing funds for this study, project WZNJ/2023/PZ/01.

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THE POTENTIAL OF USING KMNO_4 -LOADED BENTONITE IN ACTIVE PACKAGING EXTENDING FRUIT SHELF LIFE

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DOI: 10.56091/CTQS.Qual-6

Abstract

One of the most perishable foods in the supply chain are fresh fruits and vegetables, especially those transported from distant areas, for which the ripening process takes place during storage and distribution. The flagship example is banana packed in the form of harvest maturity in tropical and subtropical countries, which acquire consumption maturity during distribution. In addition to controlling the transport and storage conditions, it is important to ensure that the level of ethylene inside the banana package is reduced, which should significantly affect the rate of ripening and may extend the shelf life of the fruits.

Despite the wide availability of various ethylene scavengers, most of them are based on the use of potassium permanganate. In addition to the active agent (responsible for neutralizing the phytohormone), it is also important to select a carrier that will increase the active surface of the packaging insert. Usually, zeolites, silica gels, or other inorganic substances with an extensive surface structure are used here.

The study focused on the evaluation of the effectiveness of ethylene absorbers based on potassium permanganate deposited on bentonite or its natrified derivative, and a comparison with a popular commercial ethylene absorber in the storage of fresh bananas. Storage tests were carried out both in closed packages and those ensuring free exchange of gases with the environment, where in both cases packed fruits were stored with normal access to sunlight or with significant limitations. During the storage of bananas, changes in their appearance (signs of fruit ripening and spoilage) were assessed, as well as changes in their weight (for open packaging) and the composition of the atmosphere inside closed packaging. The introduction of ethylene scavengers slowed down the fruit ripening process and extended significantly its shelf life. Absorbers on a bentonite carrier (natrified and natural) turned out to be more effective in this action.

Keywords: active packaging, bentonite, ethylene scavenger, potassium permanganate

Introduction

Fresh fruits are difficult to handle in the supply chain due to the need to ensure the desired harvest and commercial ripeness. This is particularly important for fruits that are grown in remote geograph-

ical areas in a specific microclimate and then transported long distances to markets that absorb large quantities of unprocessed foods such as bananas. Annual world exports of bananas in 2023 reached a volume close to 19.2 million tons, which came mainly from Ecuador (approx. 6.2 million tons), Guatemala (2.4 million tons), the Philippines (2.1 million tons each), Costa Rica and Colombia (2.0 million tons each), and supplied the European Union market (approx. 5.0 million tons), the United States of America (approx. 4.1 million tons), China and Japan (together approx. 2.8 million tons), and Russian Federation (1.4 million tons) (FAO, 2023). Despite the decline in banana exports in recent years, there has been a clear rebound and recovery from the more severe supply shortages of these fruits. Global trade in these fruits was estimated at \$13.6 billion in 2021 (OEC, 2023).

As a climacteric fruit, the main challenge in banana quality maintenance is to control the concentration of endogenous ethylene in packaging during banana storage and transportation (Pongprasert et al., 2020). Controlling the level of ethylene in the package significantly affects the rate of ripening and may extend the shelf life of the fruit (Álvarez-Hernández et al., 2018). Ethylene has biological activity at a concentration of $1 \mu\text{l}/\text{dm}^3$. In combination with other main storage conditions like temperature, humidity, and chemical content of the atmosphere, it plays a crucial role in the changes of fruit properties such as colour, texture, sweetness, aroma, volatile production, and nutritional value (Martínez-Romero et al., 2007, Pongprasert et al., 2020).

Damaged fruits and vegetables also release substantial amounts of ethylene, causing the spoilage of other fruits or vegetables stored in the same package. Therefore, to keep their parameters the same, it is best to remove ethylene from the place of storage. The effect associated with ethylene is used in the trade, e.g., in bananas, which are green during harvesting and transport. They are gassed with ethylene only before a sale to bring them to maturity quickly. Thanks to this, their maturation occurs simultaneously and evenly. The content of 0.02% ethylene in the air surrounding the fruit accelerates the ripening process 4–10 times (Brat et al., 2020).

Most ethylene scavengers are based on potassium permanganate (KMnO_4) and require a carrier that will increase the active surface of the packaging insert (Hu et al., 2019). KMnO_4 stimulates the oxidation of ethylene by breaking down the C_2H_4 double bond and transforming the target compound into carbon dioxide and water (Fig. 1a). The efficiency of the ethylene scavenging process can be significantly improved with porous carrier for potassium permanganate, which are usually zeolites, silica gels, or other inorganic substances with an extensive surface structure (Álvarez-Hernández et al., 2021; Gaikwad et al., 2020). The effectiveness of ethylene scavengers has been proven in the storage of packaged fresh fruit and vegetables (Dobrucka et al., 2017). Typically, ethylene scavenger contains approximately 4–6% KMnO_4 by weight. Additionally, the colour change from purple to brown associ-

ated with the reduction of permanganate can be used to observe the progress of the ethylene neutralization process (Vermeiren et al., 2003). Wills & Warton (2004) tested the ability to remove small concentrations of ethylene from the atmosphere using a potassium permanganate adsorbed on aluminum oxide (by immersing it in a saturated permanganate solution). The examined oxidant was able to remove ethylene from the closed system within 2.5 to 3 hours. It was also noticed that an increase in relative humidity harmed the ability to oxidize ethylene (Wills & Warton, 2004).

The ethylene absorber is often closed in sachets inserted inside the packaging. Several studies have shown that using KMnO_4 sachets delays fruit spoilage and extends the post-harvest shelf life of packaged fresh fruit. However, it is indicated that ethylene absorption may occur in a limited area around the sachet (Ebrahimi *et al.*, 2021). Packaging in a modified atmosphere (MAP) is sometimes used in addition to the ethylene absorber to prolong fresh fruit and vegetable shelf-life. A combined active packaging was also developed for ethylene scavengers. Álvarez-Hernández research group developed an antifungal active packaging containing thymol and C_2H_4 scavenger for cherry tomato packaging (Álvarez-Hernández et al., 2021). An ethylene scavenger with a moisture absorber was shown by Murmu et al. (2018) applied in guava MAP packaging with higher oxygen headspace and lower carboxy dioxide concentration. The active packaging eliminates molds on guava in MAP packaging after 30 days of storage. Fruits stored in optimal conditions showed lower weight loss and higher total phenol and ascorbic acid content retention, which probably resulted in more efficient guava resistance than the control sample (Murmu & Mishra, 2018). Pirsá (2021) studied a carboxymethylcellulose (CMC) hydrogel film modified with cellulose nanofibers (NFC) and potassium permanganate. The developed material was used as a moisture/ethylene absorber in banana packaging, and stored for 30 days at temperature of 0 and 25 Celsius degrees. It was found that the examined active films delay the fruits ripening, stabilizing their firmness. The carboxymethylcellulose film containing KMnO_4 can influence the ethylene concentration and humidity in the package, simultaneously, and therefore delay banana ripening and microbial spoilage.

The study focused on the evaluation of the effectiveness of ethylene scavengers based on KMnO_4 deposited on bentonite or its natrified derivative as a carrier for the active agent. Developed C_2H_4 scavengers were compared with a popular commercial ethylene absorber in the storage of fresh bananas. Storage tests were carried out in closed packages as well as in open versions which ensure free exchange of gases with the environment. Both types of fruit packaging were stored with normal access to sunlight or with significant limitations. During the storage of bananas, changes in their appearance (signs of fruit ripening and spoilage) were assessed, as well as changes in their weight (for open packaging) and the composition of the atmosphere inside closed packaging.

Materials and methods

The banana fruits were obtained from a popular retail chain just before they were displayed for sale. Fresh bananas (half green – half yellow colour and without any visible damage) from one batch were packed into polypropylene boxes (750ml cap.) and welded with lid foil (PET/PE) on the packaging machine, CAS CTP-320 (CAS Polska sp. z o.o.) sealer, tightly (closed packages) or only on one shorter side (open packages). The selected packages contained a sachet (welded Tyvek® 42 g/m², 40x40 mm), which was filled with 1 g of an ethylene scavenger (ES) – one of three: commercial DeltaTrack (Cat. No. 19010) absorbers (DES – Fig. 1b), KMnO₄-saturated bentonite (BES) or its nitrified derivative (nBES). KMnO₄ saturation of bentonite and its nitrified derivative was carried out in a saturated KMnO₄ solution, after which the absorber was dried at 80°C (Dobrucka et al., 2017). Nitrification of bentonite was aimed at increasing its active surface and was carried out using CaCO₃ as described (Dolinska et al., 2015). DeltaTrack (Cat. No. 19010) ethylene absorber represents one of the most popular forms of C₂H₄ absorbers available on the market, i.e. activated aluminium beads impregnated with potassium permanganate.

Packaged bananas were stored for min. 11 days at room temperature with full or limited access to sunlight. Storage conditions and packaging variants were selected to reflect the fruit storage conditions in retail outlets and to enable the assessment of the impact of packaging and storage conditions on the durability of bananas and their changes during ripening. Each type of tested packaging (with different storage conditions and/or tested C₂H₄ scavenger) was multiplied to eliminate accidental changes in stored fruit caused by uncontrolled factors, e.g. previously invisible banana damage or microbiological contamination. The appearance of the fruits was assessed by monitoring the colour/changes on the surface and the weight of bananas (only in open packages). In closed packages, the degree of tension and moisture of the package, as well as the atmosphere (concentration of O₂, CO₂, N₂ using OXYBABY® analyzer, WITT-Gasetechnik GmbH & Co KG) were assessed.

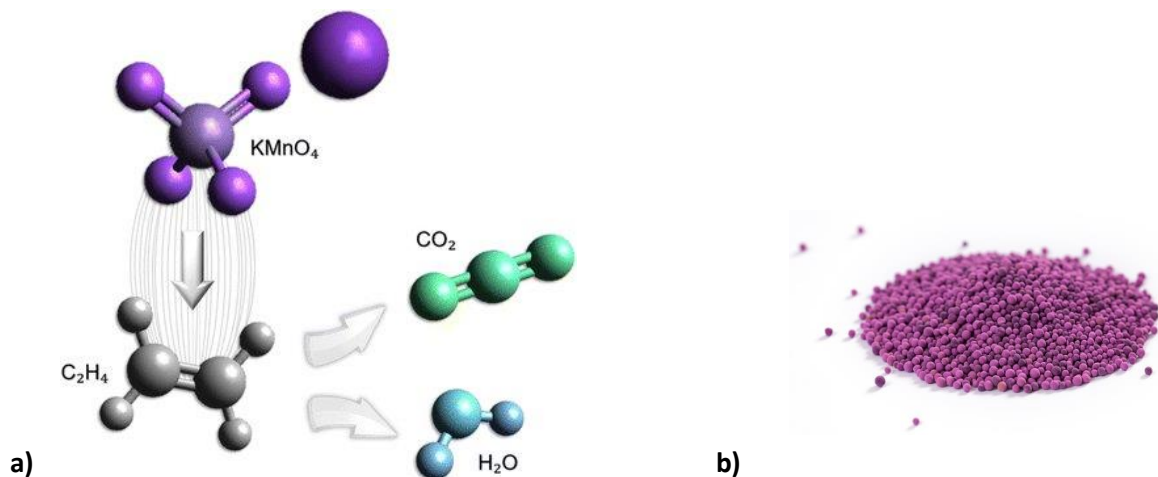


Fig. 1. Mechanism of action of KMnO₄-based ethylene scavenger (a) and commercial DeltaTrack (Cat. No 19010) ethylene absorber in the form of activated aluminium beads impregnated with potassium permanganate (b)

Source: based on Alvarez-Hernandez et al., 2018; DeltaTrack, 2023.

Results and discussion

The effectiveness of the tested ethylene absorbers was observed for packaged bananas stored at room temperature. Sunlight is one of the factors accelerating fruit ripening and ethylene release, so two types of storage tests were planned – one with unlimited access to sunlight (all packaging were transparent and exposed to daylight), the other with limited access to the sun (all packaging were stored in a closed room, without access to sunlight).

During the storage of fresh fruit, volatile compounds, and gases are exchanged in the space adjacent to them as a result of metabolic processes and fruit transpiration. This exchange runs in a different way depending on the degree of the packaging tightness and the flow of volatile and gaseous substances to/from the environment. In a tightly closed package, it is possible to influence the composition of the atmosphere inside. A 5-point scale was used to assess packaging inflation, with levels 1-5 meaning: (1) none (-) – primary concavity of the lid (lid foil) closing the package; (2) low – noticeable reduction in primary lid concavity; (3) medium – disappearance of the primary lid concavity and tension of the lid foil; (4) high – noticeable lifting of the lid and tension of the lid foil; (5) very high – significant lifting of the lid and tension of the lid foil and box walls. The action of absorbers may be more effective in the closed packaging, but there is a problem of excess metabolic products, e.g. water vapour. A 5-point scale was also used to assess the humidity level inside the packaging, in which levels 1–5 meant: (1) none (-) – none; (2) low – very small water vapor on the packaging walls; (3) medium – all walls of the packaging were covered with water vapor; (4) high – water vapor is on all the walls of the packaging and begins to flow in drops; (5) very high – the intense release of water

vapor causes water to collect at the bottom of the packaging. Open packaging allows for a more free exchange of gases and volatile compounds, but the action of absorbers may be less effective. For this reason, the assessment of the potential of the tested ethylene absorbers was checked in both open and closed packages.

The results of banana storage with different types of ethylene scavengers are presented in Tables 1–4 (depending on the type of packaging and degree of sunlight access). The light grey background indicates the moment of significant changes in the fruit’s appearance, while the dark grey background indicates the fruit's spoilage.

Efficiency of ethylene absorbers in closed packaging

Closing bananas in airtight packages allows for the assessment of the gas composition inside the package (with or without the application of the assessed ethylene absorber) in both storage variants – without restriction and with limited access to sunlight (Fig 2. a, b respectively).

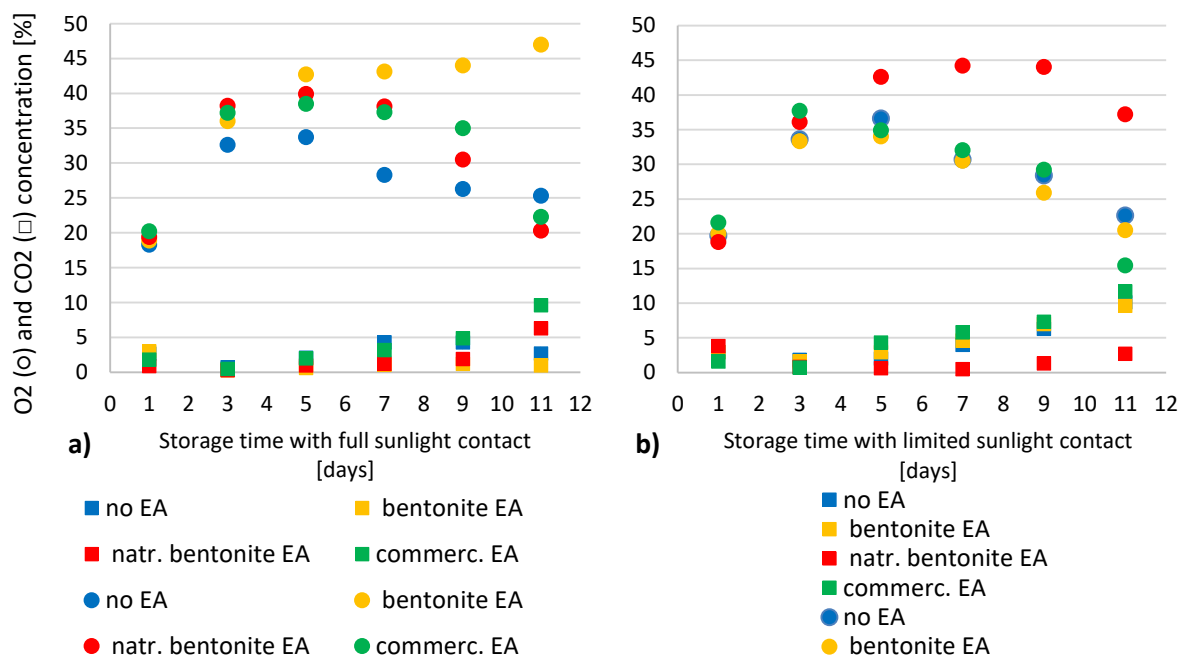


Fig. 2. Oxygen (○) and carbon dioxide (□) concentration during the banana storage inside packaging without ethylene absorber (no EA) or with different type of the absorber (EA) based on bentonite, natrified bentonite or commercial solution, with full (a – figure on the left) and limited (b – figure on the right) access to sunlight

Source: own study.

Regardless of the way the fruit was packed, an increase in oxygen concentration was observed in the first days of storage, which then decreased when signs of fruit spoilage appeared. Unfortunately, no relationship was observed between the measured concentration of oxygen and carbon dioxide inside

the packaging and the durability of fruit stored with/without the addition of the tested absorbents, but for some absorbers that allowed for prolonged storage of fruit, the oxygen level in the packaging remained at a higher level – e.g. adsorber on bentonite in packages exposed to sunlight and packages with adsorber on natrified bentonite stored with limited access to light. The above-mentioned absorbers also ensured the slowest increase in carbon dioxide concentration in packaging during fruit storage, which is probably related to the extended shelf life of the fruit in this case and the slower changes in bananas. More crucial information about the effectiveness of absorbers was provided by observations of stored fruit and their packaging.

In closed packages exposed to unlimited sunlight, a significant excess of water vapour appeared, which is associated with more intensive ripening and metabolic activity of the packaged fruit, which causes a more intense release of water vapour. Similarly, greater inflation of packaging is also observed for the packed fruits that were stored with unlimited access to sunlight (Table 1). Increased gas pressure and a raised amount of water vapour appear just before the first symptoms of over-ripeness of the packed bananas. During the progressing process of fruit spoilage, the gas pressure in the packaging slowly decreases, which may be partly due to the packaging not being completely tight, but mainly due to changes in the properties of the fruit. Unfortunately, it was not possible to thoroughly examine the condition of the fruit without opening the packages. However, the impact of the particular ethylene absorbers on the rate of ripening and spoilage of closed bananas was assessed.

The application of ethylene absorbers in closed packaging, in which bananas had unlimited access to sunlight, extended the shelf life of these fruits (Table 1). The least effective was the absorber based on commercial aluminum beads impregnated with potassium permanganate, which extended the shelf life of the fruits by two days until the first signs of spoilage and by four days until the bananas significantly spoiled. The use of an absorbent (KMnO_4) applied to bentonite additionally extended the time of visible spoilage of the fruit to eleven days (measured from the moment of packaging), where the lack of any absorbent meant spoilage after just five days (grey background). Increasing the active surface of bentonite slightly increased the effectiveness of the ethylene absorber placed in closed banana packages, delaying the first signs of fruit spoilage (brown background). It is worth noting that despite the recorded higher humidity in packages with all tested ethylene absorbers, all of them significantly extended the shelf life of fruit stored in closed packages exposed to unlimited sunlight.

Tab. 1. Results of banana storage in closed packages with full sun exposure

Storage with full sunlight access	Observations / day of storage	1st day	3rd day	5th day	7th day	9th day	11th day
Without ethylene scavenger	*Packaging inflation / <u>humidity inside</u>	- / -	medium / <u>medium</u>	low / <u>medium</u>	- / <u>medium</u>	- / <u>medium</u>	- / <u>high</u>
	Banana colour / <u>surface</u>	vivid yellow / few spots	straw yellow / <u>some greying</u>	pale yellow / <u>bruises & spots</u>	pale yellow / <u>speckles & spots</u>	dark yellow / <u>stains & spots</u>	dark yellow / <u>big brown stains</u>
Ethylene scavenger on bentonite	Packaging inflation / <u>humidity inside</u>	- / -	high / <u>low</u>	high / <u>medium</u>	low / <u>high</u>	low / <u>high</u>	low / <u>high</u>
	Banana colour / <u>surface</u>	yellow / -	vivid yellow / -	yellow / few spots	yellow / few speckles	pale yellow / speckles & spots	yellow / stains & brown edges
Ethylene scavenger on natrified bentonite	Packaging inflation / <u>humidity inside</u>	- / <u>low</u>	high / <u>high</u>	medium / <u>high</u>	medium / <u>high</u>	low / <u>high</u>	low / <u>high</u>
	Banana colour / <u>surface</u>	yellow-green / -	yellow-green / <u>few spots</u>	yellow / <u>few spots</u>	pale yellow / <u>few spots</u>	pale yellow / <u>shadows & spots</u>	yellow-grey / <u>spots & stains</u>
Commercial ethylene scavenger	Packaging inflation / <u>humidity inside</u>	- / <u>low</u>	medium / <u>high</u>	low / <u>high</u>	- / <u>high</u>	- / <u>high</u>	- / <u>high</u>
	Banana colour / <u>surface</u>	yellow / -	yellow / <u>spots</u>	pale yellow / <u>many spots</u>	pale yellow / <u>many spots</u>	yellow-grey / <u>spots & stains</u>	yellow-grey / <u>spots & stains</u>

* The scales of the packaging inflation and humidity inside the packaging are explained in the text of the article. Source: own work.

Limiting the access of packed fruit to sunlight significantly reduced the intensity of the release of water vapour and other gaseous products during banana storage, which resulted in a smaller increase in pressure inside the packaging (Table 2). The oxygen concentration inside the packages changed in a similar way as in the case of fruit stored with unlimited access to sunlight and was correlated with the moment of the appearance of the first signs of fruit spoilage. From this point on, the

falling oxygen concentration begins to increase – most likely due to limited oxygen consumption by the spoiling fruit, which begins to slowly rot.

Tab. 2. Results of banana storage in closed packages with limited sun exposure

Storage with full sunlight access	Observations / day of storage	1st day	3rd day	5th day	7th day	9th day	11th day
Without ethylene scavenger	* Packaging inflation / <u>humidity inside</u>	- / -	- / -	- / -	- / -	- / -	- / -
	Banana colour / <u>surface</u>	yellow / -	pale yellow / <u>first smudges</u>	pale yellow / <u>shadows & spots</u>	yellow-grey / <u>shadows & spots</u>	yellow-brown / <u>big brown stains</u>	yellow-brown / <u>almost all black</u>
Ethylene scavenger on bentonite	Packaging inflation / <u>humidity inside</u>	- / -	- / <u>low</u>	low / -	- / -	- / -	- / -
	Banana colour / <u>surface</u>	yellow / -	yellow / -	dark yellow / <u>small shadows</u>	yellow-grey / <u>spots</u>	yellow-grey / <u>spots</u>	yellow-grey / <u>shadows & spots</u>
Ethylene scavenger on natrified bentonite	Packaging inflation / <u>humidity inside</u>	- / -	medium / -	low / -	low / -	- / -	- / -
	Banana colour / <u>surface</u>	yellow / -	yellow / <u>few spots</u>	yellow / <u>shadows & spots</u>	yellow-grey / <u>shadows & spots</u>	yellow-grey / <u>stains & spots</u>	yellow-grey / <u>stains</u>
Commercial ethylene scavenger	Packaging inflation / <u>humidity inside</u>	- / -	medium / -	low / -	- / -	- / -	- / -
	Banana colour / <u>surface</u>	yellow-green / -	yellow / <u>spots</u>	yellow / <u>many spots</u>	dark yellow / <u>shadows & spots</u>	dark yellow / <u>stains & spots</u>	yellow-grey / <u>stains & spots</u>

* The scales of the packaging inflation and humidity inside the packaging are explained in the text of article.

Source: own work.

The use of ethylene scavengers based on both a commercial absorbent and a KMnO₄-based scavenger applied to bentonite delayed the beginning of packaged fruit spoilage by two days. The bentonite-based absorbent was found to be more effective in slowing the spoilage of packaged bananas,

which was significantly visible after eleven days of storage (Table 2, brown background). Increasing the active surface of the potassium permanganate carrier – bentonite, additionally allowed for greater effectiveness of the absorber in delaying the first signs of spoilage of the packed fruit, which were visible after seven days (from the moment the bananas were packed).

3.2. Efficiency of ethylene absorbers in open packaging

The application of open packaging for banana storage makes it impossible to monitor the composition of the atmosphere inside the packaging due to the unlimited exchange the gases and volatile compounds with the environment. For the same reason, it is not possible to note moisture and bloating of packages as a result of volatile compounds released during fruit ripening and spoilage. On the other hand, right now we can determine the weight loss of stored fruits, which is mainly related to metabolic changes and the loss of volatile products (including water vapour). This is an additional parameter, apart from observing the appearance and colour of bananas, storing the fruits in open packaging with full and limited access to solar radiation (Tables 3 and 4).

Comparing the moment of appearance of the first signs of spoilage in bananas stored in open containers with unrestricted access to sunlight, the addition of any of the tested ethylene absorbers slowed down the process of ripening the fruit by two days (Table 3, grey marking). The absorber based on saturated KMnO_4 bentonite and a commercial absorber allowed to delay the time of fruit spoilage to one week (Table 3, brown background). Increasing the active surface of bentonite through nitrification allowed the shelf life of the fruit to be extended by an additional two days – they were still edible after nine days of storage.

Limiting the access of light to stored fruit extended the shelf life of the fruit, but the first symptoms of overripening were noticed at the same time as for fruit exposed to sunlight. Bananas stored in open packaging without an ethylene absorber but with limited access to light were edible for two days longer than those stored with full exposure to solar radiation. The introduction of ethylene absorbers significantly extended the shelf life of stored fruit from seven days (without the C_4H_4 absorber) to over ten days (Table 4, brown background). The best storage results were obtained for bananas placed in open packages containing an ethylene absorber based on bentonite saturated with potassium permanganate and stored with limited access to sunlight, for which the shelf life of the fruit was over eleven days and the fruit looked quite normal after that time (Figure 3). The fruits protected in this way had an attractive yellow colour and only single brown spots could be seen on their surface.

Tab. 3. Results of changes in the colour and appearance of the surface of bananas during their storage in open packages with full sun exposure

Storage with full sunlight access	Observations / day of storage	1st day	3rd day	5th day	7th day	9th day	11th day
Without ethylene scavenger	Banana colour	yellow-green	pale yellow	pale yellow	yellow-grey	yellow-grey	yellow-grey
	Banana surface	-	shadows & spots	brown plains	many brown plains	many brown plains	many brown plains
Ethylene scavenger on bentonite	Banana colour	yellow	yellow	yellow	yellow	pale yellow	pale yellow
	Banana surface	-	few spots	few brown plains & spots	brown plains	many brown plains	many brown plains
Ethylene scavenger on natriified bentonite	Banana colour	yellow	yellow	yellow	yellow	yellow	dark yellow
	Banana surface	-	-	spots	many spots	many spots & brown plains	many spots & brown plains
Commercial ethylene scavenger	Banana colour	yellow	yellow	yellow	pale yellow	yellow-grey	yellow-grey
	Banana surface	-	few spots	few shadows & spots	spots & brown plains	many brown plains	many brown plains

Source: own work.

Tab. 4. Results of changes in the colour and appearance of the surface of bananas during their storage in open packages with limited sun exposure

Storage with full sunlight access	Observations / day of storage	1st day	3rd day	5th day	7th day	9th day	11th day
Without ethylene scavenger	Banana colour	yellow-green	yellow	pale yellow	pale yellow	pale yellow	pale yellow
	Banana surface	-	few shadows	few shadows	shadows & brown plains	brown plains	many brown plains
Ethylene scavenger on bentonite	Banana colour	yellow-green	yellow	yellow	yellow	yellow	yellow
	Banana surface	-	-	spots	spots	spots	spots
Ethylene scavenger on natrified bentonite	Banana colour	yellow	yellow	yellow	yellow	yellow	yellow
	Banana surface	-	-	spots	spots	spots	spots & brown plains
Commercial ethylene scavenger	Banana colour	yellow-green	yellow	yellow	yellow	yellow	yellow
	Banana surface	-	-	spots	spots	spots	spots & brown plains

Source: own work.

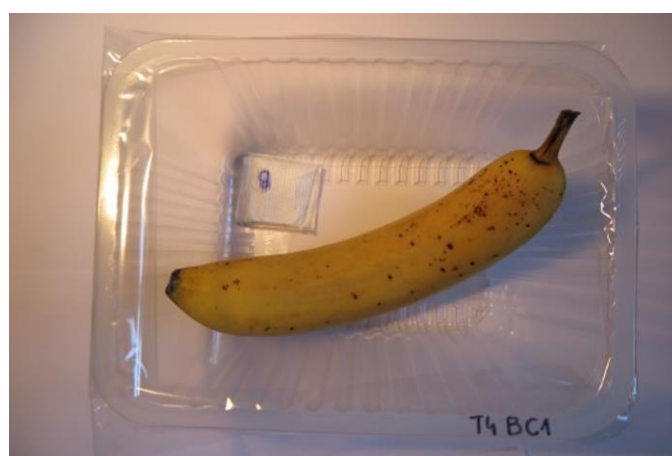


Fig. 3. Example of a banana after 11 days of storage in an open package with a bentonite ethylene absorber and limited access to sunlight

Source: own work.

Figure 4 shows the average daily weight loss of bananas stored in open packages with unlimited access to sunlight (S) and limited one (D). In each case, the introduction of an ethylene absorber slows down the rate of weight loss of stored bananas, which also confirms the impact of this type of active packaging on slowing down the changes taking place in the fruits. Of course, the rate of weight loss (and thus the rate of processes occurring in the fruit) was lower for storage with limited access to sunlight. The introduction of ethylene absorbers will reduce this rate even more significantly. The most effective were ethylene absorbers based on bentonite saturated with KMnO_4 ($0.85\% \pm 0.17\%$) and commercial sachets filling ($0.75\% \pm 0.11\%$).

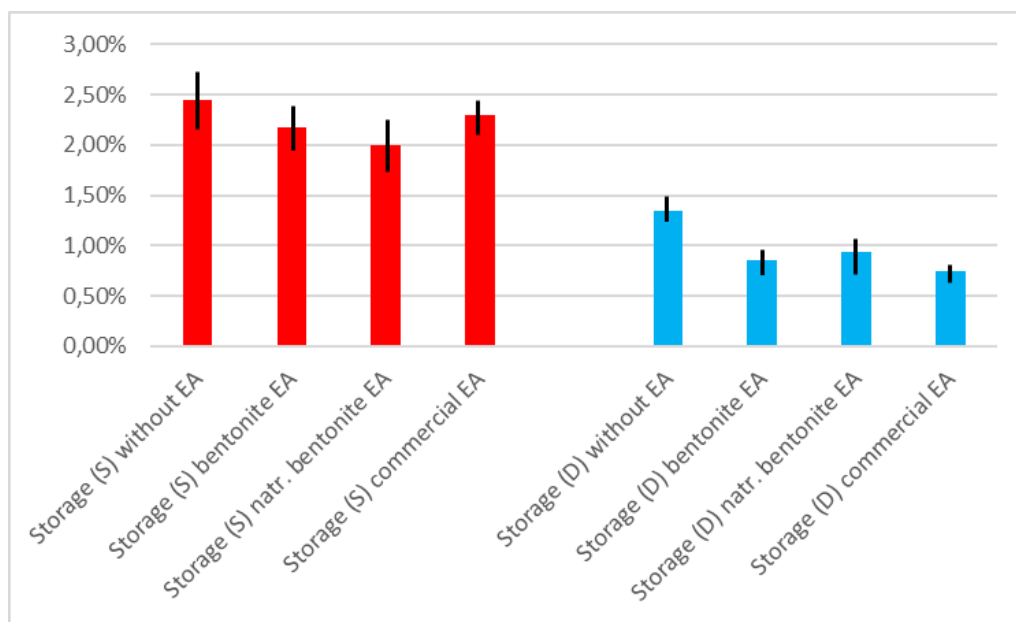


Fig. 4. Average daily weight loss of bananas stored for eleven days in open boxes with or without ethylene absorbers (EA), with full (S) or limited (D) sunlight contact

Source: own work.

Conclusions

The use of ethylene scavengers based on KMnO_4 and bentonite carriers extends the shelf life of bananas under all tested storage conditions in comparison to storage without the absorber and in some cases, they are more effective than the commercial absorber. Natrification of bentonite slightly contributes to the greater effectiveness of the tested absorber and slows down the first signs of fruit spoilage (especially stored in closed packaging). Unlimited access to sunlight has a very large impact on the shelf life of fruits. If they are exposed to intense sunlight, they release more water vapour, lose weight faster, and have a shorter shelf life. The introduction of an ethylene absorber extends the shelf life of the fruits exposed to sunlight. In the case of bananas, the best method of storage turned out keeping them in packaging with free air circulation and limited exposure to daylight. The introduction of ethylene absorbers prolongs the shelf life of the fruit, and the absorbers based on benton-

ite saturated with potassium permanganate were similarly or even better effective than compared commercial absorbers based on activated aluminium beads impregnated with KMnO_4 .

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FOOD SAFETY OF FOOD IMPORTED INTO THE EUROPEAN UNION IN TERMS OF SELECTED MICROBIOLOGICAL HAZARDS

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DOI: 10.56091/CTQS.Qual-7

Abstract

In 2020, a total of 3792 notifications were registered in the RASFF, of which 995 were food infections and of these, as many as 735 were directly related to *Salmonella* contamination of products. In contrast, in 2021, a total of 4664 notifications were registered, of which 985 were microbiological hazards and as many as 715 notifications were linked to the presence of *Salmonella* bacteria.

The aim of this article was to analyse the notifications recorded in the RASFF in 2020 and 2021 regarding the most common *Salmonella* contamination risk. Two parallel criteria were used for evaluation. The first one concerned the frequency of occurrences in notifications, and the second one the effects on human health. Sankey diagrams were used to demonstrate the relationship between the country of origin of the product, the type of product and the actions taken. In addition, a Pareto-Lorenz diagram was used for identification the products with the highest risk to consumers.

Poultry and poultry products were shown to be the most *Salmonella*-contaminated products imported into EU countries in 2020 and 2021. It is worth underlining that they represent the biggest challenge in managing the safety of food consumed by EU citizens. However, it should be borne in mind that notifications registered in the RASFF system may not be an adequate source of data to assess the extent of the microbiological risk.

Keywords: food safety, quality, microbiological hazard, UE

Introduction

The word *safety* describes conditions without care and is derived from Latin. It is commonly understood as a state of certainty, calmness and protection from dangers (Olak & Olak, 2016). Within this concept, to distinguish between the notion of food security, which is matched by the desire to satisfy hunger and the desire to feel secure (Maslow, 2009). According to FAO (*Food and Agriculture Organization*), ensuring food security means a situation in which everyone, at all times, has physical (related directly to its supply), social (related to the dietary preferences of particular social groups, e.g. due to culture or religion) and economic (determined by available financial resources) access to sufficient,

safe as well as nutritious food. According to the FAO, ensuring food safety means certifying that a food will not cause harm to the consumer when prepared and/or consumed as intended. Regularly emerging new food hazards and frauds in both the national and international arena have a significant impact on ensuring food safety. Therefore, safety of food is a particularly important factor in achieving food safety (Obiedzińska et al., 2016; Wiśniewska & Malinowska, 2011).

Currently, food safety is defined as the totality of necessary conditions and actions that must be taken during all stages of the food production and marketing process to ensure the safety of the life and health of consumers (OJ C 202/47; OJ L 338/4).

According to the provisions of the Act of 25 August 2006 food safety is defined as the totality of conditions that must be met, concerning in particular:

- a) the additives and flavourings used,
- b) the levels of polluting substances,
- c) pesticide residues,
- d) the conditions under which food is irradiated,
- e) organoleptic characteristics,

and actions that must be taken at all stages of the production and/or marketing of food – to ensure human health and life (Dz.U. z 2006, Nr 171, poz. 1225, ze zm.).

In turn, at the EU level, food safety policy is governed by Regulation (EC) No 178/2002 of the European Parliament and of the Council of 28 January 2002 laying down the general principles and requirements of food law, establishing the European Food Safety Authority and laying down procedures in matters of food safety (OJ L 031/1).

The increasing number of hazards, including microbiological hazards, associated with food and feed dictates that attention should be paid to increasing responsibility and developing cooperation between Member States across the European Union (Buczowska et al., 2014; OJ L 304/18). Due to the need to implement food safety policies throughout the European Union, a very important tool has been implemented, the RASFF (Rapid Alert System for Food and Feed) (OJ L 261/37). RASFF serves as an efficient exchange of information between the members of the entire network, which includes the European Commission, the European Food Safety Authority (EFSA), EU Member States and EFTA Member States (Norway, Switzerland, Liechtenstein and Iceland). Information on food and feed, food contact materials and information related to the follow-up of identified hazards in products is entered into the system. The creation of such a tool allows Member States to protect their populations and prevent the spread of an identified risk. Members of the network have the opportunity to take

coordinated and coherent action, thus ensuring a very high level of protection for the European Union's population (www.wetgiw.gov.pl; https://food.ec.europa.eu/safety/rasff_en).

Each Member State is obliged to include information on food, feed and food contact materials that are potentially hazardous to human health, animal health or the environment and the follow-up action as a result of identifying the products indicated. Indicated actions may include: stopping the distribution of the product, recalling the product from buyers and blocking the product at the state border. Early response allows all members of the system to monitor on an ongoing basis whether an issue concerns them and whether urgent action is required. Competent authorities in countries where the risk is present are obliged to take immediate measures, e.g. carrying out on-site inspections or withdrawing certain products from the market (OJ L 95/1).

The aim of this article was to analyse the notifications recorded in the RASFF in 2020 and 2021 regarding the most common *Salmonella* contamination risk.

Materials and methods

The subjects of the study were the RASFF notifications of 2020 and 2021 regarding the most frequently reported contamination of food with bacteria belonging to genera *Salmonella* and *Campylobacter* microorganisms. In Europe, *Salmonella* and *Campylobacter* are the most important causes of foodborne illness. The European Centre for Disease Prevention and Control was asserted that aside from campylobacteriosis which had 246,571 reported cases, *Salmonella* was responsible for the highest number of human infections causing illnesses in 91,857 people in the EU in 2018 (Basser, 2018; Ehuwa et al., 2021). A list, published by the World Health Organisation (WHO), of the most dangerous bacteria for human health, which are largely resistant to antibiotics, was used to select the microorganisms analysed. The group of twelve microorganisms was divided into three categories in terms of the degree of risk – critical, high and medium (Table 1).

When deciding on the criterion for analysis, all microorganisms that are not disseminated through food and feed were discarded. Of the microorganisms included in the table 1, genera *Campylobacter*, *Salmonella* and *Shigella* are the main ones that pose a risk associated with food consumption.

Tab. 1. Segregation of microorganisms according to the degree of risk

Degree od danger		
critical degree	high degree	medium degree
<i>Acinetobacter baumannii</i> <i>Pseudomonas aeruginosa</i> <i>Enterobacteriaceae</i>	<i>Enterococcus faecium</i> <i>Staphylococcus aureus</i> <i>Helicobacter pylori</i> <i>Campylobacter</i> <i>Salmonella</i> sp. <i>Neisseria gonorrhoeae</i>	<i>Streptococcus pneumoniae</i> <i>Haemophilus influenzae</i> <i>Shigella</i> sp.

Source: own elaboration based on a list of the 12 most dangerous bacteria against which antibiotics do not work, published by the WHO.

As *Shigella* did not appear in the RASFF notifications in the two years indicated, it was therefore not considered further in the study. Hence, *Salmonella* was selected for further analysis as the bacterium most frequently appearing in the notifications and *Campylobacter* as the bacterium with the greatest potential negative impact on human health.

The data required for the analyses, came from the publicly accessible RASFF Window portal. In a first step, it was possible to retrieve the total number of registered notifications. In a second step, the search criteria were restricted to microbiological hazards only. For the period under study, a risk-specific filter was selected by including only “non-pathogenic micro-organisms” and “pathogenic organisms”.

Tables and a Sankey diagram (Power BI) were used to present the results obtained, collating data related to the origin of the products reported, the product group and the consumer protection measures taken against them.

Result and discussion

In 2020, the RASFF received 995 notifications for non-pathogenic and pathogenic microorganisms, while 2021 saw slightly fewer notifications – 985 (Table 2).

Tab. 2. Numer and percent of notifications per year in 2020 and 2021

Microorganism	Number / percent of notifications per year	
	2020	2021
<i>Salmonella</i> sp.	735 / 74%	715 / 73%
<i>Listeria</i> sp.	114 / 11%	120 / 12%
<i>Escherichia coli</i> sp.	74 / 7%	81 / 8%
<i>Norovirus</i>	35 / 4%	14 / 1%
<i>other, such as Vibrio</i> sp.	29 / 3%	81 / 8%
<i>Bacillus</i> sp.	10 / 1%	9 / 1%

Source: own elaboration based on data extracted from the RASFF system in 2020 – 2021.

In 2021, as in 2020, the highest number of RASFF notifications related to food contamination with *Salmonella* sp.. The number of notifications related to other microorganisms remained relatively constant between 2020 and 2021, relatively lower than for *Salmonella* genus.

Salmonella is a Gram-negative rods genus belonging to the Enterobacteriaceae family. Within 2 species, *Salmonella bongori* and *Salmonella enterica*, over 2500 different serotypes or serovars have been identified to date. *Salmonella* is a ubiquitous and hardy bacteria that can survive several weeks in a dry environment and several months in water (www.who.int). Salmonellosis is regarded as a foodborne infection of the gastrointestinal tract and has been reported to have high incidence rates. Salmonelle is the leading cause of foodborne outbreaks in Europe (Fawzy & Abdelrazeg, 2024). Salmonellosis is the second most reported gastrointestinal disorder in the EU resulting from the consumption of *Salmonella*-contaminated foods (Basser, 2018; Ehuwa & Jaiswal & Jaiswal, 2021). Usually the bacteria reside in the intestines and are excreted in the faeces. The source of infection is usually water or food. This pathogenic bacteria is common and resistant to environmental factors. It can survive for several weeks in a dry environment and even several months in water. Symptoms of infection include high fever, abdominal pain, nausea and diarrhoea and vomiting. These are fairly mild and most patients do not require specialised treatment. In the case of weakened people, e.g. children, elderly people, poisoning can be dangerous. The dehydration that occurs can be life-threatening. The onset of symptoms is observed within 6–72 hours of ingestion of infected food and the illness itself lasts an average of 2–7 day (Dźygóra, 2020). In 2020, 5468 cases of salmonellosis per 100,000 population were reported. In 2021, as many as 8294 (per 100,000 population) *Salmonella* infections were confirmed, an increase of 51% on the previous year (NIH, 2021; NIH, 2022). The lower number of recorded *Salmonella* infections in 2020 may have been influenced by the COVID-19 virus pandemic, which, on the one hand, raised people's awareness of hygiene and forced citizens to observe sanitary rigour. On the other hand, the state busy fighting the pandemic was focused on the diagnosis of coronavirus (EFSA 2021). Some symptoms of salmonellosis may have been erroneously attributed to coronavirus and further testing for salmonellosis was not performed, which may have translated into fewer recorded cases of infection with the microorganism (D'Amico et al., 2020).

In 2020, a total of 3,792 notifications were registered in the RASFF, of which 995 were related to food and feed contamination with micro-organisms. Of the 995 notifications, 735 were directly related to *Salmonella* contamination of products with bacteria form *Salmonella* genus. In contrast, in 2021, a total of 4664 notifications were registered in the system, of which 985 were microbiological hazards of food and feed products. Of the 985 notifications, 715 were related to the presence of *Salmonella* sp.

Table 3 presents notifications related to *Salmonella* infection in 2020 and 2021 by group of infected products, together with the number of notifications recorded within groups.

Tab. 3. Number of notifications recorded within each *Salmonella*-infected product group

Group of products infected by <i>Salmonella</i> sp.	Number of notifications recorded in the year	
	2020	2021
Poultry and poultry products	392	330
Prepared meals and snacks	1	1
Other food products/mixes	4	5
Eggs and egg products	12	7
Animal food	9	13
Bivalve molluscs and related products	5	9
Feed mixes	2	4
Meat and meat products (other than poultry)	42	55
Milk and dairy products	3	5
Nuts, nut products and seeds	62	38
Fruits and vegetables	19	5
Breakfast cereals and bakery products	1	4
Fish and derived products	4	3
Animal feed ingredients	87	77
Crustaceans and products derived therefrom	1	2
Confectionery products	2	3
Herbs and spices	88	145
Dietetic foods, food supplements and fortified foods	1	6

Source: own elaboration based on RASFF data from 2020 and 2021.

Analysing the results, it was found that the group with the highest number of notifications in 2020 was: poultry and poultry products. Other groups with a high number of notifications, there were: herbs and spices, animal feed ingredients, nuts, nut products and seeds, and eggs and egg products. In 2021, as in 2020, the poultry and poultry products group had the highest number of notifications. Other groups with a high number of notifications were: herbs and spices, animal feed ingredients, meat and meat products (other than poultry), and nuts, nut products and seeds. Most notifications were related to *Salmonella* contamination.

Figures 1 and 2 show the countries with the highest number of notifications to the system in 2020–2021 (more than 25 notifications).

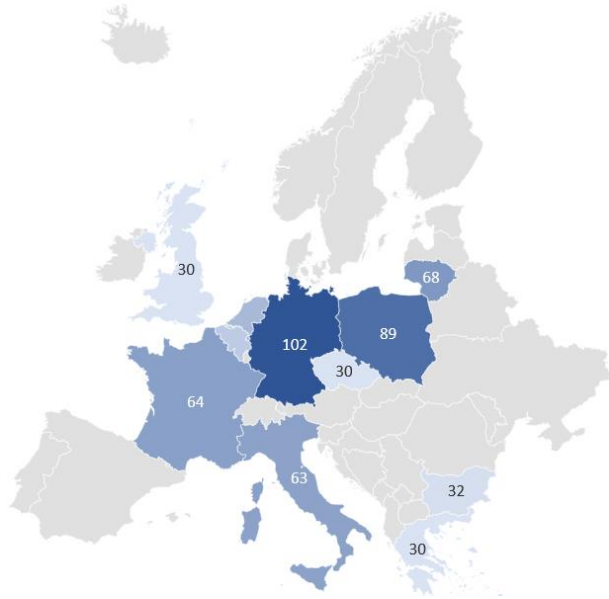


Fig. 1. Number of RASFF notifications by reporting country in 2020
 Source: own elaboration based on RASFF data from 2020.

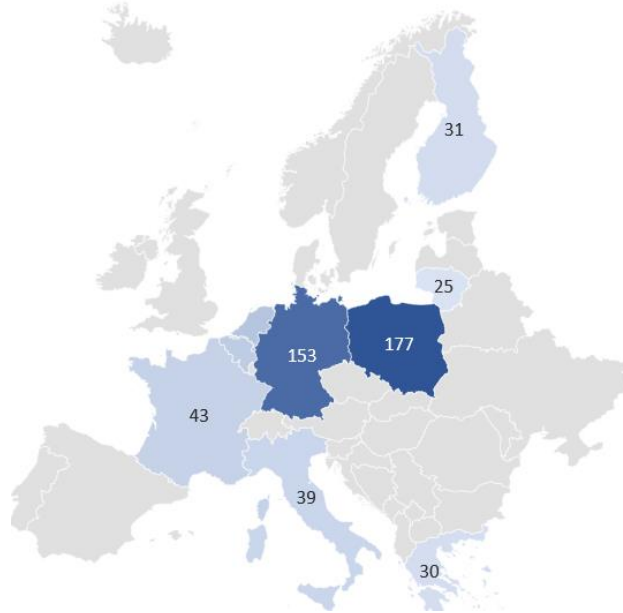


Fig. 2. Number of RASFF notifications by reporting country in 2021
 Source: own elaboration based on RASFF data from 2021.

In 2020, Poland mainly notified products of its own origin (87% of all notifications). The remaining notifications concerned isolated cases originating from countries such as India (4), Germany (2), Egypt (2), Bulgaria (1), or Spain (1). The notifications mainly concerned poultry and poultry products (71), eggs and egg products, herbs and spices and feed ingredients. In 2021, on the other hand, main-

ly own-origin products were also reported (151). The remaining products concerned individual cases and came from, among others: India (2), Brazil (2), Spain (2), Ukraine (1), Egypt (1). The notifications mainly concerned poultry and poultry products (155), meat and meat products (other than poultry) (7), eggs and egg products (5), nuts, nut products and seeds (4), herbs and spices (3), feed ingredients (3).

However, it should be borne in mind that not every product imported into the European Union is checked. Presumably, the risks detected are only a percentage of the total number of products. Given the volume of goods that are imported into the EU, it would not be possible to inspect every single one of them. The European Union, in economic terms, ranks third as an importer of agri-food products (after China and the United States). In 2020, the value of imports of these products was €122 billion (www.wwf.eu.awsassets.panda.org), of which imports of agri-food products to Poland were valued at €23.5 billion. In 2020 alone, 146,000 tonnes of poultry products arrived in the EU (www.oec.world/en), and this was just one of many types of imported agri-food products. In view of this significant trade in commodities and the limited control capacities of the RASFF Member States, 3792 notifications of detected hazards were made among the products tested in 2020. This number represents a significant proportion of notifications in relation to the number of products examined. On the basis of only a fraction of the tested commodities, it is difficult to make a reliable assessment of the actual extent of contamination of food that enters the RASFF countries, as well as of food safety for the consumers of these countries.

Figure 3 shows a comparison of the number of notifications related to the origin of *Salmonella*-infected products in 2020 and 2021.

Poland, is the country with the highest number of *Salmonella*-infected products reported under the RASFF in both 2020 and 2021. In all countries, the recorded number of risks remained at a similar level, with an average of 28 notifications. Brazil is the only country where the number of recorded hazards in 2020 increased significantly compared to 2021 (an increase of 49%).

In the case of Brazil, it is difficult to find data relating directly to 2020 and 2021, but the information obtained indicates a general problem of related to food safety in terms of *Salmonella* detection. In 2018, the Brazilian Ministry of Agriculture notified of adulteration of *Salmonella* test results. These activities were carried out by one of the largest food organisations worldwide, BRF. The adulteration was carried out for several years, from 2012 to 2015. In response to these actions, the European Union decided to carry out meticulous checks on all meat imported from the country. Subsequently, the European Union banned the import of meat from Brazil in general, and complaints from BRF and SHB were dismissed due to numerous detections of the microorganism in imported meat. It is im-

portant to remember that food entering the EU market from a third country must meet identical requirements to food produced within the EU. Acceptable levels of, for example, micro-organisms in meat in Brazil are much higher than in EU territories.

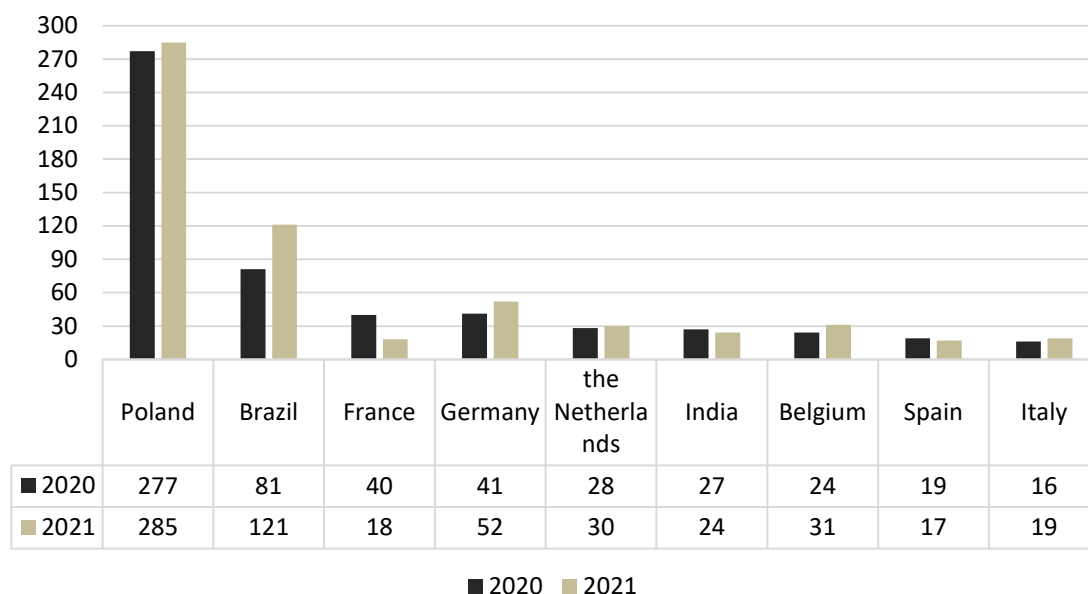


Fig. 3. Number of notifications to the RASFF by reporting country in 2020–2021

Source: own compilation based on RASFF data from 2020 and 2021.

Tab. 4. Number of RASFF notifications attributed to each type in 2020 and 2021

Notification classification	Number of notifications in 2020	Number of notifications in 2021
Information notification for follow-up	234	210
Alert notifications	211	205
Border rejections notification	159	173
Information notification for attention	130	127
News – any information related to the safety of food and feed products which has not been communicated as an alert or an information notification, but which is judged interesting for the control authorities, is transmitted to the RASFF members under the heading News.	1	0

Source: own compilation based on RASFF data from 2020 and 2021.

Table 4 summarises the types of notifications reported to the RASFF and the volumes recorded under the scheme in 2020 and 2021. In both 2020 and 2021, the highest number of notifications was attributed to notifications for attention. Alert notifications came in second place. The lowest number of notifications was associated with notifications of the News type, with only one reported in 2020 and

none in 2021. On the one hand, a significant number of information notifications were indicated for attention. These are non-threatening and are intended to present a problem, but they do not require immediate action. Usually, the notified products were no longer available on the market or remained in the area of the country that notified them, allowing the spread of the risk to be narrowed down to a specific Member State. On the other hand, there were notifications that compelled immediate action from Member States. In third place were notifications related to related to border rejections.

Actions taken in the framework of the RASFF system operation for selected product groups and country

Figures 4 and 5 show the product groups reported to the RASFF in 2020 and 2021 respectively, their origin and the actions taken to eliminate the *Salmonella* risk.

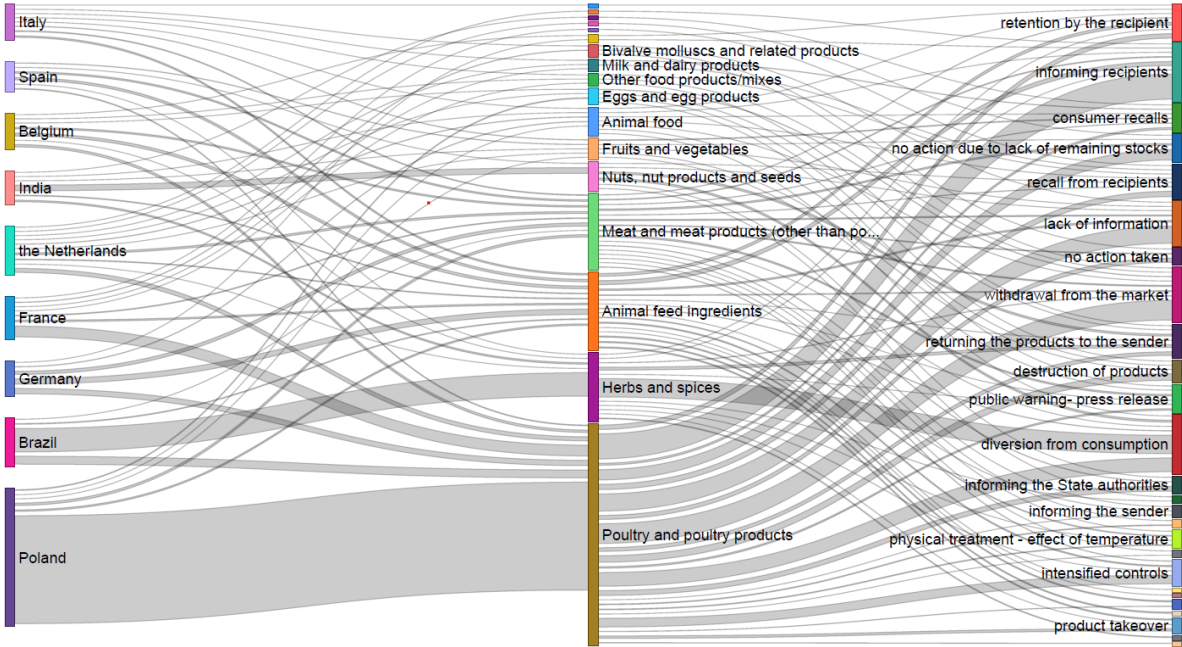


Fig. 4. Structure of RASFF notifications and action taken against them in 2020

Source: own compilation based on RASFF data from 2020 and 2021.

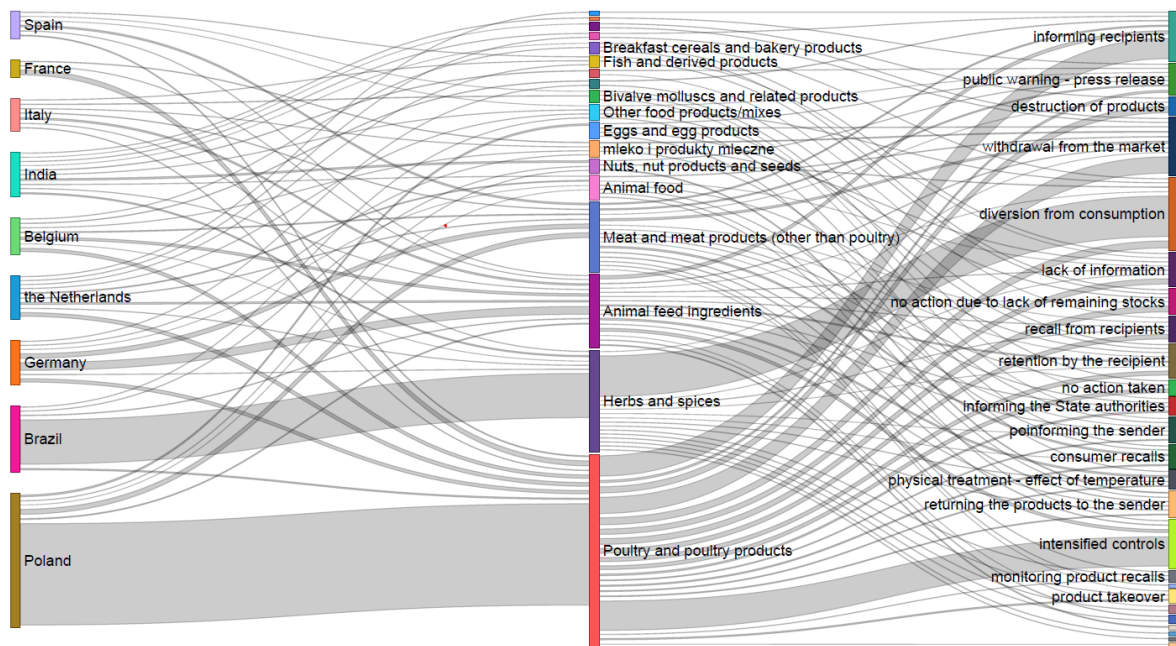


Fig. 5. Structure of RASFF notifications and action taken against them in 2021

Source: own compilation based on RASFF data from 2020 and 2021.

Sankey diagrams show the origin of selected product groups and the actions taken against these products to ensure the safety of RASFF countries' consumers. The nine countries from which the products most frequently reported to the system originated are shown in the diagrams. From the selected countries, all reported product groups and the actions taken against them have been analysed.

In 2020, the most common actions taken to protect the health and lives of consumers against food contaminated with bacteria from *Salmonella* genus were: diversion from consumption, informing recipients, and withdrawal from the market. Unfortunately, in many cases the notification did not include information on the action taken. In several cases, no action was taken at all. In contrast to data from 2021, the most common actions taken to ensure consumer safety against food infected with bacteria form *Salmonella* genus were: to remove products from consumption, to introduce increased controls, to recall products from the market and to inform consumers of the incident. Actions that have been taken against poultry and poultry products most frequently reported in RASFF notifications are shown in Table 5.

Tab. 5. Actions against the group of poultry and poultry products most frequently reported in RASFF notifications in years 2020 and 2021

Action	Number of actions in 2020	Number of actions in 2021
no information was provided on the steps taken	62	15
informing recipients	61	52
withdrawal from the market	48	42
push back from consumption	34	19
no action due to lack of remaining stocks	26	16
intensified controls	22	74
destruction of products	17	17
recall from recipients	14	13
informing the State authorities	13	6
retention by the recipient	12	12
no action was taken	11	11
returning the products to the sender	10	4
product takeover	7	5
consumer recalls	6	5
public warning – press release	6	7
physical treatment – effect of temperature	3	4
monitoring product recalls	3	1
chemical treatment	2	-
release to the market	2	2
informing the sender	1	4

Source: own compilation based on RASFF data from 2020 and 2021.

The likely reason for the non-record information was the late notification of the message to the RASFF system. By the time of the notification, all suspicious products could have been effectively removed. At the same time, it is surprising to see the large number of notifications in which no information about the action taken was included. A recipient looking for data could speculate that this is the same as no action taken. Within a system that is intended to be a tool to help ensure food safety, every action described is relevant to assess the validity of its operation. It is worth noting that the number of notifications with missing action taken information has decreased from 61 notifications in 2020 to 15 notifications in 2021, which is a positive development. It is also important to look at notifications where absolutely no action was taken, what the rationale was for doing so and whether the failure to act may have affected consumer health.

Conclusions

The aim of this article was to determine to what extent microbiological food hazards pose a challenge to ensuring consumer safety based on the operation of the RASFF system. The most frequently notified microbiological risk caused by *Salmonella* was analysed. Bearing in mind that only a proportion of incoming food and feed into the EU is tested, the number of risks detected in 2020 was 3792, of which 735 were related to the detection of *Salmonella* bacteria. In the following year, there were 4664 notifications, of which 715 were related to the detection of *Salmonella* bacteria in tested products. These figures indicate the presence of a microbiological risk that, on the scale of all food and feed products arriving, may be even higher than what we can assess from the RASFF notifications alone.

The analysis of the RASFF data from 2020–2021 allowed the following conclusions to be drawn:

A significantly higher number of notifications of detected hazards were recorded in 2021 than in the previous year. It was suspected that following the notifications recorded, actions would be implemented in 2020 leading to a reduction in the number of detected hazards in tested commodities, which would promote increased consumer food safety. The number of notifications to the system increased, however, this did not affect the types of alerts recorded. In 2020–2021, invariably the largest number of alerts was for information notifications for attention, alerts and those related to border rejections.

The maintenance of the number of product notifications in the years analysed may have been due to the period of the COVID-19 pandemic. Many countries were involved in coronavirus infection control activities in 2020 and 2021, which unfortunately may have had the effect of sidelining food safety activities.

In both 2020 and 2021, the highest number of notifications related to the detection of bacteria belonging to *Salmonella* genus came from Poland. Poultry and poultry products were the most frequently notified group. The fact that Poland self-reported *Salmonella* detections in its country is considered a symptom that is conducive to consumer health and life. Brazil came second, with herbs and spices (mainly black pepper) being the most frequently reported product group. This means that certainly compliance with EU requirements by third countries and a reduction in food imports from non-compliant countries could improve food safety.

Notifications registered under the RASFF may not be an adequate source of data for assessing the extent of the microbiological risk. This could be confirmed by the epidemiological situation related to *Campylobacter*. The number of notifications of the system did not indicate the presence of a risk

related to the spread of this bacterium in food and feed, yet the data published by EFSA and ECDC confirmed the continuously high number of cases of campylobacteriosis, the source of infection being mainly food.

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SAFETY OF FOOD LABELLING – THE ANALYSIS OF RASFF NOTIFICATIONS ON FAULTY LABELLING

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DOI: 10.56091/CTQS.Qual-8

Abstract

Food labelling is perceived as communication channel between the food system, producers and consumers, in the absence of a face-to-face encounter. Labels should be designed to provide accurate information so that consumers can make informed food choices, both taking into consideration nutritional aspects and safety matters.

The objective of this work was to analyze the European notifications in Rapid Alert System for Food and Feed (RASFF) portal related to faulty labelling issued in the period from 01/01/2020 to 31/07/2023. The following data were extracted and analyzed: date of notification, notifying country, country of origin, product category, subject, notification basis, notification type, risk decision.

A total of 647 notifications on faulty labelling in food products were published in the analyzed period, among which 79.3% related to allergens, and 20.7% concerned other labelling deficiencies. Cereals and bakery products were the most reported category for undeclared allergens and among 14 groups of allergens notifications mostly concerned milk (including lactose) (19.3%) and cereals containing gluten (12.7%). Other labelling deficiencies were mostly found in the category of dietetic foods, food supplements and fortified foods.

Keywords: food label, food safety, RASFF, allergens

Introduction

The consumer spends as little as 12 seconds on one food item in a supermarket (Osman & Jenkins, 2021). Despite that, the food product packaging and labelling seems to play a crucial role in shaping consumer interest in certain product, impacting confidence in the quality of a product and influencing purchase decision. Food labelling is perceived as communication channel between the food system, producers and consumers, in the absence of a face-to-face encounter. Labels should be designed to provide accurate information so that consumers can make informed choices. Recently, the consumer demand for detailed, accurate and accessible information on food quality and safety has greatly increased. Consumers seek to make healthy food choices by reading the information content

on product packaging including nutrition information panels, list of ingredients, environmental and warning labels and claims. Consequently, food labelling and consumers' label information search (LIS) have emerged as important aspects of the consumer behaviour literature (Drichoutis et al., 2006).

The trust in labels seems to be of great importance. The model presented by Tonkin et al. (2015) suggest that the factors that influence trust in labels are often driven by the attributions of trust in regulatory bodies, food industry actors, brands, and topical news stories concerning food scares and scandals. The authors indicate that trust in the labels (trust in the literal message being communicated by the label) impacts trust through the labels (trust in system/ governance/ actors). As a result, food labelling reinforces confidence and trust in food regulatory and supply systems. Food labelling is a key issue to guarantee consumers their right to information, that is why labelling regulations and legislations should be accurate and transparent. Regulation (EU) No. 1169/2011 laid down rules on food labelling applicable to all foods, in particular, it drew up a list of mandatory information which should, in principle, be provided. Other EU regulations introduced the possibility to place voluntary information on food packaging such as health claims, nutrition claims or other nutritional statements. Both mandatory and voluntary information or their lack may influence perceived safety and thus shape buying behaviour.

As mislabeling may impose public health risk, there is a need to control and communicate products with faulty labels. The European Commission created RASFF (Rapid Alert System for Food and Feed) database to make information about any food, feed or food contact material that is unsafe, hazardous or does not comply with labeling or information supplied with the product available to consumers, business operators and authorities worldwide. The RASFF portal is therefore a key tool to ensure the flow of information to enable a swift reaction when risks to public health are detected in the food supply chain, also those concerning faulty labelling. However, using the RASFF database both as a predictive tool and for trend analysis should be made with caution as iterative changes in food law impact on the frequency of regulatory sampling associated with border and inland regulatory checks (Kowalska & Manning, 2021), which, in turn, may significantly increase the number of notifications in certain time period (Pádua et al., 2019).

The objective of this work was to analyze the European notifications in RASFF portal related to faulty labelling (related to allergens and other labelling deficiencies) in the period from 01/01/2020 to 31/07/2023.

Materials and methods

The data was extracted directly from the RASFF platform in .xls format on 1st August 2023, through the existing functionality for this purpose. The analysis concerned notifications in food products published between 1st January 2020 and 31th July 2023. Notifications related to undeclared allergens were searched in RASFF database under the hazard category “allergens” and by keyword “allergen” in the subject field. Notifications related to other labelling deficiencies were extracted from RASFF database under the hazard category “labelling absent/incomplete/incorrect” and by keyword “label”. Retrieved notifications that referred to allergens were moved to database concerning allergens and possible recaps were removed for further data processing.

The collected data provided information on the: date of notification, notifying country, country of origin, product category, subject, notification basis, notification type, risk decision. The database gave a detailed description of the product involved in the notification. However, no commercial details such as brands and business operators were obtained. Each notified product was classified within one of the food categories established by the RASFF system: alcoholic beverages; bivalve mollusks and products thereof; cephalopods and products thereof; cereals and bakery products; cocoa and cocoa preparations, coffee, and tea; confectionery; crustaceans and products thereof; dietetic foods, food supplements, and fortified foods; eggs and egg products; fats and oils; fish and fish products; food additives and flavorings; fruits and vegetables; herbs and spices; ices and desserts; meat and meat products (other than poultry); milk and milk products; natural mineral water; non-alcoholic beverages; nuts, nut products, and seeds; other food products/mixed; poultry meat and poultry-meat products; prepared dishes and snacks and soups, broths, sauces, and condiments.

Basing on the description of the notifications further analysis encompassed the division of notifications according to the specific allergen concerned, according to the list of 14 substances or products causing allergies or intolerances (hereinafter referred to as “allergens”) listed in Annex II of Regulation (EU) No. 1169/2011. All data were analyzed in Microsoft Excel 2010.

Results and discussion

RASFF notifications on allergens

A total of 513 RASFF notifications were published on allergens in the period from 01/01/2020 to 31/07/2023. In consecutive years, the number of notifications amounted to 137 in 2020, 132 in 2021, 145 in 2022, while in the first seven months of 2023 already 99 notifications were published. The increase in notifications in recent period could be due to adoption of the global standard

CXC 80-2020 through entering into force Commission Regulation (EU) No. 2021/382 amending the Annexes to Regulation (EC) No. 853/2004 of the European Parliament and of the Council on the hygiene of foodstuffs as regards food allergen management, redistribution of food and food safety culture. The regulation introduced requirements introducing good hygiene practices to prevent or limit the presence of substances causing allergies or intolerances at the level of primary production and at stages beyond that production.

There are two types of labelling with respect to food allergens – precautionary and mandatory (Allen et al., 2014). Precautionary allergen labelling (PAL), also referred to as advisory labelling, is voluntary and highlights to the consumer that one or more regulated allergens could be unintentionally (for example due to cross contamination), but unavoidably, present in a product, and thus pose a risk to susceptible consumers (for example “may contain milk”). For mandatory allergen labelling (MAL), food business are required to highlight (for example in underline and/or bold) the contents of packaged food that contain any of 14 allergens. The RASFF database does not automatically differentiate if the product unfulfilled PAL or MAL requirements. In-depth analyses of notifications in the period from 01/01/2020 to 31/07/2023 showed that allergens were either undeclared in the ingredients, or possible traces of allergen were undeclared, or presence of allergens as contaminants was determined, or content of allergens was too high in allergen-free products. All of the reasons are connected with the faulty labelling and have possibility of posing a health threat. In some cases, the information about allergens was not in proper language, which is non-compliant with requirements of EU Regulation 1169/2011.

Five countries were notifying countries in 65.3% of cases. Netherlands was notifying country in 22.4% of the published cases, followed by Germany (13.8%), Belgium (11.1%), Italy (9.6%), and Sweden (8.4%) (Table 1). The results are corresponding with the data presented by Martinez-Pineda and Yagüe-Ruiz (2022) who showed that the main countries that emitted notifications between 1 January 2018 and 31 December 2021 about allergen hazards were Belgium, the Netherlands, and the United Kingdom. It should be noted that starting from January 2021 the RASFF portal did not report notifications issued in the United Kingdom since it was no longer part of the EU.

The products in question originated in 28 RASFF member countries and 36 RASFF non-member countries (including United Kingdom). The country of origin of products in question in 16.7% was Netherlands. Products from Belgium and Germany were a bit less often notified, in 11.1% and 8.7% cases respectively. Polish products originated in 4.0% of notifications.

Tab. 1. RASFF notifications on allergens in the period from 01/01/2020 to 31/07/2023 by year and notifying country

Notifying country	Year and number of notifications				Total		
	2020	2021	2022	2023 ¹	No.	%	
Austria	1	0	1	3	5	1.0	
Belgium	21	11	16	9	57	11.1	
Croatia	1	0	2	0	3	0.6	
Cyprus	2	2	5	1	10	1.9	
Czech Republic	5	2	3	3	13	2.5	
Denmark	2	6	5	7	20	3.9	
Finland	2	4	5	2	13	2.5	
France	0	1	2	5	8	1.6	
Germany	16	15	25	15	71	13.8	
Greece	0	1	0	0	1	0.2	
Hungary	1	1	1	2	5	1.0	
Ireland	4	1	4	0	9	1.8	
Italy	11	13	16	9	49	9.6	
Latvia	1	0	0	0	1	0.2	
Lithuania	0	1	1	0	2	0.4	
Luxembourg	0	0	0	1	1	0.2	
Netherlands	34	30	27	24	115	22.4	
Norway	0	1	1	0	2	0.4	
Poland	1	4	1	0	6	1.2	
Romania	1	0	0	0	1	0.2	
Slovakia	1	0	3	2	6	1.2	
Slovenia	0	3	0	1	4	0.8	
Spain	6	15	12	5	38	7.4	
Sweden	7	16	11	9	43	8.4	
Switzerland	0	1	3	0	4	0.8	
United Kingdom	19	0	0	0	19	3.7	
European Commission	1	4	1	1	7	1.4	
Total	No.	137	132	145	99	513	100.0
	%	26.7	25.7	28.3	19.3	100.0	

¹until 31/07/2023

Source: own compilation based on RASFF data from 2020 to 2023.

Notifications on allergens were mostly related to the following product categories: cereals and bakery products in 16.4% of cases, prepared dishes and snacks in 14.8% of cases and other food product/ mixed in 13.5% of cases (Figure 1). Fewer notifications concern confectionery (7.2% of cases), soups, broths, sauces and condiments (7.0%), meat and meat products (other than poultry) (6.2%) and fruits and vegetables (6.0%). In general, those categories that include food products with a higher degree of processing and number of ingredients proved to be the ones that collected the highest number of notifications. Products from categories such as bivalve molluscs and products thereof and eggs and egg products were notified only once.

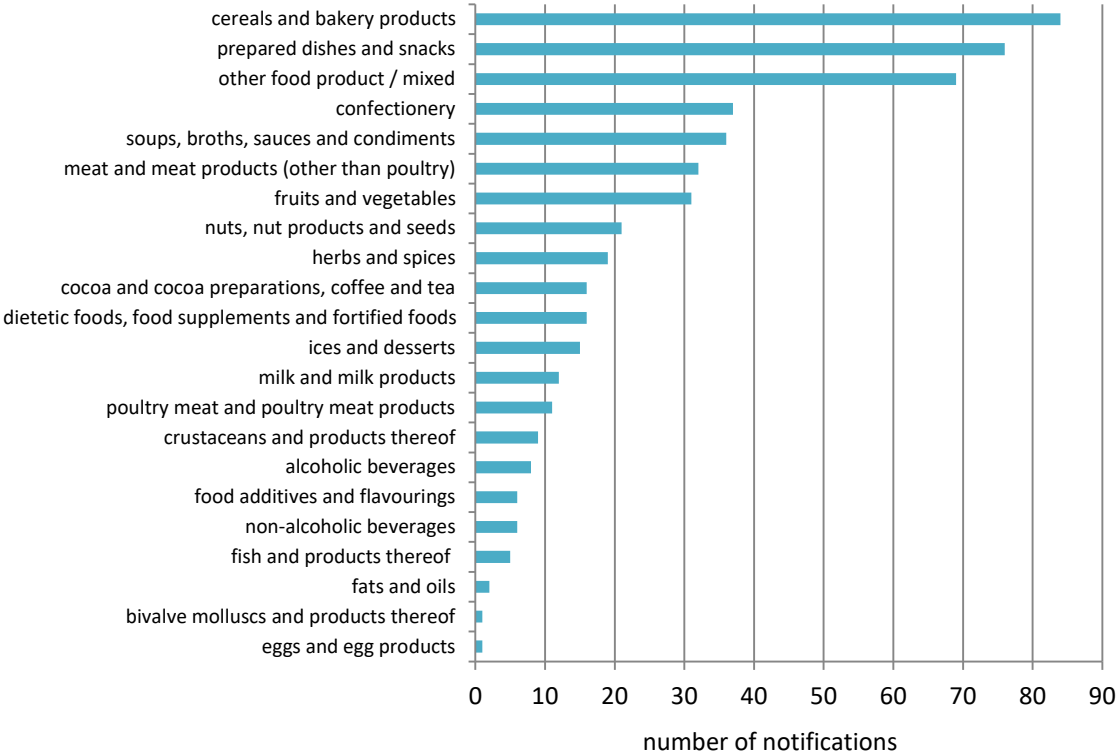


Fig. 1. RASFF notifications on allergens in certain product categories in the period from 01/01/2020 to 31/07/2023

Source: own compilation based on RASFF data from 2020 to 2023.

In comparison, Friganović et al. (2022) analyzed 160 notifications on the presence of allergens between 2015 and 2019 year only in cereals and cereal-based products. Each year between 25 and 39 cases were reported for cereal products, while in above analysis 84 notifications concerning cereal and bakery products were reported (in 2020 – 23 cases, 2021 – 30, 2022 – 18, in the seven months of 2023 – 13). It confirms decrease in the amount of notifications on allergens in recent years in this product category.

The most common allergens in RASFF notifications on allergens in the period from 01/01/2020 to 31/07/2023 were milk and cereals containing gluten in 19.3% and 12.7% of cases, respectively. Nuts and soybeans took third and fourth position with 9.8% and 9.6% notifications (Table 2). In some food products in question more than one allergen was an issue. Mostly notified allergens overlapped with the global priority list proposed by Expert Committee who reached a consensus on the importance of prevalence, potency and severity factors of certain allergens. However, soybean was recommended by Expert Committee to be removed from the global list of priority allergenic food, indicating that it may be kept on a list of allergens for regional consideration (FAO & WHO, 2022).

Tab. 2. Allergens and mostly notified product categories in RASFF notifications in the period from 01/01/2020 to 31/07/2023

Allergens	Total		Mostly notified product categories ¹
	No.	%	
Milk (including lactose)	117	19.3%	prepared dishes and snacks (4.1%), cereals and bakery products (3.0%), other food product / mixed (2.5%), confectionary (1.8%)
Cereals containing gluten	77	12.7%	cereals and bakery products (3.5%), prepared dishes and snacks (2.6%), other food product / mixed (1.7%)
Nuts	59	9.8%	cereals and bakery products (2.3%), confectionary (1.5%), nuts, nut products and seeds (1.3%)
Soybeans	58	9.6%	cereals and bakery products (2.5%), prepared dishes and snacks (2.0%), other food product / mixed (1.5%)
Egg	46	7.6%	prepared dishes and snacks (1.7%), other food product / mixed (1.5%), cereals and bakery products (1.3%)
Sulphur dioxide and sulphites	44	7.3%	fruits and vegetables (3.3%), crustaceans and products thereof (1.0%)
Peanut	38	6.3%	cereals and bakery products (1.3%), nuts, nut products and seeds (1.0%)
Mustard	36	6.0%	meat and meat products (other than poultry) (1.3%), herbs and spices (1.2%), soups, broths, sauces and condiments (1.2%)
Celery	32	5.3%	other food product / mixed (1.3%), meat and meat products (other than poultry) (1.0%), prepared dishes and snacks (1.0%)
Fish	15	2.5%	prepared dishes and snacks (1.2%), other food product / mixed (0.7%)
Crustaceans	14	2.3%	prepared dishes and snacks (0.7%), dietetic foods, food supplements and fortified foods (0.5%)
Sesame seeds	14	2.3%	soups, broths, sauces and condiments (0.5%)
Lupin	6	1.0%	prepared dishes and snacks (0.3%)
Molluscs	2	0.3%	prepared dishes and snacks (0.3%)
Undeclared	47	7.8%	prepared dishes and snacks (1.8%), cereals and bakery products (1.1%), other food product / mixed (1.1%)
Total	605		

¹percentage of all notifications

Source: own compilation based on RASFF data from 2020 to 2023.

Undeclared milk (including lactose) and cereals containing gluten were mostly present in prepared dishes and snacks and cereals and bakery products. The obtained results are worrying since products from both categories are likely to be frequently consumed by children and adolescents, and at the same time contain the most prevalent allergens for this population (Martínez-Pineda & Yagüe-Ruiz, 2022). The presence of undeclared nuts and peanuts was high in nuts, nut products and seeds products, which can be easily explained due to cross-contamination. The undeclared peanuts were found in cashew paste, almond paste and nut mix, while undeclared nuts such as cashew nuts, macadamia nuts, pistachio, almonds were notified in other nut spreads and pastas. In fruits and vegetables category prevailed notifications related to sulphur dioxide or sulphites. In many cases, the undeclared allergen was not defined (8.4%).

The majority of RASFF notifications on allergens in the analyzed period were classified as alert notifications (80.9%), while 13.6% of cases were regarded as information notification for attention (Table 3). Only 1.8% of notifications were qualified as border rejection. 92.4% of total number of notifications was of serious risk.

Tab. 3. Types of RASFF notifications on allergens and labelling deficiencies in the period from 01/01/2020 to 31/07/2023 [in %]

Notification type	Notification subject	
	allergens	other labelling deficiencies
alert notification	80.9	19.4
border rejection	1.8	37.3
information notification for attention	13.6	13.4
information notification for follow-up	3.5	29.9
news	0.2	0.0

Source: own compilation based on RASFF data from 2020 to 2023.

RASFF notifications on other labelling deficiencies

A total of 134 RASFF notifications were published due to labelling deficiencies (other than related to allergens) in food products in the period from 01/01/2020 to 31/07/2023. The number of notifications increased between the years – in 2020 20 notifications were reported, in 2021 – 29, in 2022 – 47, while in the first seven months of 2023 – already 38. However, it must be noted that not all notifications related to faulty labelling are available in RASFF database. As established by Regulation 2019/1715 on the Information Management System for Official Controls (the IMSOC Regulation) notifications concerning non-compliance of a food, food contact material or feed that does not pre-

sent a public health risk (so called non-compliance) are notified by Administrative Assistance and Cooperation system network (AAC) members. In 2022 AAC notifications have reached the number of 2 554, among which faulty labelling or claims were the top reported type of violation (35.2%). The cases concerned labels in which the mandatory information to consumers was non-compliant or lacking (name of the product, list of ingredients, nutrition declaration) or unauthorised health claims (ACN report, 2022). Moreover, notifications concerning possible faulty labelling are exchanged by Agri-Food Fraud Network (FFN) established by Regulation 2019/1715. Misdescription/ mislabeling/ misbranding was the second most reported category (22.5%) including “placing of explicit false claims” or “distorting the information on the label/packaging”. This covered a wide variety of matters ranging from false quality terms (23 cases), misleading weight declaration of a product (11 cases) to unauthorized nutrition and health claims (6 cases) (ACN report, 2022).

Basing on RASFF database, we found that one fifth of the notifications related to other labelling deficiencies were reported by Germany (20.1%). Other countries that frequently notified labelling inaccuracies were Italy (12.7%), Spain (8.2%) and Ireland (7.5%). One out of nine notifications was reported by European Commission (Figure 2).

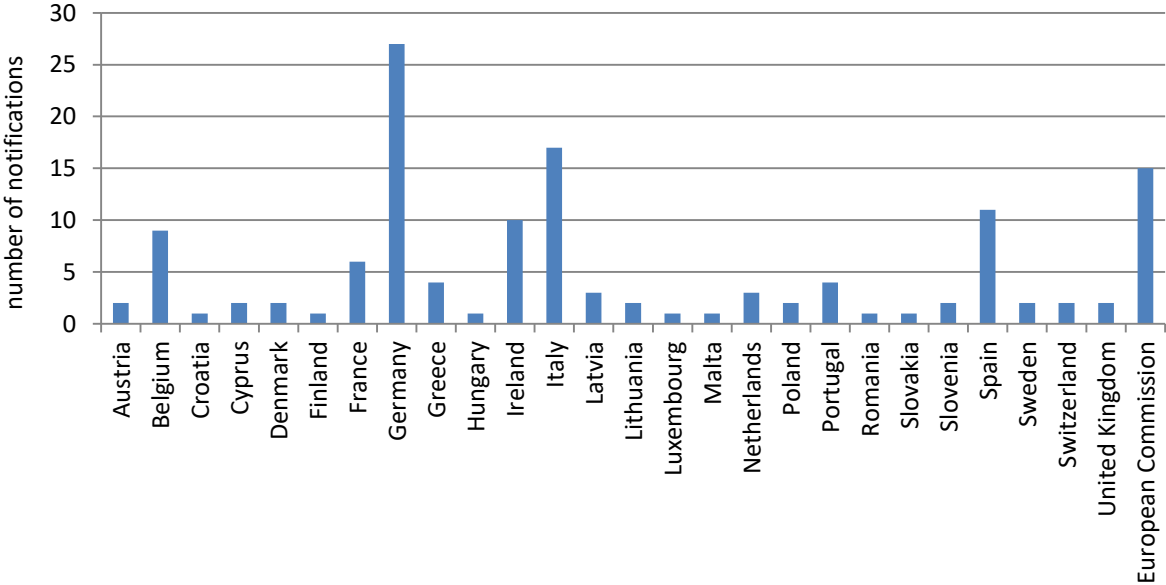


Fig. 2. RASFF notifications on other labelling deficiencies in the period from 01/01/2020 to 31/07/2023 by notifying country

Source: own compilation based on RASFF data from 2020 to 2023.

One-third of analyzed notifications were connected with improper composition, that affected to great extend the information on labels (31.9%). The subjects of the notifications included:

- the use of unauthorized/prohibited substances (e.g. Lippia alba, sibutramine),
- presence of novel food (e.g. Tetrapleura sp, chaga mushroom),
- too high or too low content of certain substances (e.g. too high zinc content in food supplement, maximum level of erucic acid exceeded in mustard oil, increased borate content in natural mineral water, too low sugar/fructose content in chocolate),
- undeclared ingredients (e.g. sugar in sugar-free soft drink).

One-tenth of notifications (10.4%) concerned date marking insufficient labelling. Wrong use of data format, missing date of minimum durability or use-by-date or extended expiry date was reported. The other problem concerned label/document mismatch (5.9%) and the absence of labelling in required language (4.4%). Unlabelled irradiation was indicated in few notifications (3.0%), among which three-fourth of cases concerned food supplements from United States. A few cases were connected with presence of unlabelled GMO (1.5%). Remaining notifications concerned insufficient/incorrect or absence of some elements of labelling such as incorrect storage instructions, missing lot number, missing warning notice for food supplement or other issues not in-depth reported.

The products in question originated in 19 RASFF member countries and 29 RASFF non-member countries (including United Kingdom). The country of origin of products in question in 12.1% was China. Among notified products from China most common were food supplements, fishes, fruits and vegetables. Products from Germany and Netherlands were notified in 7.3% cases, each.

The number of notifications due to labelling deficiencies in certain food category is shown in Figure 3. The results show that the main categories subject to notifications related to other labelling deficiencies were those of dietetic foods, food supplements and fortified foods (13.0%) and other food product / mixed (11.6%), followed by others that were classified as fish and products thereof (9.4%) and cereals and bakery products (8.7%).

Border rejections were the most common notifications on insufficient labelling (37.3%), while one-fifth of notifications in the analyzed period were classified as alert notifications (19.4%). Almost one-third of notifications (29.9%) were classified as information notifications for follow-up (Table 3).

One-fourth of RASFF notifications related to labelling deficiencies other than allergens in the period from 01/01/2020 to 31/07/2023 were of serious risk (26.1%). Approximate percentage of notifications was undecided (29.9%) and graded as not serious (25.4%) (Figure 4).

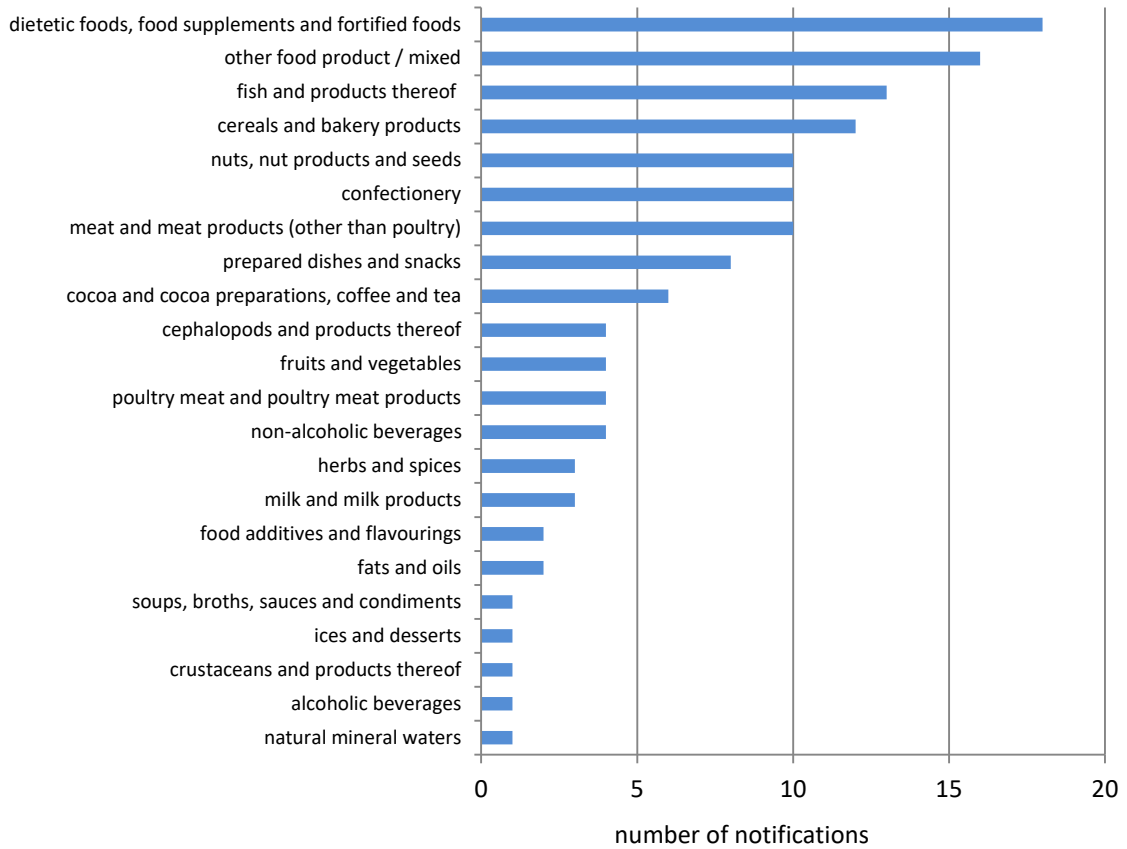


Fig. 3. RASFF notifications on other labelling deficiencies in certain product categories in the period from 01/01/2020 to 31/07/2023

Source: own compilation based on RASFF data from 2020 to 2023.

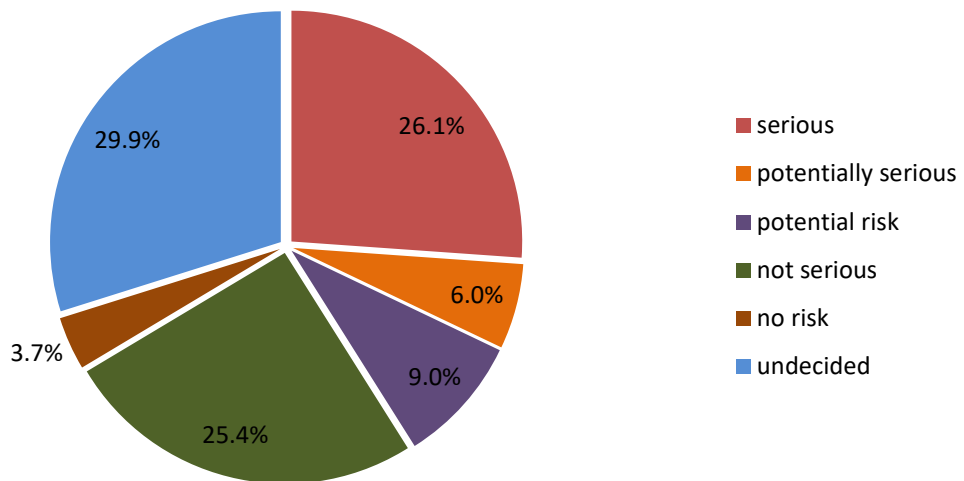


Fig. 4. Level of risk towards notifications concerning other labelling deficiencies in the period from 01/01/2020 to 31/07/2023

Source: own compilation based on RASFF data from 2020 to 2023.

Conclusions

Despite growing interest and expansion of use of QR codes and digital labelling, the traditional labels on food product packaging are important source of information by which consumers can acquire knowledge about the food they consider buying at the point of purchase. Mislabelling, lack of all mandatory information on label or imprecise information may lead to loss of trust in certain products, but also, in some cases, may pose a public health risk for a certain population. That is why the efforts made by the European Union to increase controls and consumers' information through labelling are of great importance. The undertaken study showed that there are still many cases of faulty labelling (related to allergens and other labelling deficiencies). In the period from 01/01/2020 to 31/07/2023 647 notifications were available in RASFF database, among which 79.3% was related to allergens and the other 20.7% concerned other labelling deficiencies. In consecutive years, the number of notifications has been increasing as in 2020 amounted to 157, 161 in 2021, 192 in 2022, while in the first seven months of 2023 already 137 notifications have been published. There is a big need to educate food business operators so that the official controls on the market and company's own check will limit revealing labelling deficiencies.

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NOTIFICATIONS ON HONEY IN THE RAPID ALERT SYSTEM FOR FOOD AND FEED (RASFF)

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DOI: 10.56091/CTQS.Qual-9

Abstract

Notifications concerning honey reported in the Rapid Alert System For Food and Feed (RASFF) accounted for 0.6% of all notifications and were submitted between 1999 and 2021, with the highest number of notifications in 2000–2008. These 514 notifications were sent within the product category “honey and royal jelly”. The aim of article was to analyse the notifications on honey in the RASFF taking into account the following variables: hazard category, hazard, notifying country, country of origin, notification type, notification basis and action taken. The data was exported from the archived RASFF database to a Microsoft Excel file and pre-processed used the “vertical search” function, pivot tables, sorting and transposition. Then the data was transferred to Statistica 13.3 and subjected to joining cluster analysis.

Notifications on honey related mainly to residues of veterinary medicinal products (e.g. chloramphenicol, streptomycin, sulfathiazole, nitrofurantoin metabolite, tylosin). Reported products were submitted by Western European countries and originated mainly from Asian, South American and Eastern European countries. These were primarily information notifications, and to the lesser extent also alerts. In order to maintain the safety of a particular type of food such as honey, it is necessary to continue with official market and border controls.

Keywords: cluster analysis, European Union, food safety, honey, RASFF

Introduction

The Rapid Alert System For Food and Feed (RASFF) was established in 1979 to exchange information between member states of the European Union (EU) in the event of public health risks arising from the food chain. It is currently based on Regulation (EC) No 178/2002 of the European Parliament and of the Council of 28 January 2002 laying down the general principles and requirements of food law, establishing the European Food Safety Authority and laying down procedures in matters of food safety.

Alert notifications are sent when a product presenting a serious health risk is already on the market and rapid action is needed (e.g. a product withdrawal). Information notifications are used when a risk

has been identified but rapid action is not needed because the product is not yet on the market, is no longer on the market or the nature of the risk does not require rapid action. In turn, border rejections refer to consignments that have been tested and rejected at the EU external border. These are sent to all border posts in the European Economic Area to prevent the product from entering through another post (EC, 2023b).

Natural honey, which is the subject of this study, is produced by *Apis mellifera* honey bees from the nectar collected by them or from honeydew. Honey occupies a special place in the diet of humans. It owes its role in the nutrition of society to its rich taste and strengthening and nourishing effect on the human body (Rozp. MRiRW, 2003; Dyrektywa 2014/63/UE, 2014; Standard for Honey, 2019).

The quality of honey is determined by many factors depending on the honeyflow, the bees and the beekeeper, as well as the conditions in the natural honey production environment (Fakhlaei et al., 2020; Kiczorowski et al., 2020; Wilczyńska, 2012).

The specific taste and health-promoting properties of honey contribute to their high price. The desire to increase profits drives producers or traders to adulterate honey. Counterfeiting process in progress for example, by mixing varieties, mislabeling them, using the addition of sugar syrups, repeated heating of honeys and other processes are deliberately used by producers (Džugan et al., 2018; Piotraszewska-Pajak & Gliszczyńska-Świgło, 2015; Soares et al., 2017; Śmiechowska, 2007).

Substances that may pose a threat to human health include the presence of veterinary drugs and pesticide residues in honey. These substances may have a negative impact on the reproductive system in the form of infertility, malignant tumors and congenital defects. There is also data about the adverse effects of these chemical compounds on the functioning of the thyroid gland and brain, the development of obesity and metabolic disorders involving insulin and glucose (Grigoryan, 2016; Mahmoudi et al., 2016; Szponar et al., 2002; Wei et al., 2012).

The consumption of adulterated honey and the potentially negative effects on human health and life would seem to be negligible. However, it should be emphasized that the scale of honey production in Poland is very large, and quality control is not complete. This is a product that goes for direct sale, often not included in the group of products formally available on the market “from the apiary to the consumer”, because beekeeping is often a hobby (D’Ascenzi et al., 2019; Grigoryan, 2016; Szczęsna, 2003). In 2022, domestic honey production amounted to approximately 24 thousand tons. Compared to 2021, it increased by 5.6 thousand tons and at the same time it was almost twice as high as two years earlier (Wysoczańska, 2023). The largest producer of honey in the world is China, which produces approx. 550 thousand tons of honey per year. The European Union honey market is over 280

thousand tons per year, which gives it second place in the ranking. The third largest producer of honey is Turkey (Miody Manuka, 2021).

The total number of notifications (more precisely, records) saved in the RASFF database in the period 1979–2021 was 82,396. Only 514 notifications (i.e. 0.6% of all notifications) were reported against the product category “honey and royal jelly”. These were reported between 1999 and 2021, with the highest number of notifications in 2000–2008 (the peak was in 2005 with over 70 notifications). After that, the number of notifications related to honey declined rapidly, and has only been around 10 per year in recent years (Figure 1) (EC, 2023a).

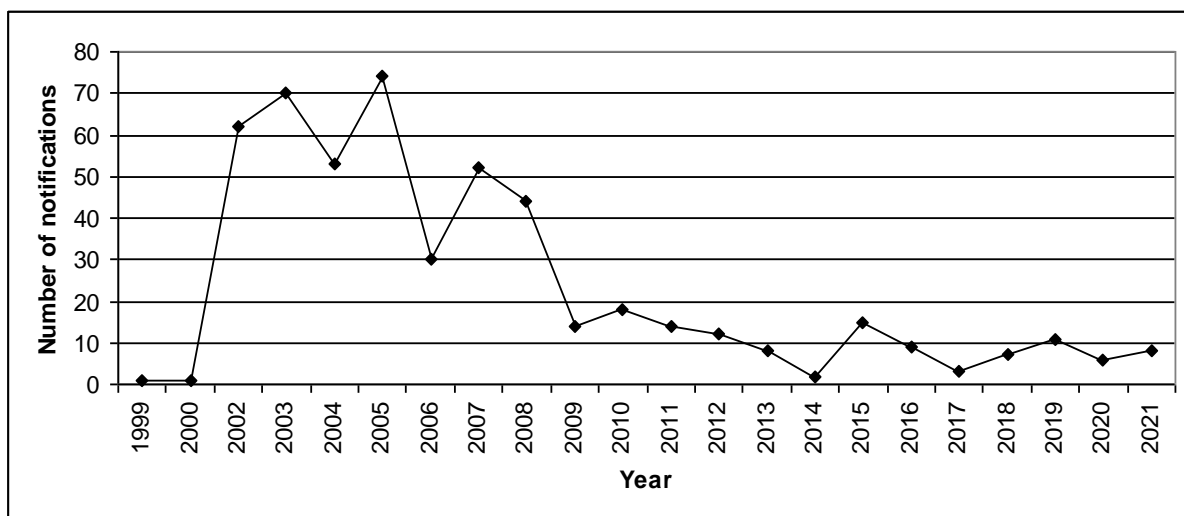


Fig. 1. Number of notifications on honey in the RASFF in 1999–2021

Source: own compilation based on RASFF data from 1999 to 2021.

Due to the small number of notifications to honey in the RASFF, no wider research has been carried out in this area to date. Therefore, the aim of this article was to analyse the notifications on honey in the RASFF taking into account the following variables: hazard category, hazard, notifying country, country of origin, notification type, notification basis and action taken.

Materials and methods

As only data from 2020 onwards are currently available on the official website of the European Commission (EC, 2023b), data was downloaded from the archived RASFF database (EC, 2023a). The data was exported to a Microsoft Excel file (Microsoft Corporation, Redmond, USA). Data pre-processing used the “vertical search” function, pivot tables and sorting. This provided information on the number of notification types for each variable, i.e. hazard category, hazard (in database: substance/finding), notifying country, country of origin, notification type, notification basis and action taken. For the last two variables, missing data (empty cells) were filled with the phrase “(not speci-

fied)". In the case of the variable "notification type", as of 2011, information notifications have been divided into two subtypes: information for attention and information for follow-up. In order to standardise this type of notification, its original name, i.e. information, has been restored.

Five other pivot tables were then constructed, with the types of the primary variable "hazard" in the rows and the types of the other variables i.e. notifying country, country of origin, notification type, notification basis and action taken in the columns. Transposition and sorting functions were also used in creating these tables. Empty cells (no notification) were filled with the value "0". For the variable "country of origin", the number of items was limited to those for which there were at least five notifications (due to the fact that the chart axes do not hold more than approx. 30 items).

The tables containing the source data were then transferred to Statistica 13.3 (TIBCO Software Inc., Palo Alto, USA) and subjected to joining cluster analysis. The following settings were used: linkage rule – Ward's method, distance measure – Euclidean distance and vertical icicle plots. The purpose of the joining cluster analysis was to join together objects into successively larger clusters based on their similarity and distance. The Ward's method uses analysis of variance to assess the distances between clusters, attempting to minimise the sum of squares of any two hypothetical clusters that may form at each stage. This method is considered very efficient, although it tends to flatten the clusters. In turn, the Euclidean distance is a geometric distance in a multidimensional space (the most commonly chosen distance type in joining cluster analysis) (TIBCO, 2022).

Results

Number of notifications by variable

In Table 1 presented the number of notifications on honey reported in the RASFF by variable.

As many as around 80% of all reported hazards were residues of veterinary medicinal products. These were drugs with antibacterial activity, such as antibiotics (e.g. chloramphenicol, streptomycin, nitrofurran, tylosin) or sulfonamides (e.g. sulfathiazole, sulfadimidine). The products were mainly notified by Western European countries (Spain, Germany, the United Kingdom, Italy and Belgium) and came primarily from China, other Asian countries (Turkey, Vietnam), South American countries (Argentina, Mexico), and Eastern European countries, including EU countries (Bulgaria, Hungary, Slovakia), Spain and Ukraine.

As many as two thirds were information notifications and a quarter were alerts. Notifications were reported on the basis of official controls on the market and border controls, after which consignments were detained or released. Actions taken against the reported products mainly included re-

dispatch, withdrawal of the product from the market or recall, destruction, but sometimes were also not specified.

Tab. 1. Number of notifications on honey reported in the RASFF by variable

Variable	Values (number of notifications)
Hazard category	Residues of veterinary medicinal products (416), Adulteration / fraud (25), Foreign bodies (12), Pesticide residues (11), Poor or insufficient controls (10), Packaging defective / incorrect (9), Allergens (5), Natural toxins (other) (4), Distribution restricted to notifying country (3), Distribution to other member countries (3), Labelling absent/incomplete/incorrect (3), Other (13)
Hazard	Chloramphenicol (117), Streptomycin (49), Sulfathiazole (38), Nitrofurantoin (metabolite) (22), Tylosin (20), Sulfadimidine (19), Tetracycline (17), Sulfamethoxazole (16), Sulfonamide (16), Oxytetracycline (15), Other (185)
Notifying country	Spain (86), Germany (81), United Kingdom (74), Italy (53), Belgium (39), Slovakia (24), Czechia (22), Greece (20), Poland (19), Cyprus (16), Other (80)
Country of origin	China (136), Ukraine (36), Bulgaria (32), Argentina (28), Turkey (25), Vietnam (25), Spain (20), Hungary (15), Mexico (15), Slovakia (14), Other (168)
Notification type	Information (333), Alert (125), Border rejection (56)
Notification basis	Official control on the market (186), Border control – consignment detained (169), Border control – consignment released (72), (Not specified) (64), Company's own check (13), Consumer complaint (3), Surveillance programme / monitoring sample (3), Monitoring of media (2), Border control – consignment under customs (1), Official control following RASFF notification (1)
Action taken	Re-dispatch (142), Product recall or withdrawal (64), (Not specified) (62), Destruction (48), Withdrawal from the market (41), Official detention (24), Import not authorised (20), Recall from consumers (14), Return to consignor (14), Informing authorities (12), Other (73)

Source: own compilation based on EC, 2023a.

Result of joining cluster analysis

In Figures 2–6 the results of the joining cluster analysis for the following variables were presented: notifying country, country of origin, notification type, notification basis and action taken, respectively. In the case of two variables (notification basis and action taken), the names of some values have been shortened to fit in the figure. For the variable notification basis, these were: official control (original name: official control on the market), border control – detained (border control – consignment detained), border control – released (border control – consignment released), border control – customs (border control – consignment under customs), surveillance programme (surveillance programme / monitoring sample) and official control – notification (official control following RASFF notification). As mentioned before, the names have also been shortened for the variable action taken, but this needs no explanation.

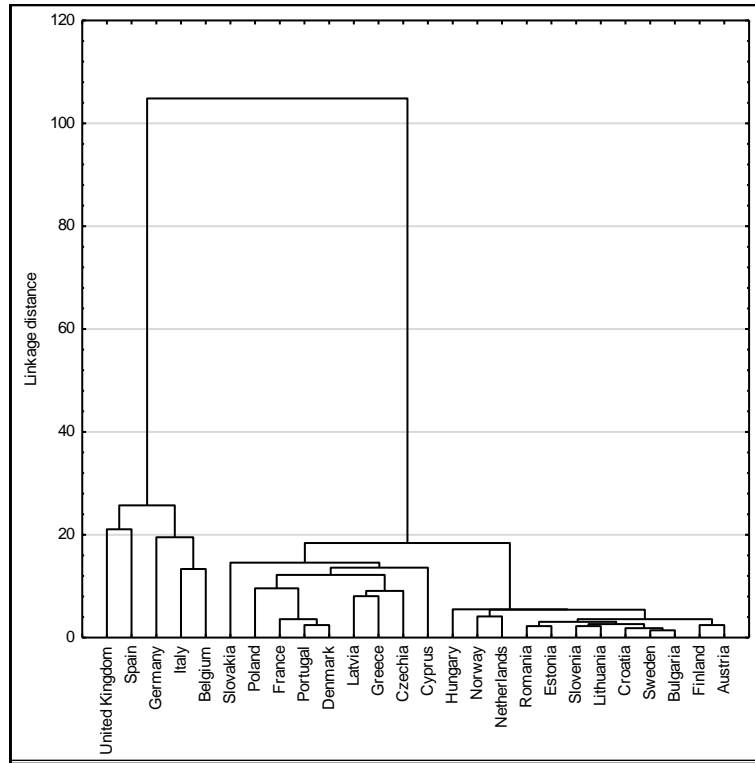


Fig. 2. Results of joining cluster analysis for notifying country
 Source: own compilation based on RASFF data from 1999 to 2021.

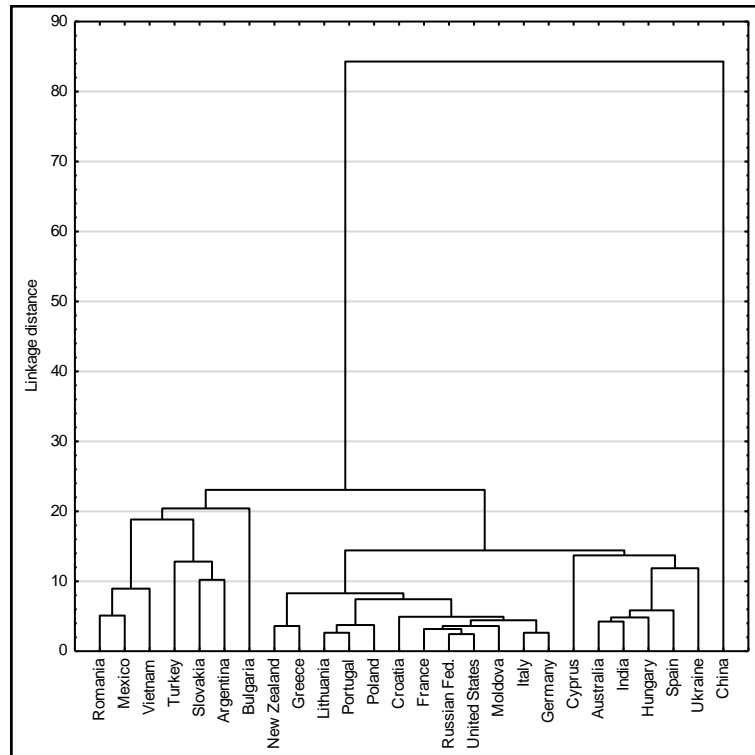


Fig. 3. Results of joining cluster analysis for country of origin
 Source: own compilation based on RASFF data from 1999 to 2021.

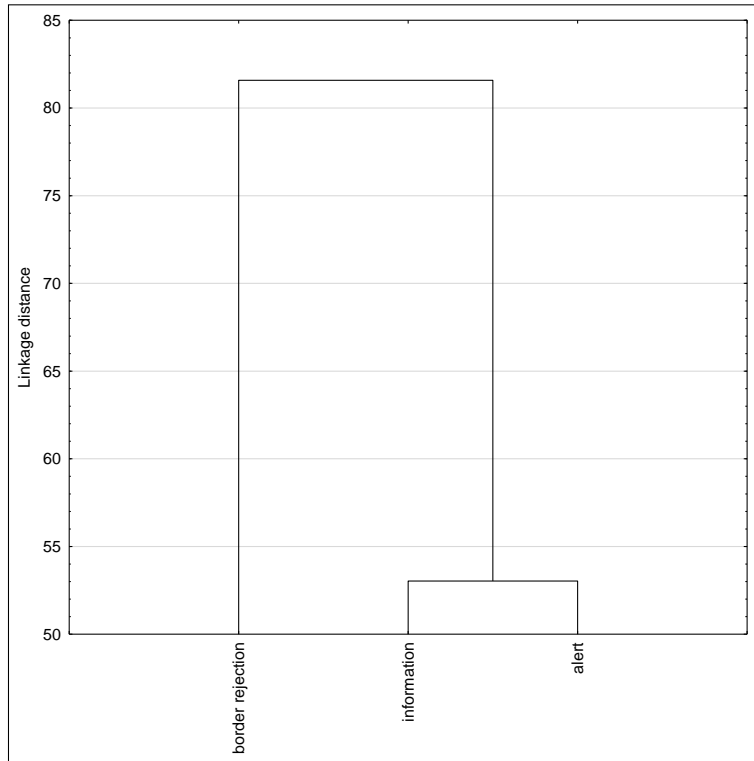


Fig. 4. Results of joining cluster analysis for notification type
 Source: own compilation based on RASFF data from 1999 to 2021.

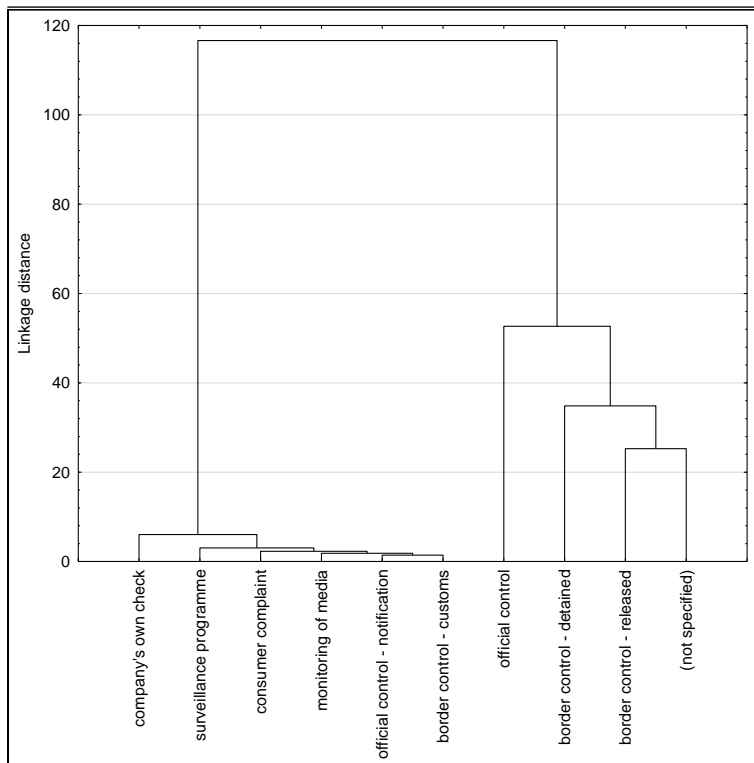


Fig. 5. Results of joining cluster analysis for notification basis
 Source: own compilation based on RASFF data from 1999 to 2021.

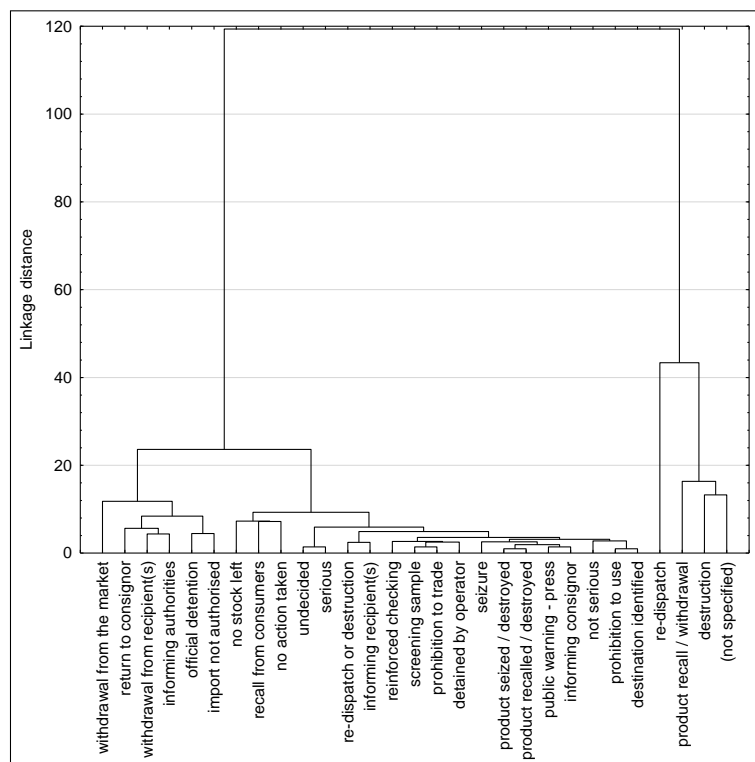


Fig. 6. Results of joining cluster analysis for action taken

Source: own compilation based on RASFF data from 1999 to 2021.

As mentioned in Materials and methods Figures 1–6 were generated from the source tables. In each of these tables, there were hazards in the rows (they were the same for each variable), but these are not visible in the charts due to the fact that this is a one-dimensional analysis. It is worth noting, however, that the sum of the number of notifications in each row was the same, but changed in each column (these were the values of the variables studied). As part of the joining cluster analysis performed, individual clusters were automatically grouped. The values of the individual variables with the highest number of notifications (one to several) are clearly separated from the others, further confirming the linkage distance.

The results of the cluster analysis show that the notifications with the highest number of notifications were combined into separate and very distinct clusters. On the left side of Figure 2, the cluster consisting of Western European countries making the most notifications against honey in the RASFF can be seen. These were the United Kingdom and Spain, Italy and Belgium (these pairs of countries are linked directly, indicating the greatest similarity of notifications), and also Germany. Due to the large number of notifications against honey originating from China, a separate, one-element cluster was created on the right side of Figure 3. Whereas on its left side, a separate cluster was formed by other countries from which the reported honey also originated, i.e. Romania and Mexico, Slovakia

and Argentina (directly linked), as well as Vietnam, Turkey and Bulgaria. Thus, it can be concluded that the hazards reported on honey were indirectly linked to the geographical region.

In the case of variable “notification type” the similarity can be seen for alert and information notification (Figure 4). In turn, considering the variable “notification basis”, it can be observed that notifications made under border controls after which the consignment was released were similar to those that were not specified. In turn, notifications under border controls, after which the consignment was detained, as well as official controls in the market were also in the same cluster (right side of Figure 5). All this indicates the similarity of the controls, carried out both at the external border and on the market. Finally, Figure 6 presents the actions taken against the notified honey. On the right, a cluster has been created with the most frequent actions applied. Notifications indicating destruction and those not specified are more similar to each other. This cluster also included recalls and withdrawals. These types of actions show that the reported products have not found their way to consumers.

Discussion

Despite many methods used to assess the quality of honey and to detect inaccuracies, honeys of disturbed quality still appear on the market (Szwedziak et al., 2017).

As a result of the analysis of the literature in the field of honey quality and authenticity assessment, it was found that there are many studies indicating deviations related to the quality of honey (Dżugan et al., 2018; Dykiel et al., 2022; Majewska et al., 2015; Szwedziak et al., 2017; Żak & Wilczyńska, 2018;). For example, according to the team of Witczak & Ciemniak, honey has residues of chemical compounds (Witczak & Ciemniak, 2012). However, according to the team of Sykut et al., residues of organic pollutants were observed in honey (Sykut et al., 2018). In addition, other parameters are tested: adulteration with starch, the presence of heavy metals and the microbiological quality of honeys (Gomes et al., 2010).

A separate aspect is the parameter for overheating honey or storing it in inappropriate conditions (5-hydroxymethylfurfural, diastase number, colour parameters) (Divelos et al., 2021; Piotraszewska-Pająk & Gliszczyńska-Świgło, 2015; Wilczyńska & Chomaniuk, 2018; Żak et al., 2018).

Meanwhile, recent Alert and Cooperation Network annual reports (for 2021 and 2022) issued by the European Commission draw attention to the suspected adulteration of honey through the substitution of extraneous sugars (EU, 2022; EU, 2023). It is also pointed out that these adulterated products come from China and Turkey (EU, 2023), the main honey producers besides the European Union. It is worth noting that, while this is not a significant problem from a food safety point of view, it does put

the consumer's financial interest at risk. It is notable at this point that in the earlier years of the operation of the RASFF, notifications of adulteration/fraud concerning honey mainly referred to problems with health certificate or import (EC, 2023a).

The above analysis of the literature was also confirmed in a study conducted on data from the RASFF system from 1999–2021. However, the use of the joining cluster analysis with different variables has allowed attention to be drawn to various relevant aspects, i.e. notifying country, country of origin, notification type, notification basis and action taken, which significantly extends the previous research. This research could be further deepened by applying two-way joining cluster analysis (two-dimensional analysis), taking into account simultaneously both the individual hazards and the values of the above-mentioned variables.

Conclusions

In the RASFF, notifications concerning honey are reported under the product category “honey and royal jelly”. Between 1999 and 2021, 514 notifications were made for this category, representing only 0.6% of all notifications in the system over the period indicated. The highest number of notifications was submitted between 2000 and 2008.

Notifications on honey related mainly to residues of veterinary medicinal products (e.g. chloramphenicol, streptomycin, sulfathiazole, nitrofurantoin metabolite, tylosin). Reported products were submitted by Western European countries (Spain, Germany, United Kingdom, Italy, Belgium) and originated mainly from Asian (China, Turkey, Vietnam), South American (Argentina) and Eastern European countries (Ukraine, Bulgaria). These were primarily information notifications, and to the lesser extent also alerts. Products were submitted after official controls on the market, border controls and were re-dispatched, recalled and withdrawn from the market.

In order to maintain the safety of a particular type of food such as honey, it is necessary to continue with official market and border controls. Producers should pay particular attention to the proper use of veterinary medicines on bees. Furthermore, as the honey notified in the RASFF originated mainly from non-EU countries, consumers should take special note of the origin of this product.

The use of joining cluster analysis highlighted the most relevant aspects related to honey notifications in the RASFF. This research could also be deepened through the use of two-way joining cluster analysis. However, it would be necessary to merge the data from the restored database (containing historical data) with the currently available official RASFF database.

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Part II

Quality and safety of non-food products

EVALUATION OF QUALITY OF NOBLE METALS RECOVERY AND SEPARATION USING IONIC LIQUIDS

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DOI: 10.56091/CTQS.Qual-10

Abstract

In recent years, there is a notable increase in the interest in noble metals, especially precious metals (PMs), such as palladium(II), rhodium(III), gold(III) and platinum(IV) because of a wide range of their industrial applications. They are used as catalysts in organic technology processes, as value added components in metal alloys and as vehicle catalytic converter systems, in jewellery making and also in the chemical, pharmaceutical, petroleum and electronic industries. Recovery of precious metals from spent materials is very important to replenish a gap between the demand and the limited supply from natural resources. PMs have been classified as critical raw materials, thus a circular economy model should be implemented for their effective recovery. Therefore perspective of recycling PMs from secondary resources is beneficial not only for the environment (less waste) but also for the economy.

Ionic liquid-assisted solvent extraction has shown excellent quality for precious metals extraction/separation from chloride solutions and therefore proved to be an alternative to the conventional method of separation. Metal extraction is very sensitive with the types of anions and cations incorporated to the ILs. Different mechanisms might be involved, which could be ion exchange, neutral extraction, split-anion mechanism, and combined ion exchange-neutral extraction.

Keywords: noble metals, ionic liquids, solvent extraction, quality of separation

Introduction

Technological processes all over the world show relentless demand for precious metals, in particular palladium (Pd), rhodium (Rh), platinum (Pt) and gold (Au). The demand is still increasing, as these metals have been for many years used as catalysts in the organic technology processes, as boosters in motor vehicle catalytic converter systems and recently as afterburners in modern, environmentally friendly domestic furnaces installations. Besides the application in vehicle catalytic converter systems, palladium is used mainly in dentistry, jewelry and electrical engineering. Palladium has been widely used in electronic applications on account of its electrical conductivity and durability. Palladium-containing components are used in virtually every type of electronic device, from basic consumer products to complex military hardware. In addition, platinum is also used in jewelry products, in in-

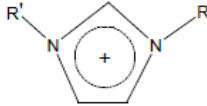
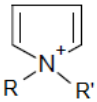
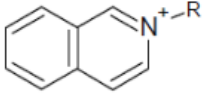
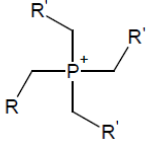
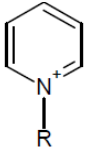
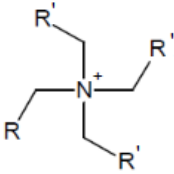
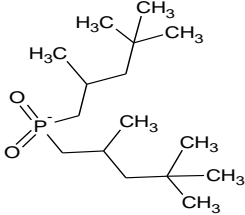
organic chemical, petrochemical, electrical, glass industry, dentistry, it also is a good capital investment. Rhodium finds applications mainly in catalytic converters, chemical and glass industry. While gold plays an increasingly important role in industrial applications, particularly in electronics, despite its larger use in jewelry and investment products. In 2022, demand for palladium was 309.0 tons, platinum 194.8 tons, rhodium 31.0 tons and gold 4,742.7 tons, and their prices were 2,061.06 \$/oz, 958.06 \$/oz, 14,750.05 \$/oz and 1,775.50 \$/oz, respectively (Hagelüken & Corti, 2010; Matthey, 2023, Statista; World Gold Council).

A large numbers of PMs applications have increased the demand for these metals, whereas the natural resources are limited. Therefore, the gap between the demand and supply from natural sources, must be replenished by recycling of spent materials. In 2022 near 1,445 tons of gold, 46 tons of platinum, 96.5 tons of palladium and 10.5 tons of rhodium were recycled worldwide (World Gold Council, Matthey 2023).

Various metallurgical methods for recovering PMs from these resources have been developed. Among them, hydrometallurgical processes through chloride leaching are the most preferred methods for extracting PMs, except Ag, as metal-chloro complexes MCl_n^{m-} where M = PM cation, m = 1–3, and n = 2–6. These processes have been well understood with strong theoretical background and successfully implemented on industrial scale. However, subsequent refining of the chloro-complexes faces continual challenges due to their similar physico-chemical properties, and has drawn attention of academics and industries in order to develop an efficient metal refining method (Lee et al., 2020; Lee et al., 2023; Trinh et al., 2020).

Currently, the recovery of PMs from natural and spent materials is carried out using traditional pyrometallurgical or hydrometallurgical methods. Hydrometallurgical processing of waste materials has been the area of the most intensive research in the field of wet techniques for the last two decades. Solvent extraction in hydrometallurgical processing is one of the major industrial-scale refining processes of PM-chloro complexes. This method has benefits for its suitability to treat solutions with high metal content, improved degree of extraction and separation yields, capability of continuous operation, recycling of the organic extractants, flexible and versatile process control and fast kinetic. Until now many different extractants have been studied and proposed for recovery and separation of precious metals, e.g. hydrophobic amines (Jha et al., 2014; Nguyen et al., 2016), organophosphorus extractants (Gupta et al., 2014; Nguyen et al., 2015), different derivatives of amides (Costa et al., 2018; Huang et al., 2015; Mowafy & Mohamed, 2016; Ortet & Paiva, 2015; Sasaki et al., 2017), pyridine (Khogare et al., 2016a, 2016b) and piperidine (Cieszyńska & Wieczorek, 2018).

Tab. 2. Examples of ILs used in metals extraction

Cations			
			
Imidazolium	1,1'-alkylpyrolium	N-alkylisoquinolinium	
			
Phosphonium	Alkylpyridinium	Ammonium	
Anions			
Cl^-	Br^-	BF_4^-	PF_6^-
Chloride	Bromide	Tetrafluoroborate	Hexafluorophosphate
$\text{B}(\text{CN})_4^-$	$\text{N}(\text{CN})_2^-$	CF_3SO_3^-	
Tetracyanborate	Dicyanamide	Trifluoromethylsulfonate [TfO]	
$\text{N}(\text{SO}_2\text{CF}_3)_2^-$			
Bis(trifluoromethylsulfonyl)imide [NTf ₂]	Bis(2,4,4-trimethylpentyl)phosphinate [C ₈ C ₈ PO ₂]		
$\text{N}(\text{SO}_2\text{CF}_2\text{CF}_3)_2^-$			
Bis(perfluoroethylsulfonyl)imide [BETI]			

R, R' – alkyl groups, the same or different

Ionic liquids (ILs) are molecules, which consist of cation (the most common: ammonium, imidazolium, pyridinium, piperidinium or phosphonium cations) and an inorganic or organic anion (Table 2). They are liquid at temperatures below 100°C and indicate some unique properties, like negligible volatility, nonflammability and thermal stability. Their solubility in water, cloud point and viscosity depend on the cation and anion used (Lee et al., 2023; Regel-Rosocka & Materna, 2014).

In solvent extraction process, ILs have found prospects of being used as active extractants (diluted or undiluted), diluents for both MEs and ILs, and task-specific ionic liquids (TSILs) (Kurniawan et al., 2022). ILs as active extractants have shown capability of high extraction and successful separation of complex metal ion systems, including PM-chloro complexes e.g. ammonium, phosphonium, imidazolium, pyridinium, piperidinium, pyrrolidinium and betainium ionic liquids (Table 3).

Tab. 3. Studies on the IL-assisted solvent extraction of PM-chloro complexes

Organic phase	Aqueous phase	Results	Reference
Methyltrioctyl-ammonium chloride [N1888][Cl] (Aliquat 336) in kerosene	Pt(IV), Rh(III), Al(III), Mg(II), Fe(III) in 1 M HCl	Selective and effective extraction of Pd (100%) after preliminary precipitation of Al and Fe. Stripping of Pd with 0.5 M thiourea in HCl.	Raju et al., 2012
Aliquat 336 in kerosene	Pd(II), Pt(IV) in HCl (1–8 M)	Most Pd and Pt is extracted when HCl concentration was lower than 3 M. When HCl concentration increases to 8 M Pd extraction decreases more rapidly than Pt.	Nguyen et al., 2015
Aliquat 336 in benzene	Pd(II), Au(III), Pt(IV) in 0.1 M HCl	Quantitative and selective extraction of Au (100%). Poor extraction of Pd and Pt (<10%)	Wei et al., 2016
Aliquat 336 in chloroform	Pd(II) in HNO ₃ (0.1–6 M)	Extraction of Pd decreases with an increase in the concentration of nitric acid	Giridhar et al., 2006
Aliquat 336 in cyclohexane	Pd(II), pH=2,	Pd extraction = 94.7%	Mohdee et al., 2018
Cyphos IL 101 in toluene	Pd(II), Ni(II), Cu(II), Pb(II), Fe(III), Rh(III), Ru(III), Pt(IV) in 0.1 M HCl	Efficient extraction of Pd (65%). Less effective extraction of Pt (20%). Poor extraction of the other metals (<3%). Stripping of Pd with 0.5 M ammonia solution.	Cieszyńska & Wiśniewski, 2011
Cyphos IL 104 in toluene	Pd(II) in 0.1 or 3 M HCl	Efficient extraction of Pd (96%) from 0.1 M HCl. Less effective extraction of Pd (52%) from 3 M HCl. Effective stripping with 0.5 M ammonia solution.	Cieszyńska & Wiśniewski, 2012

Cyphos IL 102 in toluene	Pd(II), Rh(III), Ru(III), Pt(IV) in 0.8 M HCl	Extraction of Pd, Pt, Ru, Rh amounted to 95%, 80.5%, 19.7% and 10.7%, respectively. Stripping of Pd with 0.1 M thiourea in 0.5 M HCl.	Rzelewska-Piekut & Regel-Rosocka, 2019
1-butyronitrile-4-methyl-pyridinium bis(trifluoromethylsulfonyl)imide	Pd(II) in water (pH=7)	Very efficient extraction of Pd.	Papaiconomou et al., 2008
1-methyl-2-pentene-pyrrolidinium bis(trifluoromethanesulfonyl)imide or 1-methyl-2-pentene-piperidinium bis(trifluoromethanesulfonyl)imide	Pd(II) in water (pH=2.8)	Quantitative extraction of Pd (100%) with both ionic liquids.	Lee, 2012
Betainium bis-(trifluoromethanesulfonyl)imide	Pd(II), Rh(III), Ru(III) in HNO ₃	Pd - the most extractable, Rh - medium extractable, and Ru - the least extractable.	Saski et al., 2014
Trioctyl(dodecyl) phosphonium chloride [P ₈₈₈₁₂][Cl]	Pt(IV), Pd(II), Rh(III) in 0.1 – 5 M HCl	Extraction of Pt, Pd: 99.9%, Rh: 10.0–90.0% (decreasing with increasing HCl conc.)	Firmansyah et al., 2018
Methyltrioctyl-ammonium chloride/bromide/iodide [N ₁₈₈₈][Cl/Br/I] in p-cymene	Au(III), Pt(IV), Pd(II), Rh(III) in 0.001–6.0 M HCl	Extraction of Au, Pt, Pd > 99.0%; Rh: 20.0–80.0% (I, Br); 10.0–38.0% (Cl)	Nguyen et al., 2020
1-butyl-3-benzimidazolium bromate [HBBIm][Br] in chloroform	Pd(II), Pt(IV) in 0.007 M HCl	Recovery of P and Pd: 99.9% (Pt precipitated while Pd was extracted)	Liu et al., 2017
1-benzyl-3-methylimidazolium bis(trifluoromethylsulfonyl) imide [BMIm][NTf ₂]	Pd(II), Pt(IV) in 0.1 M HCl	Pt extraction equals to 90%	Lim et al., 2021

Materials and methods

Reagents

Commercial palladium chloride PdCl₂ (99%, Avantor Performance Materials Poland S.A., Poland), platinum chloride PtCl₄ (99%, Avantor Performance Materials Poland S.A., Poland), gold chloride AuCl₃ (99%, Aldrich, Poland), hydrochloric acid (analytically pure, 35–38%, Chempur, Poland), nitric acid (analytically pure, 65%, Avantor Performance Materials Poland S.A., Poland), thiourea (analytically pure, Chempur, Poland) and aqueous ammonia (analytically pure, 25%, Chempur, Poland) were used to prepare aqueous solutions. Commercial pure trihexyl(tetradecyl)phosphonium chloride [3C₆C₁₄P]Cl (pure, ≥95%, Sigma-Aldrich), trihexyl(tetradecyl)phosphonium bis(2,4,4-trimethylpentyl)phosphinate [3C₆C₁₄P][C₈C₈PO₂] (pure, ≥95%, Sigma-Aldrich) and trihexyl(tetradecyl)phosphonium dicyanamide [3C₆C₁₄P]DCA were used as extractant. Toluene (analytically pure, Chempur, Poland) was used as diluent of the extractant.

Procedure

Aqueous feed contains 85 mg/dm³ Pd(II), 200 mg/dm³ Au(III) and 30 mg/dm³ Pt(IV). Stock solutions were prepared by dissolving appropriate amounts of their suitable chlorides in double distilled water containing a minimum amount of the corresponding mineral acid. The acidity of the solution was controlled by HCl solution (0.1 and 3.0 M). The organic phases were 2.5 mM solutions of [3C₆C₁₄P]Cl, [3C₆C₁₄P][C₈C₈PO₂] and [3C₆C₁₄P]DCA in the presence of toluene.

Extraction was carried out in a typical way. Both phases were mechanically shaken in glass separatory funnels (volume ratio A/O = 1) for a 10 minutes at room temperature (20±1°C). After mixing, both phases were left to stand and then separated. The loaded organic phase was stripped with 3.0 M HNO₃, 0.5 M aqueous ammonia and 0.1 M thiourea in 0.1HCl (A/O = 1). Microwave plasma-atomic emission spectroscopy (4210 MP AES, Agilent, USA) was used for metal determination in the initial aqueous solutions and in the aqueous phases after extraction and stripping.

Percentage extraction (E) was calculated from concentration of metal ions in the aqueous phases before [M]_(i) and after [M]_(aq)* extraction:

$$E = \frac{[M]_{(i)} - [M]_{(aq)}^*}{[M]_{(i)}} \cdot 100\%$$

The volumes of phases did not change. Each experiment was carried out three times and the error did not exceed 5%.

Results and discussion

It is well known that precious metals (PMs) forms series of complexes in acidic chloride solutions depending on the HCl concentration and oxidation states of metals. Previous research and many publications unambiguously indicate that the predominant species of palladium(II), platinum(IV), and gold(III) and in HCl (0.1–5 M) are $[\text{PdCl}_4]^{2-}$, $[\text{PtCl}_6]^{2-}$, $[\text{AuCl}_4]^-$, respectively (Bernardis et al., 2005). The effect of hydrochloric acid concentration on palladium(II), platinum(IV) and gold(III) extraction and separation with examined extractants was investigated (Fig. 2).

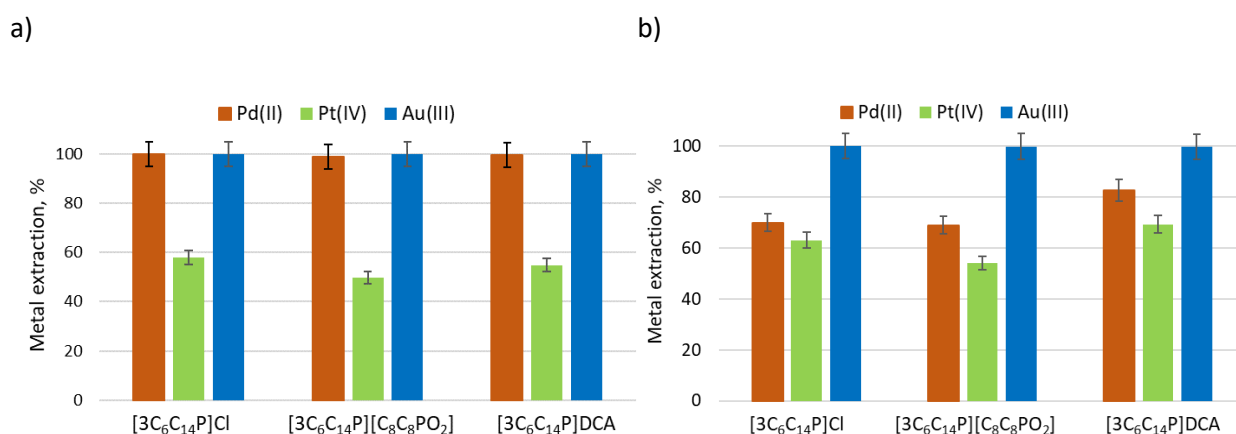


Fig. 2. Extraction of palladium(II), platinum(IV) and gold(III) from multi-metal solution. Initial aqueous phase: $[\text{Pd(II)}] = 85 \text{ mg/dm}^3$, $[\text{Au(III)}] = 200 \text{ mg/dm}^3$, $[\text{Pt(IV)}] = 30 \text{ mg/dm}^3$ in a) 0.1 M HCl and b) 3.0 M HCl; organic phase: $[\text{3C}_6\text{C}_{14}\text{P}]\text{Cl}$, $[\text{3C}_6\text{C}_{14}\text{P}][\text{C}_8\text{C}_8\text{PO}_2]$ and $[\text{3C}_6\text{C}_{14}\text{P}]\text{DCA}$ in toluene; A/O = 1
Source: own research.

The extraction effectiveness of gold(III) not depends on HCl concentration and equals near 100% both from 0.1 and 3.0 M HCl. The increase HCl concentration has unfavorable influence on palladium(II) extraction. The extent of palladium(II) extraction with $[\text{3C}_6\text{C}_{14}\text{P}]\text{Cl}$, $[\text{3C}_6\text{C}_{14}\text{P}][\text{C}_8\text{C}_8\text{PO}_2]$ and $[\text{3C}_6\text{C}_{14}\text{P}]\text{DCA}$ from 0.1 M HCl amounts to nearly 100%, while from 3.0 M HCl it decreases to approximately 70%, 69% and 83%, respectively. While the efficiency of platinum(IV) extraction slightly increases (by about 5 – 14%) with the increase of HCl concentration. Studied phosphonium ionic liquids as extractants indicate the extraction ability towards examined noble metals ion according to the following order $\text{Au(III)} > \text{Pd(II)} > \text{Pt(IV)}$ from 0.1 M HCl and $\text{Au(III)} > \text{Pt(IV)} > \text{Pd(II)}$ from 3 M HCl. The effectiveness and quality of separation of palladium(II), platinum(IV) and gold(III) is not satisfactory, but it is possible using the stripping process.

The organic phase loaded with metal ions should be stripped to obtain the extractant, which would be reusable in the next extraction. The stripping of palladium(II), platinum(IV) and gold(III) from loaded organic phase was investigated (Table 4).

Tab. 4. Stripping of palladium(II), platinum(IV) and gold(III) from loaded organic phase after extraction. Extraction: initial aqueous phase: [Pd(II)] = 85 mg/dm³, [Au(III)]=200 mg/dm³, [Pt(IV)]=30 mg/dm³ in 0.1 M HCl; organic phase: [3C₆C₁₄P]Cl, [3C₆C₁₄P][C₈C₈PO₂] and [3C₆C₁₄P]DCA in toluene

Metal loaded organic phase	Stripping	Stripping percent [%]		
		Pd(II)	Pt(IV)	Au(III)
[3C ₆ C ₁₄ P]Cl	0.5 aqueous ammonia	100	0	5
	3 M HNO ₃	2	93	0
	0.1 M thiourea in 0.1 M HCl	89	5	100
[3C ₆ C ₁₄ P][C ₈ C ₈ PO ₂]	0.5 aqueous ammonia	99	0	5
	3 M HNO ₃	3	97	0
	0.1 M thiourea in 0.1 M HCl	81	3	100
[3C ₆ C ₁₄ P]DCA	0.5 aqueous ammonia	98	7	4
	3 M HNO ₃	3	95	0
	0.1 M thiourea in 0.1 M HCl	77	0	100

Source: own research.

Palladium(II), platinum(IV) and gold(III) can be successfully separated through stripping from the loaded organic phase. On the basis of the extraction-stripping data, a scheme for the recovery and separation of palladium(II), platinum(IV) and gold(III) from 0.1 M HCl with examined ionic liquids was proposed (Fig. 3).

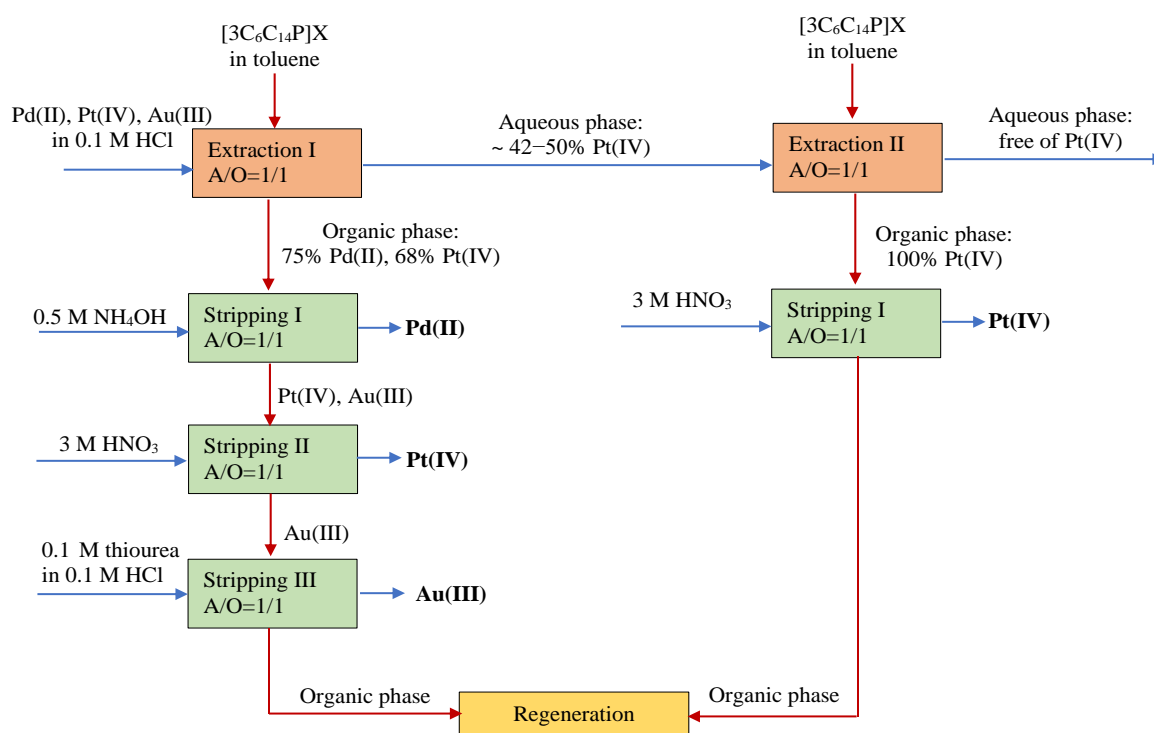


Fig. 3. Separation of palladium(II), gold(III) and platinum(IV), where X = anion Cl, [C₈C₈PO₂] or DCA

Source: own research.

Conclusions

The ability of three phosphonium ionic liquids to recovery and separation of palladium(II), platinum(IV) and gold(III) from hydrochloric acid solutions was studied. The results presented in this paper prove that all examined reagents can be used as extractants for the removal of palladium, platinum and gold ions from chloride media. The efficiency and quality of separation of palladium(II) from platinum(IV) and gold(III) is not so effective and it depends upon the acidity of aqueous solution. $[3C_6C_{14}P]Cl$, $[3C_6C_{14}P][C_8C_8PO_2]$ and $[3C_6C_{14}P]DCA$ indicate also that the extraction ability towards examined noble metals ion decreases according to the following order $Au(III) > Pd(II) > Pt(IV)$ from 0.1 M HCl and $Au(III) > Pt(IV) > Pd(II)$ from 3 M HCl. Palladium(II), platinum(IV) and gold(III) can be successfully separated with high effectiveness and quality through stripping from the loaded organic phase. The extraction-stripping data obtained and further research can be used to develop method for effective recovery and separation of palladium(III), platinum(IV) and gold(III) palladium, from spent materials, e.g. waste electronic and electrical equipment (WEEE).

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TESTING OF SURFACE PRIMERS FOR AUTOMOTIVE REFINISHING

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DOI: 10.56091/CTQS.Qual-11

Abstract

Automotive coatings have gone a long way since the early 1900s and new application methods as well as compounds used have been developed since. Novel coatings can produce durable surfaces that protect from extreme temperatures, UV radiation, or foreign particles. Automotive coatings enhance vehicle's appearance and more importantly provide anticorrosion protection. They are used in new cars, but also for repairing and refurbishment of damaged vehicles. That's why the Original Equipment Manufacturer (OEM) market develops rapidly. Vehicle refinishing is a process that involves the local repair of defects in the car's coating or the full upgrade of the vehicle by providing a more attractive coating. The choice of the appropriate coating method is very important and should be made based on many factors, including: the purpose, the type of material and its properties, coating conditions and ecological and economic requirements. The choice of application method will largely depend on the chosen coating product. The paper compares the physical and chemical properties of surface primers commonly used on the European market in the vehicle refinishing industry. The study included research on, mechanical and physical properties of each primer, as well as chemical composition analysis.

Keywords: coatings, automotive refinishing, OEM, VOC

Introduction

The paint industry is a vital part of the automotive industry and plays a key role in the production of automobiles and vehicle repair and refurbishment. Its main task is to provide a durable and attractive protective coating on the surface of vehicles. The auto refinishing coatings sector is an industry with major products such as primers, putties, basecoats, topcoats and clearcoats. In addition to these main products, there are also various types of additives that help in the car refinishing process. These products allow to prepare the surface, fill cavities, protect the surface and give the car a new, durable look.

Before paint products reach the market, they undergo a series of tests and tests to make sure they meet the highest quality standards and customer requirements. There are hundreds of different techniques for analysing paints and coatings that provide detailed information about the product,

such as viscosity, gloss, opacity, or hardness. Through these analyses, paint manufacturers can provide their customers with the best possible products.

The coatings industry is considered a mature and stable segment of the global economy, and demand for coatings has followed overall economic activity. Nonetheless, the Coronavirus outbreak has caused production to stall and a problem with raw material availability in 2020 and 2021. Solvent-based technologies accounted for the largest share of the global coatings market in 2021, but the thinner coatings segment is expected to grow slowly due to regulations on VOC emission levels. In 2022, the paints and coatings market in Europe reached a value of 39.4 billion euros. Decorative paints have the largest market share, accounting for 54.7% of the market value. The value of the paints and coatings market is projected to grow steadily from 2022 to 2028, with an expected volume growth of 1.5% and a CAGR (compound annual growth rate) of 3.4% (European Coatings Journal, 2022; European Coatings Journal, 2023).

The choice of a suitable coating method depends on many factors, including environmental considerations. Before choosing, it is important to consider the type of objects to be coated and what coating conditions need to be met. In the case of commercial vehicle refinishing, airless spraying has gained the most popularity, as it allows great flexibility in controlling the outcome of the coating, adjusting the amount of paint, air, and spraying time. In some branches of the paint industry, visual quality is not required as much as for automobiles, so the choice of method usually depends on performance and the type of paint material used.

The basic division of the types of paints include organic solutions, aqueous solutions, aqueous dispersions, non-aqueous dispersions, non-diluting so-called 100% systems and powder systems. Even with one group of products, attention must be paid to the spray viscosity, which must be adjusted by dosing the thinner accordingly. The paint industry is obliged to pay a fee for the emission of VOCs into the atmosphere, so sometimes the use of ecological solutions such as water-based coating is more advantageous and allows to reduce emissions of harmful substances (Brock et al., 2010; Mischke, 2010).

Vehicle refinishing is a process that involves the local repair of defects in the car's coating or the full upgrade of the vehicle by providing a more attractive coating. Due to the extended life of OEM coatings, the number of colours continues to grow. Original coatings, differ from those after car refinishing, are divided into three-layer and four-layer (Müller & Poth, 2017). Moreover, the knowledge and experience of the painters and the quality of the materials used are crucial to achieving satisfactory results in the vehicle refinishing process (Goldchmidt & Streutberger, 2018).

In the commercial vehicle restoration industry, the restoration process begins with surface preparation for further treatment. As the first stage, sheet metal work is usually performed. In this phase, knocking out dents is a labour-intensive process requiring a lot of manual work. Unfortunately, it is impossible to achieve a perfectly smooth surface, and the remaining irregularities are smoothed out with polyester putty. The next step is to remove old layers of paint, oil, grease, rust, and scale for better adhesion usually by hand sanding. Often this type of work is done with an angle grinder or sandblasting (Streitberger & Goldschmidt, 2018; Poth, 2008).

The phenomenon of spalling caused by streamlined cars and higher driving speeds has become one of the main reasons for the introduction of sleepers. However, many other important factors affecting the quality of sleepers must be considered when designing them including:

- covering strength,
- surface wetting,
- adhesion,
- stone impact resistance (Müller & Poth, 2017; Poth, 2008).

Topcoats of the same thickness and colour, but with different surface textures, can differ in their filling ability. Filling does not depend only on the thickness, but also on the appearance of the surface structure itself. To ensure the best impression of the primer's opacity, a small amount of long-lasting structure should be used, as too much of such structure can lead to an orange peel effect on the primer's surface. Nowadays, more and more topcoats are characterized by a higher gloss, which is because no sanding is required when applying the product wet-on-wet. Matte coatings are characterized by a texture that reflects light in a diffuse manner, which makes it difficult to recognize the levelling effect of the surface, while shinier surfaces allow better recognition of this effect. This depends mainly on the pigment volume concentration (PVC). However, for this type of product, PVC should not exceed the critical pigment volume concentration (CPVC), as otherwise the pigments will not be fully wetted with the binder and layer of absorption, which will directly affect the appearance of the coating. The selection of suitable fillers for the formulation also affects the quality of the coating (Gysau, 2017; Poth, 2008).

The aim of this work was to compare the physicochemical properties of surface primers commonly used on the European market in the vehicle refinishing industry. As part of the work, tests were carried out on the mechanical and physical properties of each primer, as well as an analysis of the chemical composition. The results of the study allowed a thorough comparison of the properties of the different surfacing primers and the determination of the level of differences between products from the same product group. The results of the research may be useful for professionals in the vehi-

cle refinishing industry, who can use the results obtained to select the right product and optimise refinishing processes. Based on previous findings, we hypothesize that:

Hypothesis 1: Rheological properties (viscosity, thixotropy) affect the sedimentation of pigments and fillers.

Hypothesis 2: The amount of fillers introduced influences the mechanical resistance of the finished coatings.

Hypothesis 3: The composition of the mixtures influences the drying time of the coatings, which in turn plays a major role in the applicability of the filler and primer systems.

Materials and methods

The study used two-component isocyanate-cured topcoats, which are commonly used in the car refinishing industry. Three commercially available products were selected for the study: Cromax topcoat primer (Axalta Coating Systems group, batch numbers: 4710333010, 4729970020, 4700035010), a hardener and a thinner. Also Glasurit (BASF, batch numbers: 0132782352210, 0132342905107, 0132288163103). The third one was a Spectral (Novol, batch numbers: 1010044, 990947, 1014971). Table 1 presents the mixing ratios of selected primers according to manufacturers' technical sheets.

Tab. 1. Mixing ratios of selected primers

Topcoat primer, hardener and thinner	Volume ratio	Weight ratio [g]
Cromax	4:1:1.5	100:16:19
Glasurit	4:1:1.5	100:22.8:20
Spectral	4:1:1.5	100:16:35

Source: Manufacturers' technical sheets.

The tests were carried out on three different production batches of each primer, and the results were averaged. The following tests were carried out according to their methodology:

1. determination of drying times according to ASTM D1640,
2. determination of the density using the pycnometric method according to PN-EN ISO 2811-1:2016-04,
3. determination of volatile organic compound content according to PN-EN ISO 11890-1:2008
4. flow viscosity according to DIN 53 211,
5. determination of dynamic viscosity by the Brookfield method according to PN-EN ISO 2555:2018-07,

6. testing of ash content according to ASTM D 5630,
7. level of spreading according to PN-EN ISO 1524:2020-12,
8. determination of adhesion to steel by the notch grid method according to PN-EN ISO 2409:2021-03,
9. flexibility testing of the coating according to PN-EN ISO 1519:2012,
10. determination of hardness by the pendulum method according to PN-EN ISO 1522:2023-02,
11. impact testing according to PN-EN ISO 6272-1:2011.

Results and discussion

Determination of drying times according to ASTM D1640

The method used in the study is only a reference method. It can't be compared with drying times given in technical sheets, since these are usually made with specialized equipment according to ASTM D5895, ISO 9117-4 or DIN EN 14022 standards. Despite the limitations of this method, it allowed to obtain an approximate estimate of paint drying times (Table 2).

Tab. 2. Drying times of investigated coatings according to ASTM D1640

Coating	Drying times	Time [min]	Standard deviation [s]
Cromax	set-to-touch	6	± 49
	tack free	11	± 28
	dry to touch	15	± 28
	dry to handle	110	± 393
Glasurit	set-to-touch	15	± 38
	tack free	21	± 64
	dry to touch	33	± 39
	dry to handle	214	± 229
Spectral	set-to-touch	9	± 28
	tack free	15	± 49
	dry to touch	27	± 97
	dry to handle	104	± 344

Source: own study.

Cromax achieved the shortest "set-to-touch time," which may mean using solvents that evaporate faster than in other products. This solution speeds up subsequent drying times but may also result in inadequate time for the coating to spill evenly. The Glasurit mixture achieved a tack free time of 21 minutes. It can be assumed that basecoats can be applied to it after this time without any problems. Glasurit contains IPDI polyisocyanate, which affects the drying times of the coating. It is not used in the other products. Therefore, it can be inferred that Glasurit's mixture uses acrylic resins that dry relatively slowly, or the amount of catalyst is small. It is assumed that topcoats should be able to be

applied 15 minutes after the last coat of primer is applied wet-on-wet. In the case of Cromax and Spectra mixtures, there are no deviations from this rule. For Cromax and Spectra products dry to handle time was less than 2 hours. A time of 2-3 hours is thought to be the typical time for this type of primer. The long dry-to-handle time may in this case mean that the company recommends wet-on-wet primer only.

Determination of the density

The density results allowed to determine the VOC content (point 3) per liter of the ready-to-use product. Results of density testing are shown in Table 3.

Tab. 3. Densities of primers and product mixtures

Product	Primer's density	Mixture's density
Cromax	1.601 ± 0.006	1.331 ± 0.005
Spectral	1.565 ± 0.008	1.235 ± 0.004
Glasurit	1.454 ± 0.003	1.234 ± 0.005

Source: own study.

Cromax primer had the highest density, such results may suggest that the product contains a higher proportion of fillers relative to resins, thinners, and additives, or more TiO₂ or BaSO₄ were used. In the paint industry, the typical specific gravity for primers is assumed to be between 1.55–1.65 g/cm³. An interesting result is Glasurit brand primer, which has a density of 1.454 g/cm³. This may suggest the use of lighter fillers than in other products. In addition, lower density has an impact on the VOC limit, since at a lower density, the solids content preserves by a wide margin the VOC.

VOCs determination

Testing for volatile organic compound (VOC) content is important due to their emission limits and product quality. In the case of varnishes and primers, the lower the VOC content, the more environmentally friendly these products are. This test also allows for product quality control, as incorrect ingredient proportions can affect these measurements. VOCs concentrations are presented in Table 4.

Tab. 4. Results of the VOCs determination in investigated products

VOC concentration		
Product	Average content of volatile organic compounds in 1l of mixture [g/l]	Standard deviation
Cromax	525.90	± 9.35
Spectral	562.17	± 5.48
Glasurit	536.55	± 4.0
The EU limit for this product group (category: IIB. C II) in the state ready for application is 540 g/l		

Source: own study.

Cromax and Glasurit met the requirements of Directive 2004/42/EC regarding VOC limits. Spectral, on the other hand, exceeded them. Compared to Cromax and Glasurit mixtures, the Spectral mixture will have a much greater negative impact on the environment.

Flow viscosity testing

The flow viscosity or conventional viscosity is used to check whether a product has the right viscosity for spraying. The wet-on-wet method usually paints products with a mixture that has a conventional viscosity using a DIN 4 cup between 16 and 21 seconds, sometimes 24 seconds.

It is often assumed in the paint industry that adding thinner to a product will improve its distribution and make the coating smooth. However, for the coating to be ultimately even, other factors such as surface tension, the type of surfactants used, and the type of resins and thinners also have an impact. Table 5 presents results of the flow viscosity of the three products.

Tab. 5. Flow viscosity of Cromax, Spectral and Glasurit

Product	DIN 4 conventional viscosity [s]	Manufacturer's stated viscosity (DIN 4; [s])
Cromax	22.87 ± 0.49	16–24
Spectral	17.40 ± 0.47	16–21
Glasurit	18.37 ± 0.52	16–19

Source: own study.

The viscosity of all tested products was in the range stated by the manufacturer. According to growing viscosity all products can be ranked Spectral < Glasurit < Cromax.

Dynamic viscosity by the Brookfield method

Rheological agents are usually used in primers to stop the sedimentation of fillers and pigments, the amount of which is considerable. Fillers, because of their density, tend to sink to the bottom, but rheological agents prevent this and allow the substance to mix easily.

Based on the results (Figure 1), it can be concluded that the liquids are thixotropic in nature.

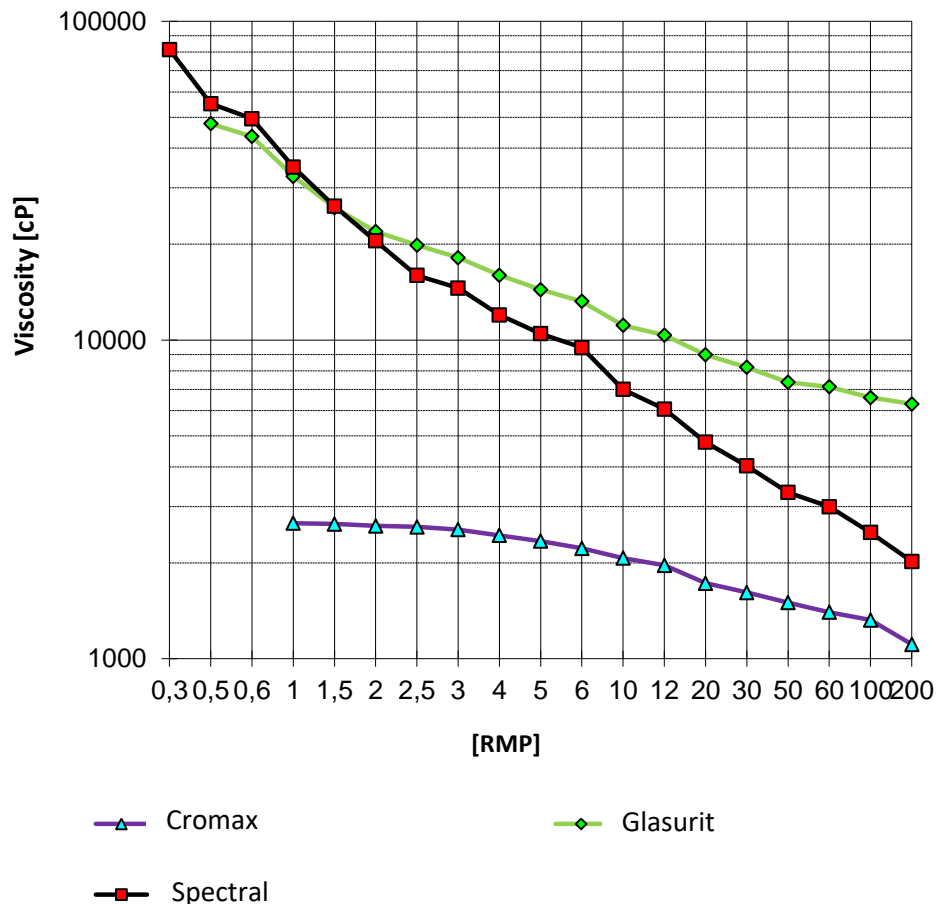


Fig. 1. Rheological profile of the tested primers

Source: own study.

Viscosity decreased as shear forces increased in all the tested products. Cromax showed the lowest viscosity compared to the other products. In the case of this primer, it was noted that a large amount of filler was deposited at the bottom of the can, which was compacted and required long mixing; unlike other products. It can be assumed that the low thixotropy is related to the high spray viscosity of the primer. At higher thixotropy, in turn, higher surface tension is observed. Low thixotropy also means low surface tension and low resin shrinkage after evaporation of thinners. With a lower surface tension, the product is prone to streaking. Presumably, the disadvantage in terms of sedimenta-

tion in this case was to be offset by low viscosity and plugging the product into an in-line mixer. Glasurit, on the other hand, was characterized by the highest thixotropy, which can be seen with the naked eye after light mixing and scooping on a stick, as the liquid does not flow, but rises. It's also worth noting that despite the highest thixotropy, this primer was much less viscous than Spectral's primer. It is worth noting that at 200 RMP, Spectral's viscosity was practically three times that of Glasurit. No filler deposits were noted in the Glasurit and Spectral products. After several months in samples left to stand, fillers and pigments did not settle. It is noteworthy that Glasurit, which despite being so thixotropic, was characterized by a smooth coating and there was no resin shrinkage after evaporation of the thinners. Moreover, the product's viscosity was low enough that it would be possible to plug it into an in-line mixer. In the case of Spectral, it is uncertain whether an in-line mixer with such a high viscosity would be able to move the primer.

Testing of ash content

As in the previous study, the primer samples themselves, rather than their mixtures, were tested to determine the content of fillers used in the products. High temperatures were used to burn off the polymers, leaving only the fillers. The results of this test are shown in Table 6.

Analyzing the results of Spectral and Glasurit, it can be concluded that the amount of fillers in both products is very similar, about 40%. However, due to the lower density of the Glasurit primer at the same dry matter content and amount of inorganic matter, it can be assumed that it was made with lighter fillers than the other primers. Cromax primer, on the other hand, contained less than 10% more fillers than the other products. It appears that despite the large amount of fillers, the PVC is within CVPC, as evidenced by the smooth surface of the coating. However, the increased filler content in the primer can negatively affect its properties, including bending resistance, which means less flexibility and greater susceptibility to mechanical damage such as impact. When coating a surface with such an underlayment, expect defects to appear more quickly. On the other hand, the advantage of higher filler content is lower production cost, which can translate into a lower price.

Tab. 6. Ash content in tested samples

Product	Exposition time [min]	Temperature [°C]	Ash content [%]	Standard deviation
Cromax	30	800	49.21	± 1.01
Spectral	30	800	40.23	± 0.58
Glasurit	30	800	39.75	±0.57

Source: own study.

Determination of the level of spreading

The optimal level of blending depends on several factors, such as the type of primer, the type of pigments and fillers, and the application conditions. The blending results are shown in the Table 7.

Tab. 7. Levels of spreading of tested products

Product	Level of spreading [μm]
Cromax	5.0
Spectral	7.0
Glasurit	5.0

Source: own study.

The low particle size in fillers indicates that the pigment will have little effect on unevenness of color and texture. A lower level of pigment spreading has a positive effect on the covering properties of a given primer. In the case of Cromax and Glasurit primers, these values are similar. In practice, carbon black pigments that are subjected to rubbing show better performance, and for gray primers they are present in each of the mentioned primers. A better surface texture, and greater uniformity of distribution, will be exhibited by primers with a 5.0 μm grind. A particle size of 7.5 μm , will have slightly worse listed properties. Smaller particle sizes will have a positive effect on sedimentation retention.

Determination of adhesion to steel

The primary attribute of the primers is that they can be applied directly to certain materials. In order to study the adhesion to steel of selected blends, the notch grid method was used, and the results are shown in Table 8.

Tab. 8. Adhesion to steel results of tested products

Product	Base	Classification (0–5)
Cromax	Steel	0
Spectral	Steel	0
Glasurit	Steel	0

Source: own study.

Primers are usually designed to have good adhesion properties, allowing a permanent bond between the coating and the substrate. A rating of 0 in the ISO 2409 method means very good adhesion, indicating excellent adhesion properties as shown in Figure 2.



Fig. 2. Photos: a) notch grid for Spectral, b) notch grid for Cromax, c) notch grid for Glasurit
Source: own study.

Flexibility testing of the coatings

Elasticity testing is one of the important tests to determine the quality of a coating material. In this study, assuming that the results would be between 2 and 6 mm, tests were performed on the same samples that were used in the pendulum hardness and adhesion tests. However, the results of the elasticity test turned out to vary quite a bit from one another (Table 9).

Tab. 9. Results of flexibility testing of tested products

Product	Diameter of the mandrel followed by coating defects [mm]
Cromax	10
Spectral	6
Glasurit	4

Source: own study.

Glasurit brand primer proved to be the most flexible. Considering previous tests, the primer that dries the slowest and has the lowest final hardness showed the greatest flexibility. It is puzzling how well the formulation was made by BASF that with isophorone diisocyanate the elasticity result is so good. All parts of a car "work" during the year, minimally changing their volume under the influence of temperature, thanks to the phenomenon of thermal expansion or because of stresses during driving. Therefore, coating materials must exhibit the right parameters to avoid cracking and crumbling. It happens that while driving, pieces of stones, sand or other small objects can hit the painted surface of the car and cause chipping or damage. Therefore, it is important that the coating be flexible and resistant to such damage. That's why there are various types of plasticizers for polyurethane products on the market that increase the flexibility of the coating. These additives help increase the coating's resistance to impact, preventing the appearance of spalling. Studies show that coatings with greater flexibility tend to have greater resistance to chipping.

Determination of hardness by the pendulum method

Testing the hardness of coatings is an important step in evaluating the quality of coating materials, since usually the harder the coating, the more resistant to abrasion. In this study, the hardness of the coating was determined based on the damping of the pendulum, which allowed us to determine the absolute hardness, or the time required to damp the pendulum, and the relative hardness, or the ratio of the damping time of the sample to that of the glass constant, which served as a reference. The samples were prepared using an automatic applicator, and the thickness of the wet coating was 150 μm . After analyzing the results, there can be noted some differences between the samples, but they are not significant (Figure 3).

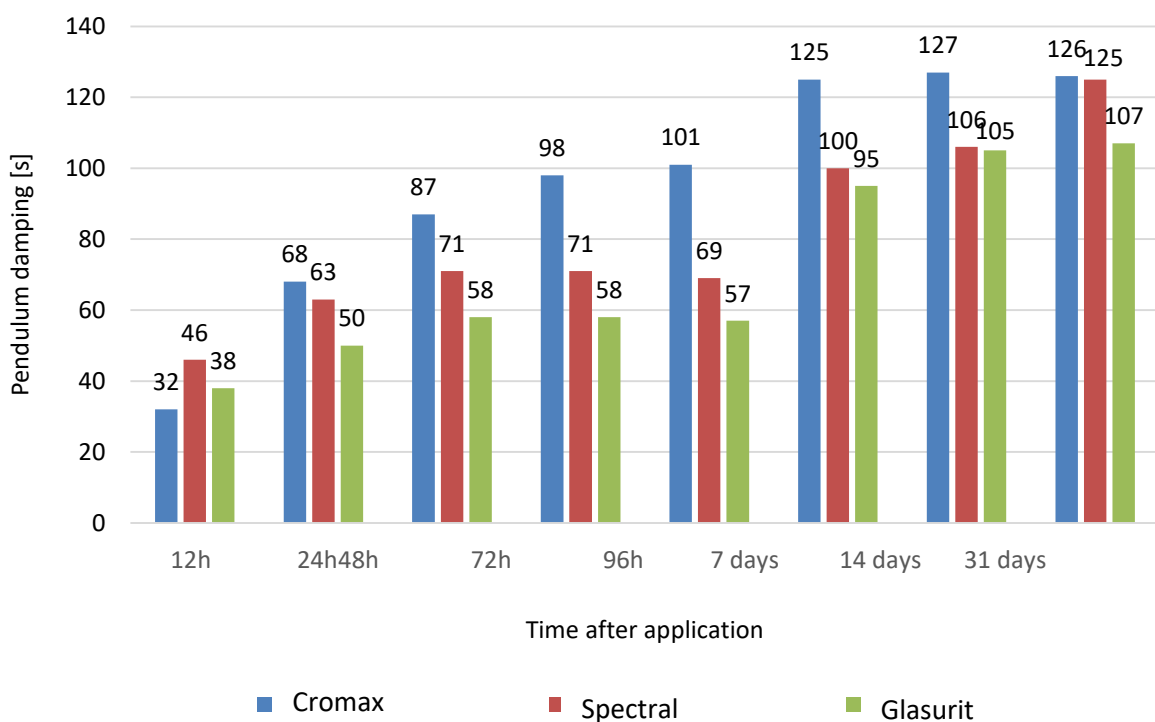


Fig. 3. Dependence of post-application time on pendulum fading value of tested products

Source: own study.

Particularly interesting results were obtained for the Glasurit mixture sample, since the hardness after 12 and 24 hours does not differ from that of the other samples and is even slightly lower. Since, IPDI isocyanates are characterized by the fact that significant differences can be observed with their use compared to ordinary aliphatic agents, one can only suspect that the resins used in the product are slow to dry or the catalyst content is low. Full cross-linking of coatings occurs 7 days after application.

Impact testing of obtained samples

Impact resistance testing is an important test because automotive coatings are constantly exposed to various types of mechanical damage while driving. These can include, for example, chipping due to stone impact, or scratches from contact with other vehicles or obstacles on the road. Results of impact tests are shown in Table 10 and Figure 4.

Tab. 10. Impact test results

Product	Impact resistance [N·m]
Cromax	0.50
Spectral	0.75
Glasurit	1.00

Source: own study.

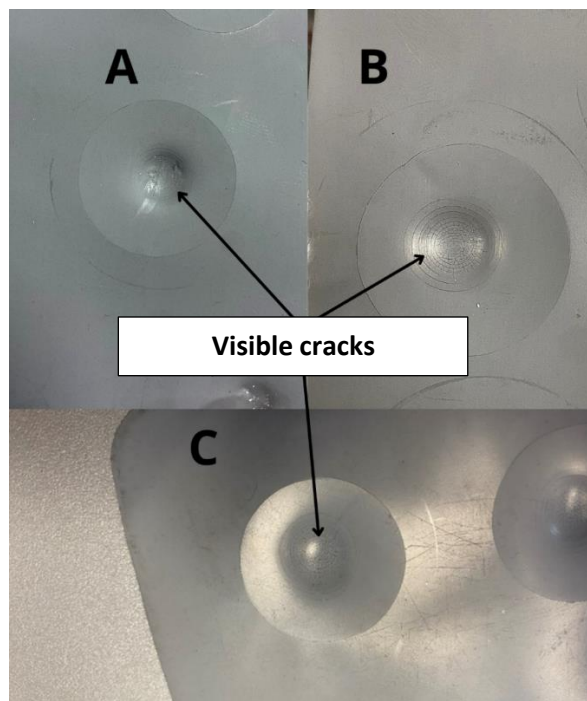


Fig. 4. Impact test results: a) Cromax, b) Spectral, c) Glasurit

Source: own study.

The test results showed that the Cromax compound had the lowest impact resistance. This is in line with previous studies, which showed that this primer had the highest final hardness and the least flexibility. The other mixtures showed higher impact resistance. This can have a significant impact on the life of the coating over time. In the short term, the differences will be small, but prolonged use of

the vehicle with the topcoat primer may lead to more coating defects than with the other two solutions. The Glasurit compound sample showed the highest impact resistance.

Conclusions

High differences in product rheology indicated that, unlike Glasurit and Spectral primers, Cromax had very low viscosity and thixotropy, which negatively affects the retention of sedimentation of pigments and fillers. On the other hand, the ash content test showed that Cromax had a high filler content and can have low mechanical resistance such as, high brittleness, and low flexural strength. The low particle size indicates that there is no difference in the texture and colour of the primers. The determination of non-volatile matter content, on the other hand, showed that each primer had high dry matter content and low volatile organic compound content, and can show good filling properties. The low density of Glasurit primer indicates that it contains lighter fillers compared to competitors' products, or that agents such as barium sulphates are absent from the formulation, which affects the mechanical properties of the product. Cromax contains epoxy resin and, like Glasurit bis[orthophosphate(V)] trizinc (($Zn_3(PO_4)_2 \cdot 2H_2O$)) primer, the additives positively affect the anticorrosive properties. In addition, the drying time determination, showed that the Glasurit mixture has significantly longer drying times than the other mixtures tested, which indicate the poor adaptation of the primer to the filler and primer system. In addition, Spectral exceeds the permissible level of the requirements of Directive 2004/42/EC on volatile organic compounds (VOCs), at the same time showing a negative impact on the environment. Hardness testing, showed that the mixtures ultimately achieve similar hardness. Glasurit showed slow crosslinking, which resulted in low hardness during curing of the coating, this characteristic confirms the possibility of problems when working in a filler system with this product. All mixtures had excellent adhesion to steel, and Glasurit formed the most flexible coating, which showed the greatest flexibility and impact resistance. The result of the impact test, on the other hand, confirmed that Glasurit's coating had the highest mechanical resistance compared to the other coatings, and the Cromax coating would be the most prone to defects over time among the competitors. Sensory evaluation of the coatings in the coating process, showed that the Cromax primer has the smoothest surface, its high flow viscosity of the mixture and sensory evaluation confirm the ability to coat optimal coatings at higher spray viscosity.

Acknowledgements

The research was performed by Adrian Kurek as part of his engineering thesis.

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THE INFLUENCE OF ANIONIC AND NONIONIC SURFACTANTS ON THE USAGE PROPERTIES OF HEAVY DUTY CLEANERS

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DOI: 10.56091/CTQS.Qual-12

Abstract

The article evaluates the effect of the anionic and nonionic surfactants on the performance of modern agents for removing tough dirt from surfaces. Formulations were developed in the form of emulsions containing fatty acid methyl esters derived from rapeseed oil as hydrophobic solvent. Anionic surfactants Triethanolamine Oleate, Sodium Oleate, and nonionic surfactants Sorbitan Monolaurate, lauryl alcohol ethoxylated with 7 moles of ethylene oxide (Laureth-7) were introduced into the formulations. On the basis of available literature data and own research, formulations of 14 prototypes of hard surface cleaners containing anionic surfactants in concentrations of 16, 18, 20, 22% by weight and non-ionic surfactants in concentrations of 8, 10, 12% by weight in the formulation were developed. The following were evaluated for the composed products: stability, viscosity, water solubility, efficiency of application and distribution over the cleaned surface, dirt removal efficiency. The obtained test results were related to those obtained for two commercial products. The formulations developed were stable, no changes were observed in the appearance of samples stored for a period of 6 months. The tests of usage properties allow us to conclude that the highest efficiency of dirt removal from cleaned surfaces, were characterized by preparations containing in their composition 22% by weight of sodium oleate and 12% by weight of ethylated lauryl alcohol. For these samples, optimal viscosity, water solubility and efficiency of spreading the formulation on the surface were obtained, comparable to their commercial counterparts.

Keywords: heavy duty cleaner, quality, anionic surfactants, nonionic surfactants

Introduction

The market for household chemicals in Poland, in the last few years, has been developing very intensively and this trend is still growing. You can see an increase in demand for both universal cleaning products and specialized products designed for thorough cleaning of strictly defined surfaces. Nowadays, consumers are guided by different factors when choosing cleaning products than just a few years ago (Friis & Skagerlind, 2008). It is no longer the price that is most important, but the effectiveness, quality and efficiency of the products chosen. The market for household chemicals is saturated with novelties, so in order to stay in the market, companies are improving their formulations, in accordance with the directions of current development and the needs of customers, in order to offer increasingly

better and more effective products. This often involves the introduction of ingredients that, while improving product quality, are often not indifferent to consumer health. Such products include cleaners containing: organic solvents, oxidizing agents, pH adjusters, bleaching or sanitizing additives (Kubiak et al., 2007).

Consumers' expectations about the safety of product use and its environmental impact are also increasing. The changing policies of companies and, above all, the requirements of the European Union, mean that more and more investment is being made in products made from natural, non-toxic to humans and biodegradable raw materials (Małysa & Zatorska, 2022). Household chemical products placed on the Polish market must meet the safety requirements of Directive 2001/95/EC of the European Parliament and of the Council of December 3, 2001 on general product safety. There are also Polish Standards, concerning household chemical products, the application of which is not mandatory (Sułek et al., 2008)

A significant group, among household chemical products containing solvents, are cleaning agents. The basic factor determining effective cleaning is the correct selection of the agent used for the type of stain to be removed and the surface to be cleaned. By the term "cleaning" we can understand the activity leading to the removal of stain from the surface, by putting additional energy, for example, in the form of mechanical work (Tomaszkiewicz-Potępa & Mitoraj, 2007).

Therefore, there is a division of cleaning agents according to the type of stain into: light duty (used for easily removable stain) and heavy duty (used for hard-to-remove stain). The first of these groups are universal cleaning products used to remove: dust, dirt, dust, etc. Heavy duty products are used for cleaning: polymerized dirt, calcium deposits, caramelized sugars, rust, ironing, etc. When developing the formulation of the products in question, it is important to know the chemical composition of the soiling, its structure and interactions with the cleaned surface. It is important whether the dirt is water-soluble or not. Water-soluble dirt can include simple carbohydrates, some complex sugars, as well as salts, i.e. sulfates, chlorides. Much more difficult to remove are soils that have changed structure under the influence of external factors, i.e. temperature, pH, or oxygen access. Physicochemical transformations that occur in such dirt can be described by the phenomenon of drying, during which the particles agglomerate and the solvent evaporates. The surface area of the soiling decreases and, at the same time, it is more difficult to remove. Important in the selection of a cleaning agent is its adaptation to the type of surface to be cleaned. For surfaces that are not sensitive to low or high pH, such as ceramic, enamel surfaces, strongly acidic or strongly alkaline preparations are used. They have excellent properties for removing dirt without scrubbing, but we can't use them for lacquered coatings or alumi-

num. The requirements for such preparations are high: they must not damage, scratch or chemically react with the surface (Dmytryshyn et al., 2004; Klimaszewska et al., 2016, Klimaszewska et al., 2017)

A separate problem is the relationship between the composition of the cleaning and washing preparation, its form and the way it is used. The form in which cleaning products are produced is very important and plays the role of a factor that facilitates application and increases the comfort of use. It also has a huge impact on the marketing qualities of the product. The classification of cleaning products in consumer terms is done mainly by evaluating the form in which the product is present. Visual evaluation is also important, especially the shape, color and smell of the cleaning agent. Even the best-cleaning scouring paste, if it is gray, in unattractive packaging, will not be a well-selling commercial product. Among cleaning products present on the Polish market and commonly used in households, there are three basic forms: pastes, milks and powders. Innovative forms such as gels, foams and aerosols are used less frequently, but fill the niche of specialized products for the needs of industries such as automotive, furniture, metal, printing, paints and varnishes (Sułek & Klimaszewska, 2008; Sułek & Małysa, 2010).

Pastes are all-purpose cleaners with relatively high viscosity. Such products do not run off cleaned surfaces, distribute well and work precisely where they are applied. In the past, these products were very popular and often used in households and industrial plants, but are now being displaced by newer forms such as milks and powders. Soap pastes, used as handwashing and handwashing agents, still exist on the market today. They are effective in contact with contaminants such as solid fats, oils, greases, oil paints, soot, tars, which is why they have found use as hand wash and power wash – in washing machines. They tend to have a greasy consistency, are characterized by resistance to hard water and good solubility, even at relatively low temperatures (Przondo, 2007; Sułek et al., 2008).

This paper presents the effect of anionic and non-ionic surfactants on the performance of hard surface cleaners. In the paper they will be interchangeably referred to as cleaning pastes, cleaning agents, cleaning emulsions, heavy duty cleaners.

Materials and methods

Materials

The testing materials were emulsion-based heavy duty cleaners containing fatty acid methyl esters derived from rapeseed oil as hydrophobic solvent. The formulations included anionic surfactants (INCI: Sodium Oleate, Triethanolamine Oleate) and non-ionic surfactants (INCI: Sorbitan Monolaurate, Laureth-7). On the basis of available literature data and our own research, we developed formulations of 14 prototypes of hard surface cleaners.

In the first stage of the work, 4 cleaning formulations containing anionic surfactants were prepared: Sodium Oleate at concentrations of 16, 18, 20, 22% by weight in the formulation (formulations 1–4) and formulations containing Triethanolamine Oleate at concentrations of 16, 18, 20, 22% by weight. Based on visual evaluation, it was decided that prototypes containing 22% by weight Sodium Oleate were selected for further testing.

In the second stage of the study, formulations of heavy duty agents containing non-ionic surfactants (INCI: Laureth-7 and Sorbitan Monolaurate in concentrations of 8, 10, 12% by weight were developed (formulas: 9, 10, 11 and 12, 13, 14, respectively) and Sodium Oleate at 22% by weight. In order to obtain an emulsion form, water desylated to 100% by weight percent was introduced.

A summary of the formulations of the original cleaning agents are shown in Table 1.

Tab. 1. Recipes of original heavy duty cleaners

Formulation Ingredients (INCI)	% by weight													
	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Rapeseed Fatty Acid Methyl Esters	14													
Sodium Oleate	16	18	20	22					22			22		
Triethanolamine Oleate					16	18	20	22						
Laureth-7									8	10	12			
Sorbitan Monolaurate												8	10	12
Aqua	up to 100													
Preservative	0.2													

Source: own research.

Two commercial products that contained hydrophobic solvents in the formulation were used to compare performance with the original formulations, designated TP1 and TP2.

Methods

Stability

Visual evaluation of emulsions consisted of observing their appearance, homogeneity and signs of instability, such as stratification, creaminess and coalescence, among others. It also allowed observation of the appearance of mold or other signs of microbial contamination present in the preparation. Visual evaluation of the prepared emulsions was carried out immediately after making them, and after 6 months (Marcinkowska et. al, 2006).

Load temperature tests allowed visual evaluation of the stability of preparations stored alternately at elevated (40°C, 1 day) and reduced (5°C, 1 day) temperatures. They were carried out in an ST-68 type hothouse and a refrigerator. The test lasted 8 days (4 full cycles) (Sulek & Klimaszewska, 2008).

Viscosity

Dynamic viscosity coefficient measurements were carried out using a Brookfield viscosity meter type HA DV III Ultra. Measurements were performed at 21°C at a spindle speed of 5 rpm using Helipath type spindles (Klimaszewska et al., 2017).

Water dissolution

The test consisted of determining the time required for the paste to completely dissolve in water, while stirring at 100 rpm. In a 150 ml beaker, 100 g of distilled water was poured and 2 cm³ of the test paste was added, applied with a syringe, to a stirring paddle set in motion. Then the time of complete dissolution of the paste in water was measured. Measurements were made at 22°C. The final result of the determination was the arithmetic average of the measurements (Klimaszewska et al., 2017).

Application and spreading of preparations on the cleaned surface

The effectiveness of applying and spreading the preparation on the cleaned surface was evaluated as follows as follows: a smooth ceramic tile measuring 15 cm x 15 cm was prepared and with a surface roughness of 0.36 µm. The surface of the plate was washed in an aqueous detergent solution and additionally degreased with ethyl alcohol. The plate thus prepared was placed in a horizontal position and was fixed to the substrate. Then, on a clean and moistened sponge measuring 5 x 8 cm and 2 cm thick, 4 g of the test preparation, weighed out with an accuracy of 0.01 g. The surface of the sponge with the applied preparation was applied to the surface of the ceramic tile. Then 5 circular movements were made, trying to make the cleaned area cover as much of the tile surface as possible. Then, the efficiency of application and spreading of the preparations was estimated according to the scores shown in Table 2. The measurements were repeated 3 times (Sulek & Małysa, 2010).

Tab. 2. Scoring evaluation of the efficiency of application and spreading of the product on the surface of the ceramic tile

Points	Assessment criteria
0	The product cannot be applied with a sponge and does not yield to spreading.
1	The product is difficult to apply to the sponge and smears over the surface or does not cover it evenly, staying on the sponge.
2	The product spreads easily over the surface and covers it evenly.

Source: Sułek & Małysa, 2010.

Stain removal efficiency

The stain removal efficiency of the tested formulations was evaluated based on the ability of the formulation to remove model dirt from a ceramic tile. For this purpose, ceramic tiles measuring 15 cm x 15 cm and with a surface roughness of 0.36 cm were prepared. The surface of the tile was washed in an aqueous detergent solution and additionally degreased with ethyl alcohol. Model stain were then prepared. The stain according to the test, proposed by IKW (The German Cosmetic, Toilet-ry, Perfumery and Detergent Association, Section Cleaning and Maintenance Products). Dirt was prepared immediately before the tests were performed. Type I dirt was made by thoroughly mixing the following ingredients: 34 g of milk margarine, 34 g of wheat flour, 20 g of milk powder, 7 g of chicken egg yolks and 50 g of distilled water. Next, 12 g of soil (weighed to the nearest 0.01 g) was applied to the surface of one plate, and then spread evenly over the entire surface. The plates prepared in this way were left to dry at room temperature (22°C) for 24 hours.

The plates prepared in this way were subjected to cleaning. For this purpose, 2 g of paste and 4 g of distilled water were applied to a sponge measuring 5 x 8 cm and 2 cm thick, 4 g of polish and 2 g distilled water, 2 g powder and 6 g distilled water, weighed to the nearest 0.01 gram. The surface of the applied sponge was applied to the center of the ceramic tile surface and weighed with a 1 kg weight. Then 60 reciprocating movements were made from one end of the tile to the other. After the cleaning process, the plate was rinsed with running water and dried. The test was conducted at room temperature. Then, on the basis of visual observation, the cleaning efficiency was evaluated according to the adopted scale (Table 3). Each measurement was repeated 3 times (Sułek & Klimaszewska, 2008; Sułek & Małysa 2010).

Tab. 3. Scoring scale used in the cleaning efficiency study

Points	Assessment criteria
0	No visible effect of the product on the stain
1	Disturbed stain structure, visible removed layer of stain, barely discernible clearance of white tile in place of application of preparations
2	Disturbed stain structure; visible removed stain layer, discernible white tile clearance at application site
3	Disturbed structure of the stain, visible but indistinct removed layer of stain; discernible clearance of white tile in the area of sample application; possibly a wide area of removed stain (about 2 cm in diameter) with not necessarily good clarity, the stain was not completely removed
4	The structure of the dirt has been severely affected, very clear clearance of the white tile, possibly a wide area of removed dirt (diameter of about 2cm), the dirt has not been completely removed
5	The structure of the stain has been severely affected, very clear clearance of the white tile, possibly a wide area of removed stain (diameter of about 4 cm) characterized by good clarity, the stain has not been completely removed
6	In the place where the preparations were applied, the dirt was completely removed

Source: Sułek & Klimaszewska, 2008.

Result and discussion

Stability

Evaluating the stability over time of cleaning agents is the most important quality parameter during storage and use. This stability refers to the ability of an emulsion to maintain a homogeneous combination of aqueous and oil phases for a specified period of time without either phase precipitating, separating, or changing its appearance or consistency.

The results of stress tests (elevated and reduced temperature) showed full stability of all tested compositions. No signs of microbiological and physicochemical instability were observed, and it was found that all heavy duty cleaners prototypes were characterized by satisfactory stability over time.

Viscosity

Viscosity (η) of heavy duty cleaners is a very important parameter from the point of view of their performance, especially dosage and application on the cleaned surface. Viscosity is derived from its composition, mainly viscosity regulators, as well as components that perform other functions in the product, such as surfactants and solvents. The effect of the type of anionic and nonionic surfactants on the dynamic viscosity of the original and commercial products was evaluated. The results of η measurements are shown in Figure 1.

The effects of anionic and nonionic surfactants on the dynamic viscosity of the original hand wash products were studied. Two commercial products, TP1 and TP2, were used as reference points in the evaluation. For the commercial products, the measured viscosity values oscillated in the range of 22 000–27000 mPa · s. An increase in viscosity from about 14 000–25 000 mPa · s was observed as the concentration of Sodium Oleate in the formulation increased. For cleaners containing Sodium Trioleate as an anionic surfactant, viscosity values reached the lowest viscosity value relative to all other samples and ranged from about 12 000 mPa · s to about 13 000 mPa · s. The highest viscosity values of 32 000 mPa · s were achieved for prototypes containing Sodium Oleate and Sorbitan Monolaurate in the formulation (formulations 12–14). Nearly twice the viscosity values discussed above were obtained for cleaners containing Sodium Oleate and Laureth-7 (formulas 9–11).

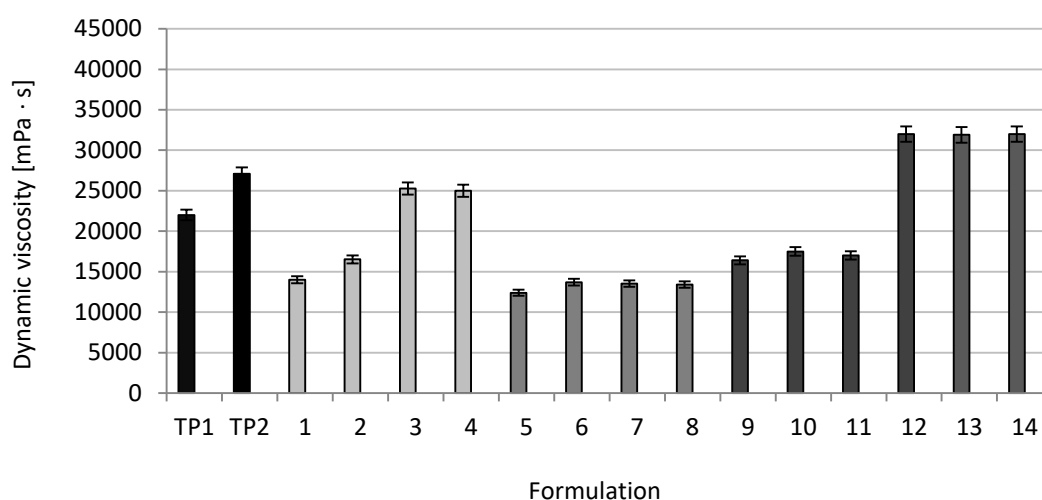


Fig. 1. Dynamic viscosity of original heavy duty cleaners and trade products

Source: own research.

Water dissolution

After cleaning the surfaces with pastes, they were rinsed with water. Therefore, it is important that the formulations have the ability to dissolve in water. The effect of the content of anionic and non-ionic surfactants on the solubility of cleaning pastes made according to the recipes in Table 1 was investigated. The results of the study are shown in Figure 2.

Dissolution times of about 13–15 seconds in water were obtained for commercial products. Very similar values of dissolution time were obtained for cleaning products made according to recipes 1–11 (Table 1). Almost three times higher values of the parameter in question were obtained for prototypes containing Sodium Oleate and Sorbitan Monolaurate (formulations 12–14).

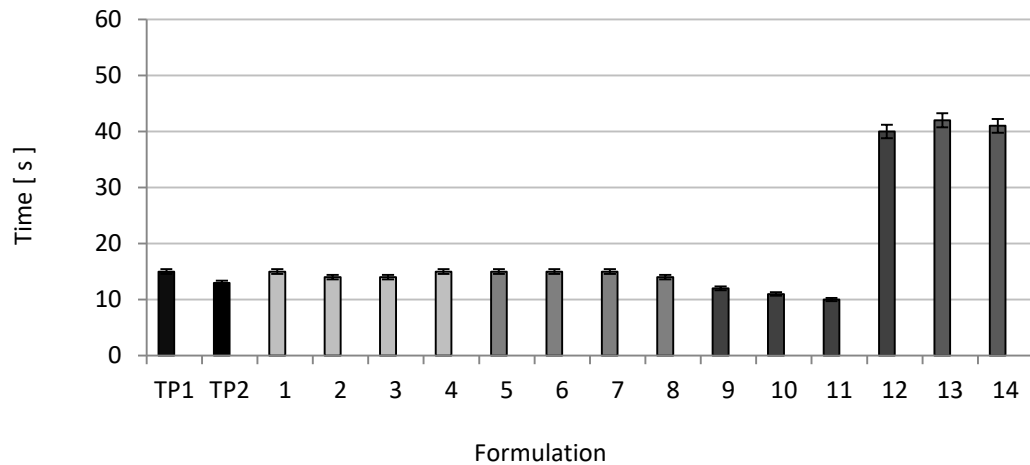


Fig. 2. Water dissolution of original heavy duty cleaners and trade products

Source: own research.

Application and spreading of preparations on the cleaned surface

The efficiency of application and spreading of pastes is an important distinguishing feature of their quality. The test of application and spreading efficiency of the formulations developed (formulations according to Table 1) consisted of visual observation of the application and spreading of the preparation, according to the point scale (0–2 points) presented in Table 2. The results of the study are shown in Figure 3.

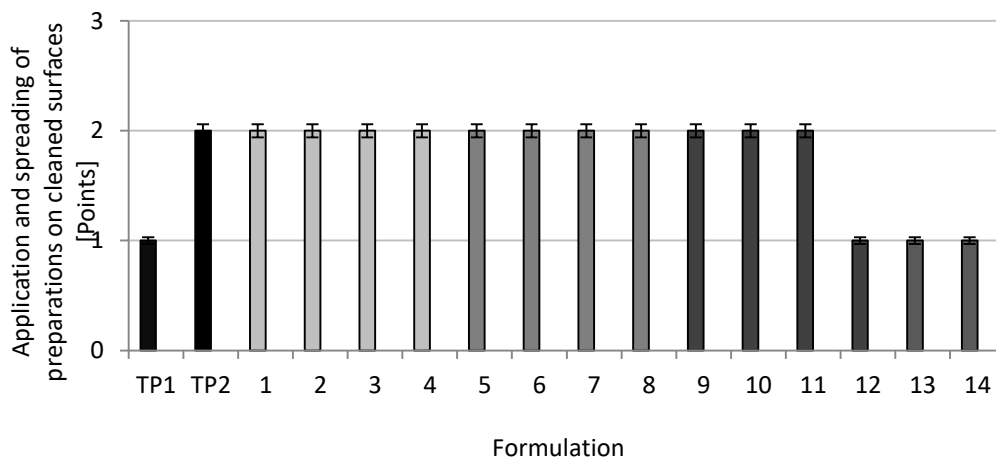


Fig. 3. Application and spreading of original heavy duty cleaners and trade products on cleaned surfaces

Source: own research

It was observed that the highest score of 2 points was obtained for the commercial product TP2 and preparations made according to formulas 1–11 (Table 1). Slightly worse (at the 1 point level) was the overlap and distribution of cleaning products made according to formulas 12–14 (Table 1) and the commercial product TP1.

Stains removal efficiency

High stains removal efficiency is the most important distinguishing feature of modern cleaning agents. The primary purpose of cleaning is the mechanical removal of dirt, originating from the external environment, which accumulates on surfaces. During the cleaning process, the solvents included in the pastes penetrate the into the greasy dirt, and the presence of anionic and nonionic surfactants helps remove stains from the cleaned surfaces. The effect of anionic and nonionic surfactants on the parameter in question is shown in Figure 4.

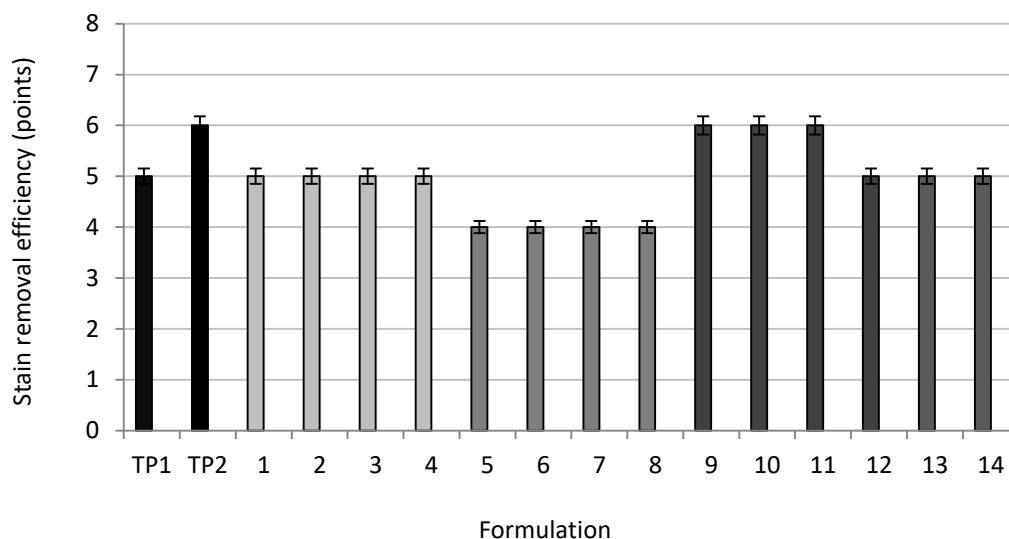


Fig. 4. Stain removal efficiency of original heavy duty cleaners and trade products

Source: own research

Based on the results obtained, it was found that the highest cleaning efficiency at the level of 6 points was shown by the commercial product TP2 and the original preparations made according to formulas 9–11. These preparations contained in their composition anionic surfactant – Sodium Oleate at a concentration of 22% by weight and as a nonionic surfactant: Laureth-7 (formulas 9–11). Relatively good stain removal capabilities at the 5 point level were shown by the commercial product TP1 and preparations made according to formulas 12–14 (Table 1). Stain removal efficiency at the level of 4 points were shown by preparations 5–8 (Table 1). Cleaning properties at level 4 points were shown by preparations 5–8, containing only one type of surfactant in the form of Triethanolamine Oleate (Table 1).

Conclusions

The result of the research was the development of 14 formulations of original heavy duty agents for removing stain from hard surfaces. It has been shown that the addition of an anionic surfactant in the form of Sodium Oleate and the nonionic surfactant Laureth-7 makes it possible to obtain hard surface cleaners with the most favorable functional properties: viscosity, water solubility, efficiency

of application and distribution over the surface and cleaning of model stain. The obtained prototypes were comparable in terms of performance properties to their market counterparts.

The use of developed methodologies for comprehensive assessment of the quality of cleaning agents, in their original form or with minor modifications, can be used by household chemistry producers to assess the quality of a wide range of cleaning products. This is valuable information due to the lack of legal regulations and standardized methods for assessing the quality of this type of products.

Acknowledgments

The article has been prepared under Project no. 3086/35/P “Development of formulations and technologies for the manufacture of innovative cosmetics, pharmacy supplies, household and industrial chemicals”.

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THE ESTIMATION OF THE SHELF LIFE OF NATURAL COSMETIC WITH VEGETABLE OIL AND TOCOPHEROL

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DOI: 10.56091/CTQS.Qual-13

Abstract

Shelf life is recommended time period that a cosmetic product can be kept after its production, during which the defined quality of the product remains acceptable under expected conditions of distribution, storage, display and usage. In this work, an attempt was made to estimate the shelf life of natural cosmetic – care oil stabilized with tocopherol. The oxidative and physicochemical stability including visual assessment, density and peroxide value determinations were estimated for products stored for 12 weeks in different conditions: $4\pm 0.5^{\circ}\text{C}$, RT+UV (room temperature with UV access), and $40\pm 1^{\circ}\text{C}$. At all storage temperatures (4°C , RT+UV and 40°C) tocopherol at tested concentrations: 0.05%, 0.075% and 0.1% effectively inhibited the peroxide formation in care oils. Shelf-life of tested care oils can be estimated at 18 months.

Keywords: oxidative and physicochemical stability of care oils, shelf life, peroxide value, natural antioxidants

Introduction

The natural cosmetics market is constantly growing, which is due to the fact that it has become ingrained in the consumer awareness as safe and environmentally friendly. Customers are increasingly willing to buy products containing raw materials of natural origin and are often able to spend more money on them than a few years ago. Confidence in certifications placed prominently on packaging is growing. The sector still has potential for growth, as consumers continue to expect new solutions (Amberg & Fogarassy, 2019; Rudnicka, 2021). Although there is no unified definition of a natural cosmetic, there are definitions of raw materials of natural origin according to International Standard 16128-1 and 16128-2, and often the declaration of their percentage content in a cosmetic is used in marketing communications. It makes that many cosmetics are perceived as “natural”. Cosmetics containing ingredients of natural origin, high concentration of vegetable oils or plant extracts) are a challenge for formulators due to the varying stability of these products.

Stability studies are aimed at assessing the ability of a product to maintain the desired physical, chemical and microbiological properties, as well as functionality and sensorial properties when stored and used under appropriate conditions by the consumer. The objective of a stability study is to determine the shelf life of a product and to evaluate whether a product in the package is stable when subjected to the market conditions in which it is sold and used. The “market conditions” encompass distribution, warehouse storage and conditions during use (ISO 18811:2018). The different criteria such as colour, odour and appearance, changes in the packagings, pH, viscosity, weight changes, microbiological changes and many other depending on the tested product are assessed during stability tests. The important issue is predicting functionality of the product under stress conditions such as temperature extremes and light. Temperature cycling or “freeze-thaw” tests can reveal some types of inadequacies more quickly than can storage at a constant temperature. As products can be expected to encounter temperature and pressure extremes during transport and storage, stability testing at these extremes should be considered, such as low-temperature and high-temperature testing, as well as freeze/ thaw testing. Cosmetics whose packaging may allow the product to be exposed to light are undergone light stability testing (CTFA, 2004; Wąsowska, 2011; Jakubowska-Stokowska, 2012). During storage cosmetics are submitted to change in chemical, microbiological and sensory respect. Depending on the type of formula we can observe emulsion breakdown, pH, perfume, colour and viscosity changes, fungal growth, wax crystallization, sediment in clear lotions, loss of activity of vitamins, antioxidants, preservatives, photo-degradation of UV filters or microbial growth (Lautenschläger, 2008; Kirkbride et al., 2021).

The aim of this work was to determine the shelf life of natural care oils by examine the oxidative and physicochemical stability of formulas containing soybean oil, prepared based on different concentrations of tocopherol.

Studies on oxidative and physicochemical stability of cosmetics with high concentration of vegetable oils are important step in development of safe and high quality formulations. Care oils are a special kind of product, that lipid oxidation can impact product quality (mainly odour and colour) and, potentially, safety of the formulations. High oxidative and physicochemical stability of care oil can also maintain product efficacy. Lipid oxidation can be triggered or enhanced by light and/or high temperatures. On the one hand, unsaturated fatty acids from the oil can nourish the skin, on the other hand, oxidation products can lead to skin irritation and aging (Thanonkaew et al., 2015; Thomsen et al., 2017). Therefore, it is important to limit lipid oxidation and to extend the shelf life of skin care products using natural antioxidants. In addition, some natural antioxidants can give the skin product added functional value e.g. vitamin E (Mukherjee et al., 2011).

Materials and methods

Refined soybean oil (from Gustav Heess), Tocomix L50-IP (from Jan Dekker) and fragrance oil (from Givaudan) were used to preparation of care oils. The characteristics of raw materials are presented appropriately in Tables 1–4.

Tab. 1. Fatty acids content in soybean oil

Fatty acids (%)	Specification	Result
14:0 myristic acid	max. 0.2	0.1
16:0 palmitic acid	9.0–13.0	10.2
16:1 palmitoleic acid	max. 0.3	0.1
18:0 stearic acid	2.5–5.0	4.5
18:1 oleic acid	17.0–30.0	23.1
18:2 linoleic acid	48.0–58.0	53.4
18:3 linolenic acid	5.0–11.0	7.2
20:0 arachidic acid	max. 1.0	0.4
20:1 eicosenoic acid/gadoleic acid	max. 1.0	0.2
22:0 behenic acid	max. 1.0	0.4

Source: Certificate of Analysis, Gustav Heess, 2023.

Tab. 2. Physical and chemical characteristics of soybean oil

Parameter	Specification	Result
Appearance at 20°C	clear, oily liquid	Passed
Colour	light-yellowish to yellowish	Passed
Odour	characteristic, rancid not accepted	Passed
Acid value	max. 0.5 mg KOH/g	0.08
Peroxide value	max. 4.0 meq O ₂ /kg	0.6
Refractive index (20°C)	ca. 1.475	1.4748
Refractive density (20°C)	ca. 0,922	0.922
Gardner colour number	<3	1.5

Source: Certificate of Analysis, Gustav Heess, 2023.

Tab. 3. Physicochemical properties and tocopherol content in Tocomix L50-IP

Parameter	Specification	Results
Appearance at 20°C	clear-slightly hazy liquid	Passed
Colour	yellow-brown	Passed
Odour	typical	Passed
Relative density (20°C)	0.931–0.950 g/cm ³	0.9350
Refractive index (20°C)	1.488–1.500	1.4902
Benzo(a)pyrene	ppb (µg/kg) max. 2	Passed
PAH-4	ppb (µg/kg) max. 10	Passed
Total content d-mixed tocopherols	%	51.1%
Tocopherol breakdown		
d-alpha tocopherol	%	4.7
d-beta and -gamma tocopherol	%	33.9
d-delta tocopherol	%	12.5

Source: Certificate of Analysis, Jan Dekker, 2023.

Tab. 4. Physical and chemical characteristics of fragrance oil

Parameter	Specification	Results
Appearance at 20°C	clear liquid	Passed
Colour	colourless to very slightly yellow	Passed
Odour	corresponds to standard	Passed
Relative density (20°C)	0.9655–0.9755 g/cm ³	0.9681
Refractive index (20°C)	1.4463–1.4563	1.4488

Source: Certificate of Analysis, Givaudan, 2023.

In this work it was decided to use only a natural antioxidant to maintain the oxidative stability of care oils, as it is safer for sensitive skin and it does not cause the skin allergy. Commission Regulation (EU) 2022/2195 limited the use of synthetic BHT by allowing its use at a maximum concentration in mouthwash products up to 0.001%, toothpastes up to 0.1% and in other leave-on and rinse-off products up to 0.8% according to the SCCS opinion (Scientific Committee on Consumer Safety SCCS/1636/21), which considered BHT (butylated hydroxytoluene) as safe at these concentrations. The use of BHT in skin care oils would be possible at lower than 0.8% effective concentrations, but market trends strongly favor the use of natural antioxidants. Commercial natural antioxidant Tocomix L50-IP is a liquid, 50% concentrate of d-alpha-tocopherol, d-beta-tocopherol, d-gamma-

tocopherol and d-delta-tocopherol in sunflower oil. It is commercially used antioxidant in food and cosmetics. Recommended by producer in-use concentration in fats and oils is 200–1000 ppm (0.02–0.1%) (Product Data Sheet Jan Dekker, 2021).

Care oils were prepared according to the formulations shown in Table 5. The control sample was prepared without an antioxidant. All prepared care oils were physically stable at the beginning of the test.

Tab. 5. Composition of the formulations of care oils

Ingredients (INCI)	Formulations (% w/w)			
	0.05%	0.075%	0.1%	Control sample
Glycine soja (Soybean) oil	99.7	99.675	99.65	99.75
Tocopherol, Helianthus annuus (sunflower) oil (Tocomix L50-IP)	0.05	0.075	0.10	-
Parfum	0.25	0.25	0.25	0.25

Source: own study.

Prepared care oils were transferred to transparent, PET bottles and they were stored for 12 weeks in different conditions: $4\pm 0.5^{\circ}\text{C}$, RT+UV (room temperature with UV access), and $40\pm 1^{\circ}\text{C}$. At the beginning of the test and periodically every 4 weeks for 12 weeks the visual and sensory assessment, density and peroxide value determinations were conducted.

The following parameters such as the appearance of the oils (possible changes as thinning, streaks, turbidity, sedimentation) and the colour of the oils (possible changes as discolouration, darkening, brightening) were evaluated during the visual assessment of the products. Sensory assessment consisted of evaluation of keeping olfactive profile and stability of the fragrance oil in care oil formulas, possible changes as off-odour, stronger and weaker odour of the tested samples.

Peroxide value (PV) in care oils was checked at the beginning of the test, and then it was determined periodically every 4 weeks. Peroxide value (PV) was determined by iodometric method according to the PN–ISO 3960:1996 standard (1996). Briefly, to 2.000 g of care oil, 10 ml of chloroform was added and stirred to dissolve. Then, 15 ml of glacial acetic acid and 1 ml of saturated potassium iodide solution were added and stirred. After 5 minutes, 75 ml of redistilled water was added and the solution was titrated with 0.002 M sodium thiosulphate solution, in the presence of the starch as an indicator, until getting decolourized.

Peroxide value (PV) was expressed as milliequivalents of active oxygen per kilogram of sample and it was calculated according to the following equation:

$$PV = \frac{(V_1 - V_o) \cdot C}{m} \cdot 1000 \quad (\text{meq O}_2/\text{kg})$$

where:

V_1 – the titrant volume used for titration of oil sample (ml),

V_o – the titrant volume used for titration of blank (ml),

C – the titrant concentration (mol/l),

m – the mass of oil sample (g).

The protective effect of Tocomix L50-IP on oxidative stability of care oils was expressed as Protection Factor (PF), calculated according to the following equation:

$$PF = \frac{T_{sample}}{T_{control}}$$

where:

T_{sample} – the time necessary to increase the peroxide value to 4 meq O_2 /kg in sample,

$T_{control}$ – the time necessary to increase the peroxide value to 4 meq O_2 /kg in control sample.

The Protection factor (PF) value indicates antioxidant or pro-oxidant activity:

- $PF > 1$ – an antioxidant protects care oils from oxidation,
- $PF = 1$ – lack of protective effect of an antioxidant,
- $PF < 1$ – an antioxidant acts as pro-oxidant (Szukalska, 2003).

The results were reported as mean \pm standard deviation of three independent determinations. All data were analyzed by SPSS Statistics 14.0 software program.

Results and discussion

The results of visual and sensory assessment of care oils conducted at the beginning of the test and periodically for 12 weeks are shown in Tables 6–9.

Tab. 6. The visual and sensory assessment of care oils containing 0.05% Tocomix L50-IP stored at different temperatures for 12 weeks

Formula with 0.05% Tocomix L50-IP	4°C	RT+UV	40°C
0 days	clear, light yellow, homogeneous oil, without mechanical impurities, pleasant odour		
4 weeks	clear, light yellow, homogeneous oil, without mechanical impurities; no changes in colour and odour	clear, homogeneous oil, without mechanical impurities; slight brightening of yellow colour, no changes in odour	clear, light yellow, homogeneous oil, without mechanical impurities; no changes in colour and odour
8 weeks		clear, homogeneous oil, without mechanical impurities; slight brightening of yellow colour, no changes in odour	clear, light yellow, homogeneous oil, without mechanical impurities; no changes in colour and odour
12 weeks		clear, homogeneous oil, without mechanical impurities; slight brightening of yellow colour, no changes in odour	clear, homogeneous oil, without mechanical impurities; slight darkening of yellow colour, no changes in odour

Source: own study.

Tab. 7. The visual and sensory assessment of care oils containing 0.075% Tocomix L50-IP stored at different temperatures for 12 weeks

Formula with 0.075% Tocomix L50-IP	4°C	RT+UV	40°C
0 days	clear, light yellow, homogeneous oil, without mechanical impurities, pleasant odour		
4 weeks	clear, light yellow, homogeneous oil, without mechanical impurities; no changes in colour and odour	clear, homogeneous oil, without mechanical impurities; slight brightening of yellow colour, no changes in odour	clear, light yellow, homogeneous oil, without mechanical impurities; no changes in colour and odour
8 weeks		clear, homogeneous oil, without mechanical impurities; slight brightening of yellow colour and odour	clear, light yellow, homogeneous oil, without mechanical impurities; no changes in colour and odour
12 weeks		clear, homogeneous oil, without mechanical impurities; slight brightening of yellow colour, no changes in odour	clear, homogeneous oil, without mechanical impurities; slight darkening of yellow colour, no changes in odour

Source: own study.

Tab. 8. The visual and sensory assessment of care oils containing 0.1% Tocomix L50-IP stored at different temperatures for 12 weeks

Formula with 0.1% Tocomix L50-IP	4°C	RT+UV	40°C
0 days	clear, light yellow, homogeneous oil, without mechanical impurities, pleasant odour		
4 weeks	clear, light yellow, homogeneous oil, without mechanical impurities; no changes in colour and odour	clear, homogeneous oil, without mechanical impurities; slight brightening of yellow colour, no changes in odour	clear, light yellow, homogeneous oil, without mechanical impurities; no changes in colour and odour
8 weeks		clear, homogeneous oil, without mechanical impurities; slight brightening of yellow colour, no changes in odour	clear, light yellow, homogeneous oil, without mechanical impurities; no changes in colour and odour
12 weeks		clear, homogeneous oil, without mechanical impurities; slight brightening of yellow colour, no changes in odour	clear, homogeneous oil, without mechanical impurities; slight darkening of yellow colour, no changes in odour

Source: own study.

Tab. 9. The visual and sensory assessment of care oils without antioxidant (control samples) stored at different temperatures for 12 weeks

Control sample without Tocomix L50-IP	4°C	RT+UV	40°C
0 days	clear, light yellow, homogeneous oil, without mechanical impurities, pleasant odour		
4 weeks	clear, light yellow, homogeneous oil, without mechanical impurities; no changes in colour and odour	clear, homogeneous oil, without mechanical impurities; slight brightening of yellow colour, no changes in odour	clear, homogeneous oil, without mechanical impurities; slight darkening of yellow colour, no changes in odour
8 weeks		clear, homogeneous oil, without mechanical impurities; slight brightening of yellow colour, no changes in odour	clear, homogeneous oil, without mechanical impurities; the colour of the oil deepened, but it remained unchanged until the end of the test, no changes in odour
12 weeks		clear, homogeneous oil, without mechanical impurities; slight brightening of yellow colour, no changes in odour	

Source: own study.

At the beginning of the test all care oils were clear, light yellow, homogeneous oils, without mechanical impurities. They had pleasant odour. All care oils containing 0.05%, 0.075% and 0.1% Tocomix L50-IP remained clear, light yellow, homogeneous, with pleasant odour during whole test at 4°C. It was observed the slight brightening of yellow colour of care oils containing 0.05%, 0.075%, 0.1% Tocomix L50-IP and in the control sample already after 30 days during storage at room temperature with UV access. The darkening of the colour of care oils was observed at 40°C after 90 days in care oils containing 0.05%, 0.075% and 0.1% Tocomix L50-IP. In the control sample the slight darkening of yellow colour appeared already after 30 days at 40°C and the darkening of the colour has deepened over time.

The density values measured at the beginning of the test and periodically for 12 weeks are presented in Figures 1–3. The values ranged from 0.9195 to 0.924 g/cm³ in care oils containing 0.05% Tocomix L50-IP, from 0.9193 to 0.922 g/cm³ in care oils containing 0.075% Tocomix L50-IP, from 0.9193 to 0.92 g/cm³ in care oils containing 0.1% Tocomix L50-IP in comparison to control samples with density from 0.9193 to 0.9199 g/cm³. As the results of stability test it was found that there were no major changes in density of all care oils tested regardless the storage time and temperature conditions.

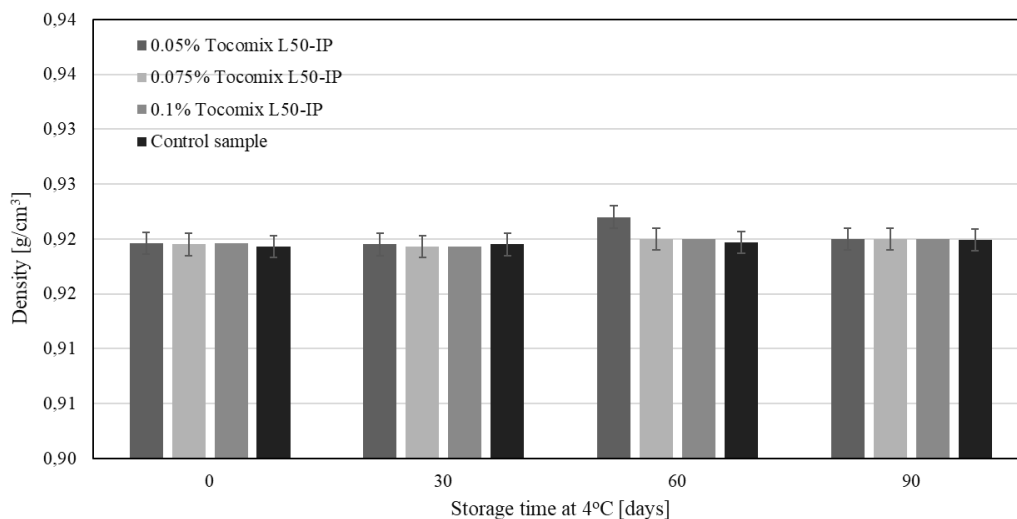


Fig. 1. Density of care oils stored at 4°C for 12 weeks (n=3)

Source: own study.

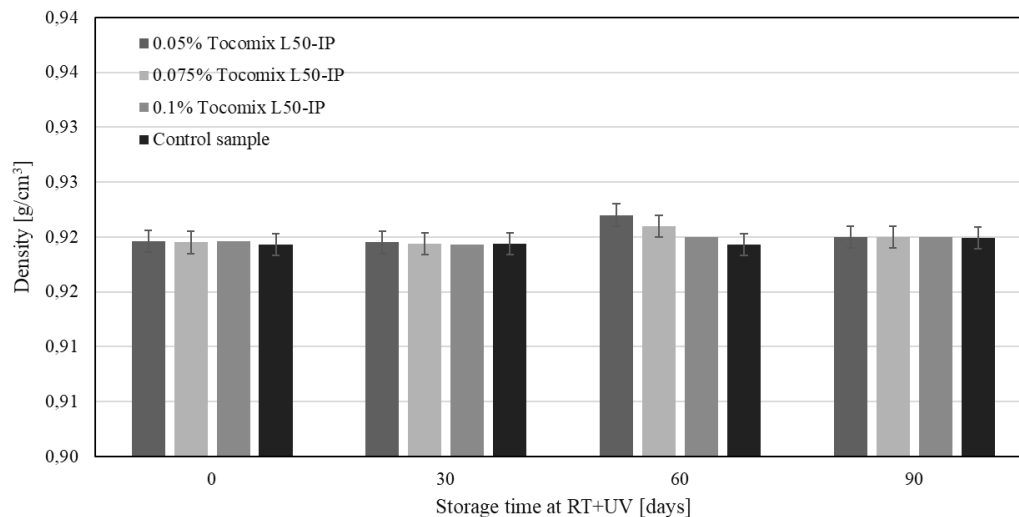


Fig. 2. Density of care oils stored at RT+UV for 12 weeks (n=3)

Source: own study.

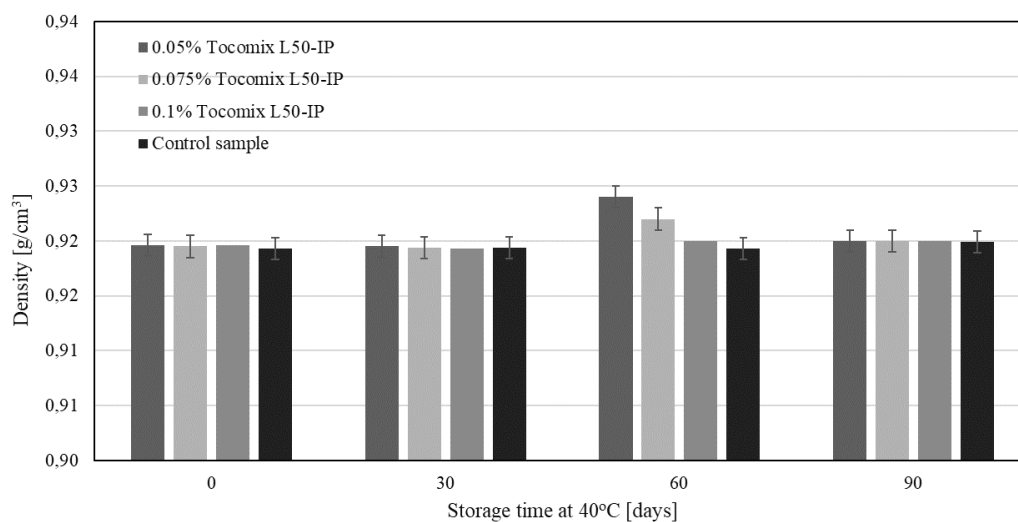


Fig. 3. Density of care oils stored at 40°C for 12 weeks (n=3)

Source: own study.

Oxidation processes, expressed as the peroxide values, of care oils, stored at different temperatures are presented in Figures 4–6. Antioxidant activities of Tocomix L50-IP to protect care oils against oxidation, expressed as the PFs values, are presented in Table 10.

Temperature is the most important factor to be considered in evaluating the oxidative stability of unsaturated oils, as the mechanism and kinetics of oxidation changes with temperature and different hydroperoxides, acting as precursors of volatile compounds, decompose at different temperatures (Frankel, 1998). In this study, factors which may influence the rate of oxidation process occurring in

oils are the high content of linoleic and oleic acids in soybean oil and the UV- and temperature-
 depended storage conditions.

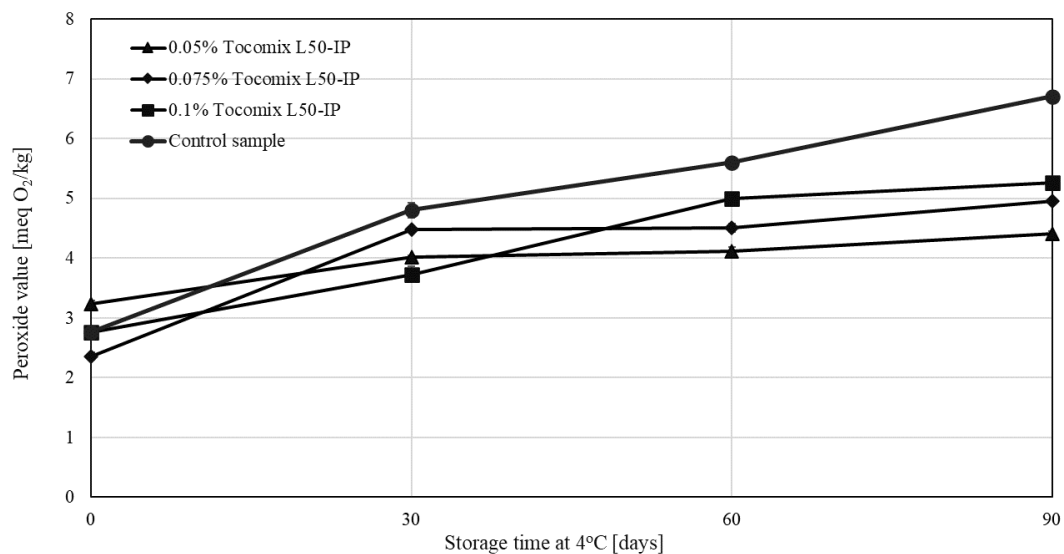


Fig. 4. Oxidation process of care oils, stored at 4°C for 12 weeks (n=3)
 Source: own study.

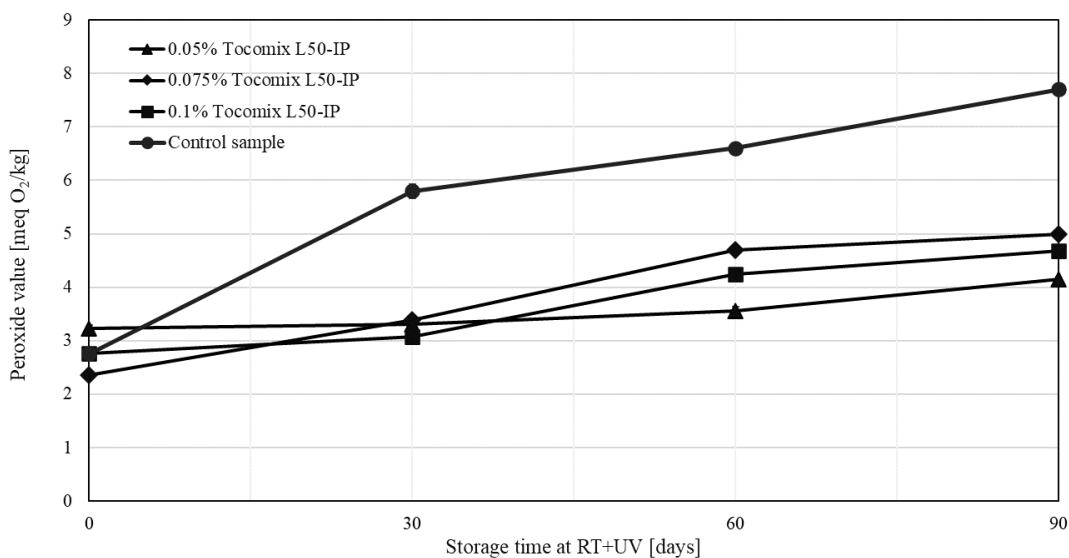


Fig. 5. Oxidation process of care oils, stored at RT+UV for 12 weeks (n=3)
 Source: own study.

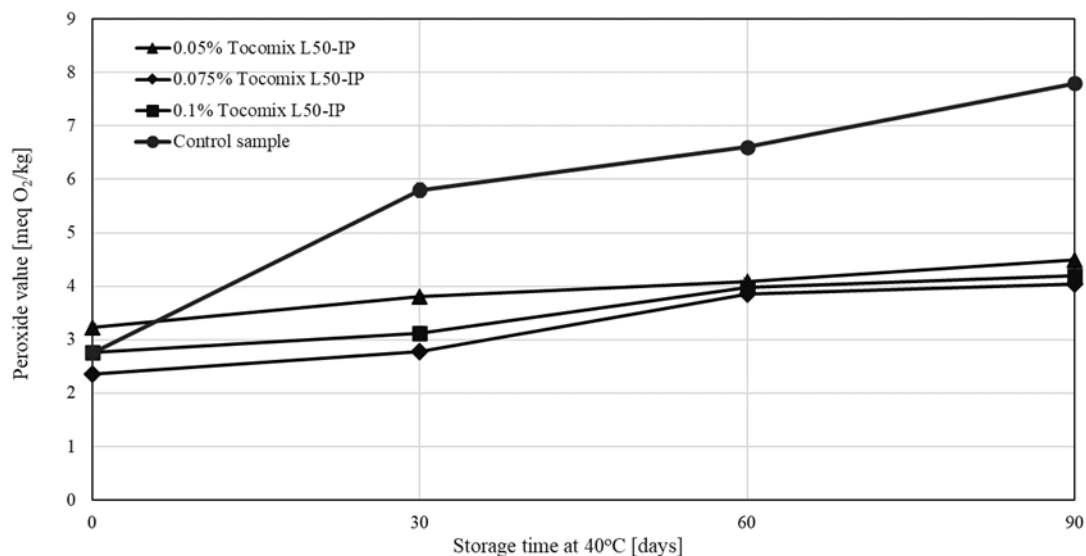


Fig. 6. Oxidation process of care oils, stored at 40°C for 12 weeks (n=3)

Source: own study.

Tab. 10. The activity of antioxidants (expressed as protective factor PF) in care oils, stored at 4°C, RT+UV and 40°C

Formulas	Protection Factor		
	4°C	RT+UV	40°C
Control sample	1.00	1.00	1.00
+ 0.05% Tocomix L50-IP	1.76	6.83	3.85
+ 0.075% Tocomix L50-IP	1.29	4.33	6.77
+ 0.1% Tocomix L50-IP	2.24	2.00	4.69

Source: own study.

It was observed that peroxide values in care oils without antioxidant (control sample) increased from 2.76 to 6.07, 7.7 and 7.8 meq O₂/kg of oils during storage at 4°C, at RT+UV and at 40°C for 12 weeks, respectively. The faster increase in peroxide values at higher temperatures and with UV access in control sample than in samples with antioxidants were observed.

During oxidation process of care oils, stored at 4°C for 12 weeks, it was observed that Tocomix L50-IP at all concentrations increased oxidative stability of care oils tested in comparison to control sample. The peroxide values in care oils stored at 4°C with 0.05%, 0.075% and 0.1% Tocomix L50-IP increased from 3.23 to 4.41 meq O₂/kg, from 2.36 to 4.95 meq O₂/kg and from 2.76 to 5.26 meq O₂/kg, respectively. None of tested concentration of antioxidant acted as pro-oxidant. The slowest growth of peroxide value was observed in care oils containing 0.05% Tocomix L50-IP, although the beginning level of peroxide value was higher than for other concentrations of Tocomix L50-IP. 0.1% Tocomix L50-IP

(PF = 2.24) was the most effective antioxidant in oil stored at 4°C. It was 1.27-fold more effective than 0.05% Tocomix L50-IP and 1.74-fold more effective than 0.075% Tocomix L50-IP. All concentrations of Tocomix L50-IP were enough effective to ensure oxidative stability of care oils at 4°C, but their activities were very low compared to other temperature conditions.

During oxidation process of care oils, stored at RT+UV for 12 weeks, it was observed that Tocomix L50-IP at all concentrations increased oxidative stability of care oils tested in comparison to control sample. The peroxide values in care oils stored at RT+UV with 0.05%, 0.075% and 0.1% Tocomix L50-IP increased from 3.23 to 4.15 meq O₂/kg, from 2.36 to 5.0 meq O₂/kg and from 2.76 to 4.68 meq O₂/kg, respectively. None of tested concentration of antioxidant acted as pro-oxidant. The slowest growth of peroxide value was observed in care oils containing 0.05% Tocomix L50-IP, although the beginning level of peroxide value was higher than for other concentrations of Tocomix L50-IP. 0.05% Tocomix L50-IP (PF = 6.83) was the most effective antioxidant in care oils stored at RT+UV. It was 1.58-fold more effective than 0.075% Tocomix L50-IP and 3.42-fold more effective than 0.1% Tocomix L50-IP.

During oxidation process of care oils, stored at 40°C for 12 weeks, it was observed that Tocomix L50-IP at all concentrations increased oxidative stability of care oils tested in comparison to control sample. The peroxide values in care oils stored at 40°C with 0.05%, 0.075% and 0.1% Tocomix L50-IP increased from 3.23 to 4.49 meq O₂/kg, from 2.36 to 4.04 meq O₂/kg and from 2.76 to 4.2 meq O₂/kg, respectively. None of tested concentration of antioxidant acted as pro-oxidant. The slowest growth of peroxide value was observed in care oils containing 0.05% Tocomix L50-IP, although the beginning level of peroxide value was higher than for other concentrations of Tocomix L50-IP. 0.075% Tocomix L50-IP (PF = 6.77) was the most effective antioxidant in care oils stored at 40°C. It was 1.44-fold more effective than 0.1% Tocomix L50-IP and 1.76-fold more effective than 0.05% Tocomix L50-IP.

At all storage temperatures (4°C, RT+UV and 40°C) Tocomix L50-IP at tested concentrations: 0.05, 0.075 and 0.1% effectively inhibited the peroxide formation in care oils. Shelf-life of care oils can be estimated at 18 months.

Conclusions

A stability test is essential to evaluate a product's shelf life. The shelf life of a product can last up to 2–3 years, but the time frame of the development of a product from brief to launch is much shorter, usually around 6 months to 1 year, so accelerated testing that only takes 3–4 months to produce a full shelf-life prediction is extremely useful (Kirkbride et al., 2021). The stability study may be seen as a prerequisite for ensuring product quality. Stability tests on cosmetic products are required for

obtaining a guidance on the formulation of the product, and the appropriate packaging material. Moreover they help to determine conditions of transportation, storage, display and manner of use, estimate and confirm the shelf life, and ensure customer safety (ISO 18811:2018). Accelerated methods for predicting cosmetic shelf-life are not easy tests due to the variety and complexity of cosmetic formulas and packaging, as well as the variety of types of changes that need to be examined, including physical, chemical, microbial, functional or aesthetic changes (CTFA, 2004).

Vegetable oil-based cosmetic development requires the selection of proper antioxidant at effective concentration. The results of the present study have shown that Tocomix L50-IP at 0.05, 0.075 and 0.1% could prolong the oxidative and physicochemical stability of care oils based on soybean oil. Especially 0.05 and 0.075% Tocomix L50-IP can be recommended for care oils based on this kind of oil, as they have revealed the highest antioxidant activity and may act as effective antioxidant in long-term storage tests as well as the active, care ingredient.

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THE ROLE OF MANUKA HONEY IN SHAPING THE QUALITY OF BODY LOTIONS FOR DRY SKIN

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DOI: 10.56091/CTQS.Qual-14

Abstract

The study analyzed the effect of manuka honey addition on selected quality parameters of body lotions intended for dry skin. For this purpose, 4 prototypes of body lotions were developed, differing in the mass ratio of beeswax to manuka honey (4:0; 3.8:0.2; 3.6:0.4; 3.4:0.6). The following tests were performed for all body lotions: stability, yield point, texture analysis, measurement of the degree of hydration and transepidermal water loss from the epidermis and a consumer evaluation of sensory attractiveness. The influence of the concentration of manuka honey on the yield point of the body lotions was found. It was established on the basis of the yield point tests that the body lotion with the beeswax to manuka honey in the mass ratio of 3.8:0.2 had the highest value, and the reference preparation had the lowest value. These results corresponded with the results obtained in the study of hardness and adhesion strength. After application of cosmetics containing manuka honey, an increase in skin hydration and a decrease in transepidermal water loss were observed. The highest number of points scored in the consumer assessment of sensory attractiveness was achieved by a body lotion with a mass ratio of beeswax to manuka honey of 3.8:0.2.

Keywords: body lotions, cosmetics, dry skin, manuka honey, quality

Introduction

In recent years, manuka honey has gained enormous popularity. It is produced by bees from the nectar of the flowers of the manuka bush. The plant is exotic and native to the east coast of the islands of New Zealand and southeastern Australia (Grzebisz & Grzebisz, 2016). It has white, red or pink flowers and belongs to ornamental plants. It is found in parks and gardens. The name "manuka" is derived from the language of the Maori people living in New Zealand, who used the medicinal properties of the honey. It is also known by the term "New Zealand tea tree," as the leaves of this plant are often brewed into tea. Since manuka honey has gained popularity around the world, exports from New Zealand have been growing rapidly and fueling the entire New Zealand honey industry. The bees that pollinate the manuka bush were brought to New Zealand in 1839 by Mary Bumby. The two native bee species were not suitable for honey production. In contrast, the new colonies multiplied rapidly, and in the 1860s the Maori – the country's first beekeepers – began

selling honey. In the late 1870s, after the introduction of the Langstroth hive, a model of hive with movable frames, commercial honey production began. Manuka honey is well known in the food market, but it has also attracted interest in the cosmetic and medical industries in recent years (Cianciosi et al., 2018; Kazmierczak & Wcisło-Dziadecka, 2017; Kędzia & Holderna, 2015; Pavlačková et al., 2020).

Manuka honey contains many valuable components that have made it popular. First of all, it consists of water and carbohydrates (Cianciosi et al., 2018). The dominant component is maltose (19.6%), followed by isomaltose (15.5%) and sucrose (15.4%). The level of glucose is low at 11.6%, and fructose at 4.3% (Grzebisz & Grzebisz, 2016). In the composition of manuka honey, one can also distinguish proteins mainly in the form of free amino acids and enzymes, minerals such as potassium, copper, iron, zinc, calcium and B vitamins (Cianciosi et al., 2018). Its properties are influenced by flavonoids. A number of studies have determined that pinobanksin, pinocembrin and chrysin are present in manuka honey, while luteolin, quercetin, 8-methoxykaempferol, isorhamnetin, kaempferol and galangin have been identified in low concentrations. Other constituents include phenolic acids and volatile norisoprenoids (Alvarez-Suarez et al., 2014).

Various 1,2-dicarbonyl compounds such as glyoxal (GO), 3-deoxyglucosulose (3-DG) and methylglyoxal (MGO) are also present in manuka honey (Alvarez-Suarez et al., 2014). In manuka honey, unlike most honeys, methylglyoxal is found in high concentrations (Adams et al., 2008; Frydman et al., 2020; Kędzia & Holderna-Kędzia, 2015). MGO influences antioxidant, bacteriostatic, anti-inflammatory and antimicrobial properties (Kazmierczak & Wcisło-Dziadecka, 2017; Kędzia & Holderna, 2015). Studies (Cianciosi et al., 2018; Tomblin et al., 2014) have shown that manuka honey has well-established anti-inflammatory effects in local wound healing. Manuka honey is also a potent anti-aging and lubricating agent (Pavlačková et al., 2020). Hence is born the interest in this bee raw material and its use in cosmetics for dry skin.

It is noteworthy that the literature does not record scientific articles treating the use of manuka honey in personal care cosmetics and the evaluation of the physicochemical and functional properties of cosmetics with its participation. Therefore, this is the apex of the present topic.

This paper presents the developed formulations of 4 body lotions in the form of emulsions differing in the mass ratio of manuka honey to beeswax. The following tests were performed for the body lotion prototypes: pH measurement, stability, dynamic viscosity, texture analysis, yield point, and tests to determine the effect against the skin, i.e. skin hydration level and measurement of transepidermal water loss from the epidermis. The paper also presents a consumer evaluation of sensory appeal for body lotion prototypes involving manuka honey.

Experimental part

Formulations

On the basis of literature data (Barel, 2009; Epstein, 2009), analysis of the cosmetics market and our own experience, a series of prototypes of body lotions in the form of emulsions with manuka honey were developed (Table 1). In order to use the same total concentration of the hydrophobic phase, the body lotions differed in the mass ratio of beeswax to manuka honey (4:0; 3.8:0.2; 3.6:0.4; 3.4:0.6).

Tab. 1. Formulations of body lotions differing in the mass ratio of beeswax to manuka honey

Phase	INCI NAME	Trade name/Producer	Concentration [%w/w]			
			BL_1	BL_2	BL_3	BL_4
A	Cetearyl Olivat / Sorbitan Olivat	Olivem 1000 / Hallstar	2.5			
	Glyceryl Stearate	Cithrol GMS / CRODA Polska	3.0			
	Cetearyl Alcohol	Lanette O / BASF Poland	2.0			
	Triisostearin	Crodamol GTIS / CRODA Polska	3.0			
	Vitis Venifere Seed Oil	Grape seed oil /ECOSPA	2.0			
	Cera Alba	Beeswax /Standard Sp. z o.o.	4.0	3.8	3.6	3.4
	Leptospermum scoparium mel (Manuka Honey)	Manuka honey / Manuka Health New Zealand Ltd.	–	0.2	0.4	0.6
B	Aqua	Watter	up to 100			
	Glycerin	Vegetable glycerin / Cremer Special Equipment BV ingredient concentration: 99.5%.	2.5			
C	Sodium Benzoate and Potassium Sorbate	KEM BS / Pol Nil S.A. Active ingredient concentration: 50%.	1.0			
	Citric Acid	Manufacturer HSH Chemie Poland	up to pH 5.5–6.0			
	Tocopherol Acetate	Vitamin E / ECOSPA	0.1			

The body lotions were made as follows: first, all the ingredients of fat phase A were combined together, mixing each time. Then phase A was heated in a water bath to 75°C. Subsequently, the components of phase B were mixed and brought to 75°C. The two phases were combined while stirring thoroughly. The lotions were then cooled to 35°C and vitamin E and preservative were added. Each cosmetic was homogenized using a Silent Crusher-M-homogenizer at 26°C at 10 rpm for 3 min. Body lotion BL_1 required pH adjustment, and citric acid was used for this purpose.

Body lotion samples were stored at a temperature of $21\pm 0.5^{\circ}\text{C}$ and a relative humidity of 40–60%. The tested preparations were placed in plastic, tightly closed containers.

Materials and methods

Stability

The Turbiscan Lab measuring instrument, manufactured by Formulacion Smart scientific analysis company, was used to assess the stability of the body lotions, allowing the symptoms of emulsion instability to be picked out at a very early stage. A light source and two detectors are present in the device, which collect information on the intensity of light reflected and passing through the sample. The body lotion samples were stored at 40°C for a period of 13 days.

Yield point

The yield point of the tested body lotions was determined using a rheometer (model HA DV III Ultra from Brookfield Engineering Laboratories, USA) equipped with a set of paddle spindles. The yield point is the lowest value of shear stress at which a substance begins to flow. Body lotions characterized by a lower flow limit tend to have a "lighter" consistency and spread more easily on the skin. Measurements were conducted at spindle speed: 1 rpm. Measurement results were recorded and analyzed using EZ-Yield software.

Texture analysis

Consistency evaluation was examined using a texture analyzer – model CT3 4500 from Brookfield Engineering Laboratories, USA. The test was conducted with a measuring probe made of nylon, with a load of 0.1 g and a measuring speed of 0.1 mm/s. The results were recorded by Texture Pro CT software. The texture profile analysis consisted of assessing the following properties: hardness (the mass required to push the probe to a depth of 10 mm, which is the maximum force recorded during 1 test cycle) and adhesion strength (the mass that must be applied to the probe to pull it out – a measure of the adhesion of the formulation to the probe (Klimaszewska et al., 2016)).

Skin hydration level

Skin hydration level was tested using a corneometer – Corneometer CM 825, which determines the capacitive resistance of the stratum corneum. Skin hydration was measured on clean, degreased skin, which served as a reference. Then 1 g of the test body lotion was applied to a 20 x 20 mm area of forearm skin. Measurements were taken 2h after the lotion was applied to the skin at 22°C . Measurements were taken for 10 women aged 35–40 years.

Transepidermal water loss

Transepidermal water loss was examined using a tewameter – Tewameter TM 300. Measurements were taken on clean, degreased skin (baseline) and 2 h after applying body lotions. Measurements were taken for 10 women between the ages of 35 and 40.

Consumer assessment of sensory attractiveness

Consumer sensory evaluation was carried out for four manufactured body lotions. Body lotions were evaluated by ten people. The evaluators were asked to describe the sensations they felt when spreading the body lotions on the skin and the sensations they experienced when touching the preparations. Each was evaluated for parameters such as fragrance, texture, consistency, cushion effect, spreading, smoothness, stickiness, greasiness and absorption. The parameters were evaluated on a scale of 1 to 5, with the number 5 indicating a high level of quality, 4 – good, 3 – sufficient, 2 – acceptable and 1 – insufficient. A detailed description of sensory parameters is presented in the works of Kulawik Pióro et al. (2020) and Szakiel & Turek (2019). The assessment was performed at room temperature, and the probands had healthy skin, without irritations or dermatological changes.

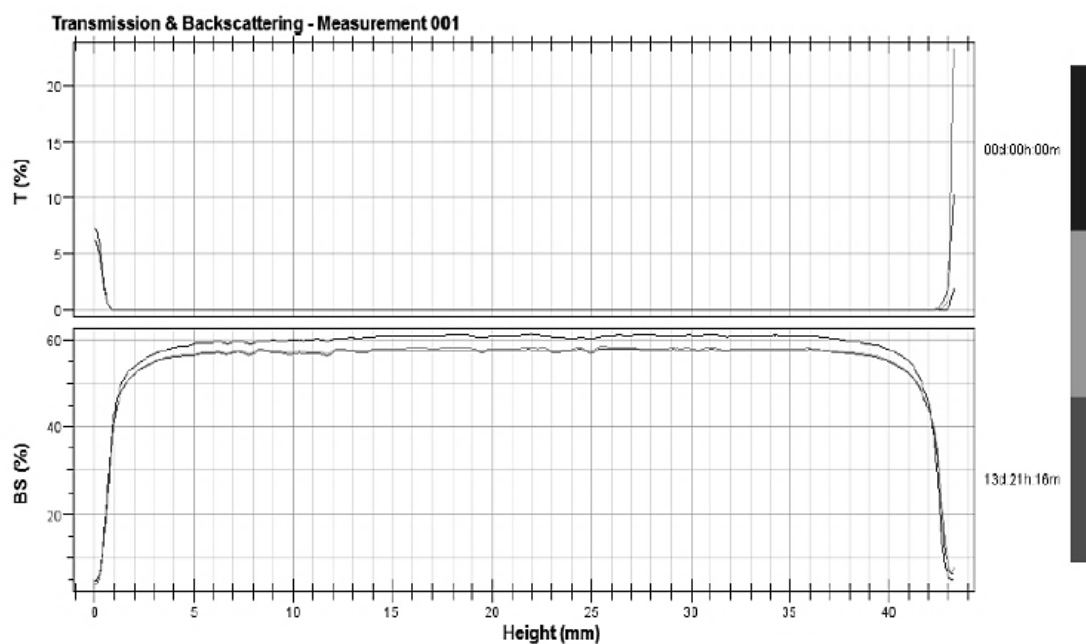
Statistical Analysis

The bars in the graphs represent the arithmetic means of the values from three independent measurements. The Student's t – test was used to analyze statistical differences between mean values ($\alpha = 0.05$). Confidence intervals representing the measurement error for a confidence level of 0.95 were determined. The error values are shown in the graphs.

Results and Discussion

Stability

In recent years, there has been an increased interest in a reliable and rapid method for testing the stability of dispersion systems based on the measurement of the intensity of transmitted and backscattered light, performed with the Turbiscan apparatus. This measurement makes it possible to identify phenomena that cannot be observed visually, predestining the detection of symptoms of emulsion instability, such as particle migration, particle size change, flocculation, coalescence, creaminess, at a very early stage (Wasilewski et al., 2021; Kowalska et al., 2022; Iyer et al., 2015). Figure 1 shows an example of the dependence of transmittance (T) and backscatteringlight (BS) on sample height for a body lotion with a mass ratio of beeswax to manuka honey: 3:4 to 0.6.



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Fig. 1. Effect of sample height on the transmittance (T) and backscatteringlight (BS) for body lotion with a beeswax to manuka honey mass ratio of 3:4 to 0.6; temperature of storage 40°C during 15 days (Turbiscan); black line – evaluation after application (time “0”), light-grey line – stability after 7 days, dark-grey line – stability after 13 days

Source: own study.

Based on the study, there was no effect of the mass ratio of beeswax to manuka honey in the body lotions. For all tested cosmetic prototypes, no significant signs of instability were observed at 13 days.

In general, it is assumed that the curves of the percentage of light transmitted and backscattered by the sample reflect the state of the emulsion as a function of its height. This indicates that any changes from the standard measurement inform about the onset of physical phenomena usually deepening over time. Analyzing the individual graphs, one can generally observe an overlap of the curves, which may indicate that the particle size varies slightly over the storage period.

Among other things, stability assessment using turbiscan was described in their work (Iyer et al., 2015; Kowalska et al. 2022; Wasilewski et al., 2021). Among other things, they pointed out that the average particle size is a very important parameter in determining the stability of an emulsion system. A larger increase in the particle size of the emulsion system over time results in an apparent lack of overlap in the curves measured by the Turbiscan test.

Yield point

The yield point of body lotions (Figure 2.) for the manufacturer, it is important in selecting the optimal packaging of the cosmetic and the method of dosage, which in turn affects the efficient and easy use of the product by the consumer. Lower values of this parameter indicate a lighter consistency and generally better distribution of the product on the skin.

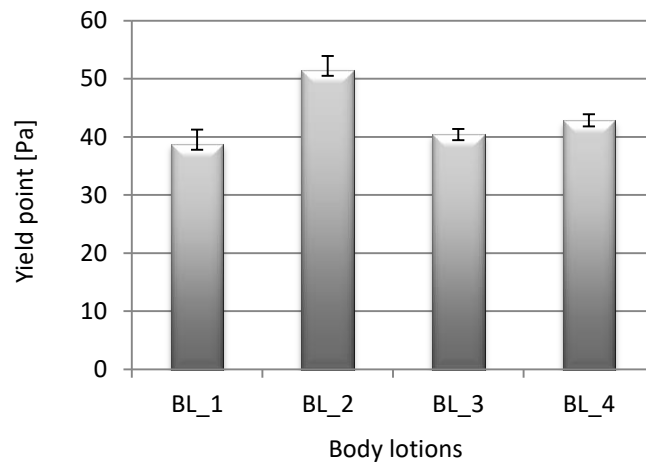


Fig. 2. Yield point of body lotions differing in the mass ratio of beeswax to manuka honey

Source: own study.

Analyzing the results of the yield point, it can be concluded that there is no close relationship between the composition of the mixture of beeswax and manuka honey in the body lotions and the values of the yield point. The lowest yield point (39 Pa) was recorded for a body lotion with a beeswax to manuka honey mass ratio of 4:0 (BL_1). Similar values were observed in BL_3 and BL_4 lotions with a beeswax to manuka honey mass ratio of 3:04 and 3.4:0.6, respectively. Only for BL_2 lotion the value of the yield point was higher by about 30–33% compared to the other tested body lotions. For this lotion, the ratio of beeswax to manuka honey was 3:8 to 0.2.

Yield point values presented in the literature for body lotions vary. For example Klimaszewska et al., (2019) examined the yield point of body lotions with chia oil and commercial lotions in their work. The values ranged from 18–136 Pa. In the work of Ogorzalek et al. (2022), the yield point of cosmetic emulsions oscillated between 52–100 Pa.

The obtained results of the study of the yield point of body lotions correspond with the results of studies presented by the Authors (Briceno, 2000; Kulawik-Pióro et al., 2019; Lukic et al., 2012). It seems that the flowability of samples depends primarily on the varying type of emollient, i.e. the rheological properties of the continuous (oil) phase.

Texture analysis

Fig. 3 summarizes the results of texture analysis of body lotions.

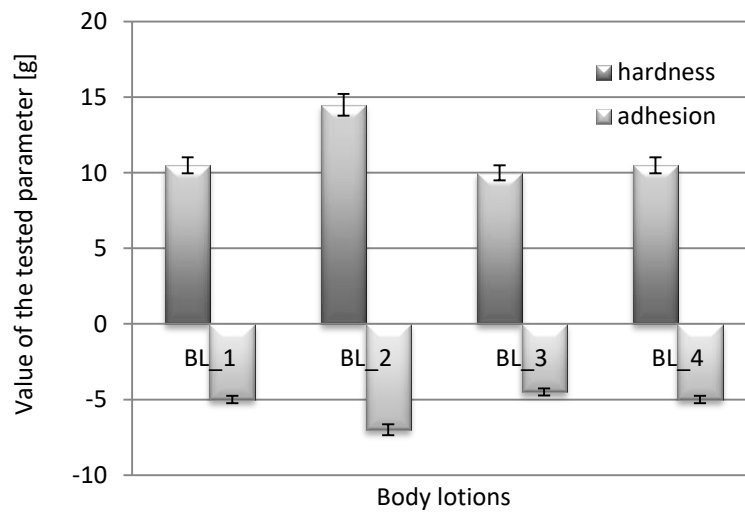


Fig. 3. Hardness and adhesive force of body lotions differing in the mass ratio of beeswax to manuka honey

Source: own study.

It was found that the highest hardness value (14.5 g) was characterized by lotion BL_2, in which the mass ratio of beeswax to manuka honey is 3.8:0.2. The lowest hardness value was recorded for lotion BL_3 (10 g). This cosmetic had beeswax to manuka honey mass ratio of 3.6:0.4 (Figure 3).

For adhesion strength tests, the lowest value was observed for BL_2 lotion (-7 g). In contrast, the highest value was obtained for BL_3 (-4.5 g). Comparable results of hardness (10.5 g) and adhesion force (-5 g) measurements were observed for BL_1 and BL_4 lotions. The results obtained correspond with those obtained for the yield point.

Hardness and adhesion force values for cosmetic emulsions presented in the literature range from; 4 to 107 g for hardness and -77 to -1.5 g, for adhesion force (Gilbert et al., 2013; Kowalska et al., 2022).

Considering the application properties, the values for both hardness and adhesion strength are favorable, especially in the context of applying body lotions from packaging. In addition, the adhesion force values obtained from the experiment indicate that spreading the product on the skin will be easy for the user.

Skin hydration level

Proper skin hydration plays an important role in many processes, such as accelerating wound healing and skin regeneration and delaying skin aging. Lack of adequate skin hydration can also greatly impede wound healing and cause poor skin condition. To this end, a study was conducted on skin hydration after application of body lotions (Figure 4).

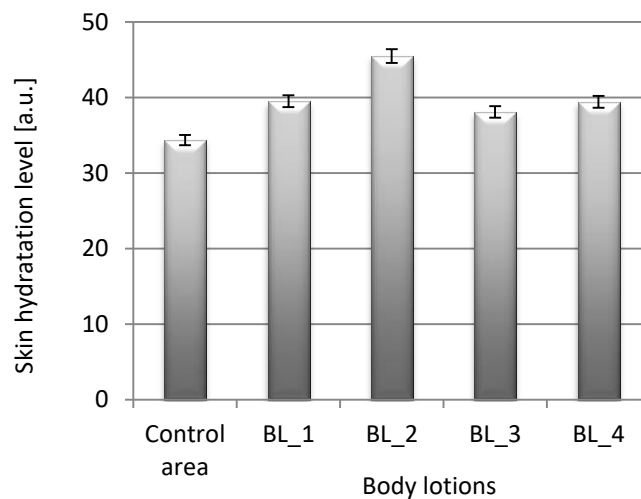


Fig. 4. Skin hydration level after 2h from application of body lotions differing in the mass ratio of beeswax to manuka honey

Source: own study.

Skin hydration for the control area was 34 a.u. Application of body lotions with different ratios of beeswax to manuka honey contributed to an increase in skin hydration to about 40–46 a.u. With the highest increase in skin hydration recorded 2 h after application of the BL_2 lotion with a beeswax to manuka honey mass ratio of 3:8 to 0.2. It can therefore be concluded that the addition of even a small concentration of manuka honey improves this parameter.

The results obtained for skin hydration are within the range observed in the literature. For example, the paper (Zięba & Wilkiewicz, 2015) studied skin hydration after applying body lotions with coffee. After 1 h after application of the cosmetics, skin hydration values ranged from 46–56 a.u. Similarly, skin hydration values were obtained by the authors in the work (Wasilewski et al., 2021) studying skin hydration 2 h after the application of body lotions with particulates from plant materials and (Lukic et al., 2012) analyzing skin hydration after the application of cosmetic emulsions with emollients, including beeswax.

In the article (Jung et al., 2015), the authors performed an in vivo study conducted on patients suffering from mild to moderate psoriasis and proved that regular use of a cosmetic, multifunctional mois-

turizing and repairing lotion involving emollients for skin care alleviated symptoms such as dryness, roughness and visible lesions after just 4 weeks of treatment.

Transepidermal water loss

TEWL testing (Fig. 5) is extremely important in terms of the condition of the skin after applying body lotions. A large amount of water vapor released from the skin can indicate a poor state of the epidermal barrier. Altered skin barrier function is characterized by elevated TEWL, which is observed in many skin diseases such as atopic dermatitis and psoriasis (Olejniczak-Staruch et al., 2022).

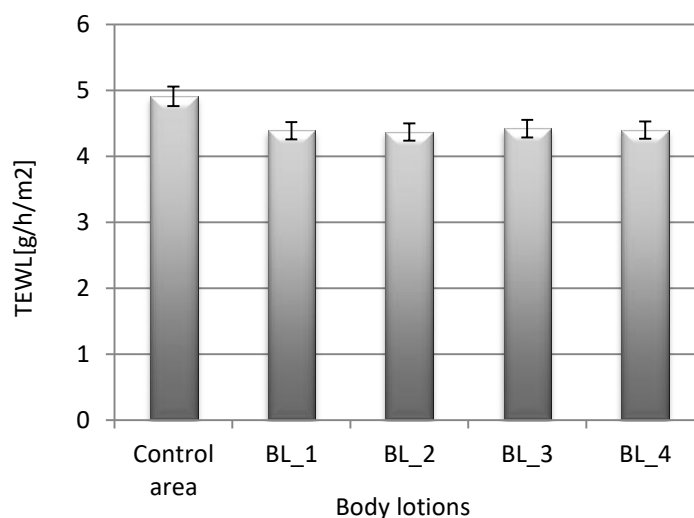


Fig. 5. TEWL after 2 h from the application of body lotions differing in the mass ratio of beeswax to manuka honey

Source: own study.

Two hours after the lotions were applied to the skin, a decrease in TEWL, i.e. a decrease in water loss from the skin relative to the control area, was noted for all the cosmetics tested. However, there was no significant effect of the composition of the beeswax mixture and manuka honey on the TEWL value. The results ranged from 4.37–4.42 g/h/m².

In conclusion, in addition to the improvement in skin hydration and slight water loss after the application of body lotions with manuka honey, there are scientific data on the improvement in wound healing after the application of this ingredient (Lacopetti et al., 2020).

Consumer assessment of sensory attractiveness

An important tool in the evaluation of body lotions, as well as in their design and quality assessment, is the consumer evaluation of sensory appeal (Fig. 6). The results obtained from it can be used to

verify consumer preferences and select cosmetics with the most desirable properties (Kulawik-Pióro et al., 2020; Szakiel & Turek, 2019; Zięba et al., 2016).

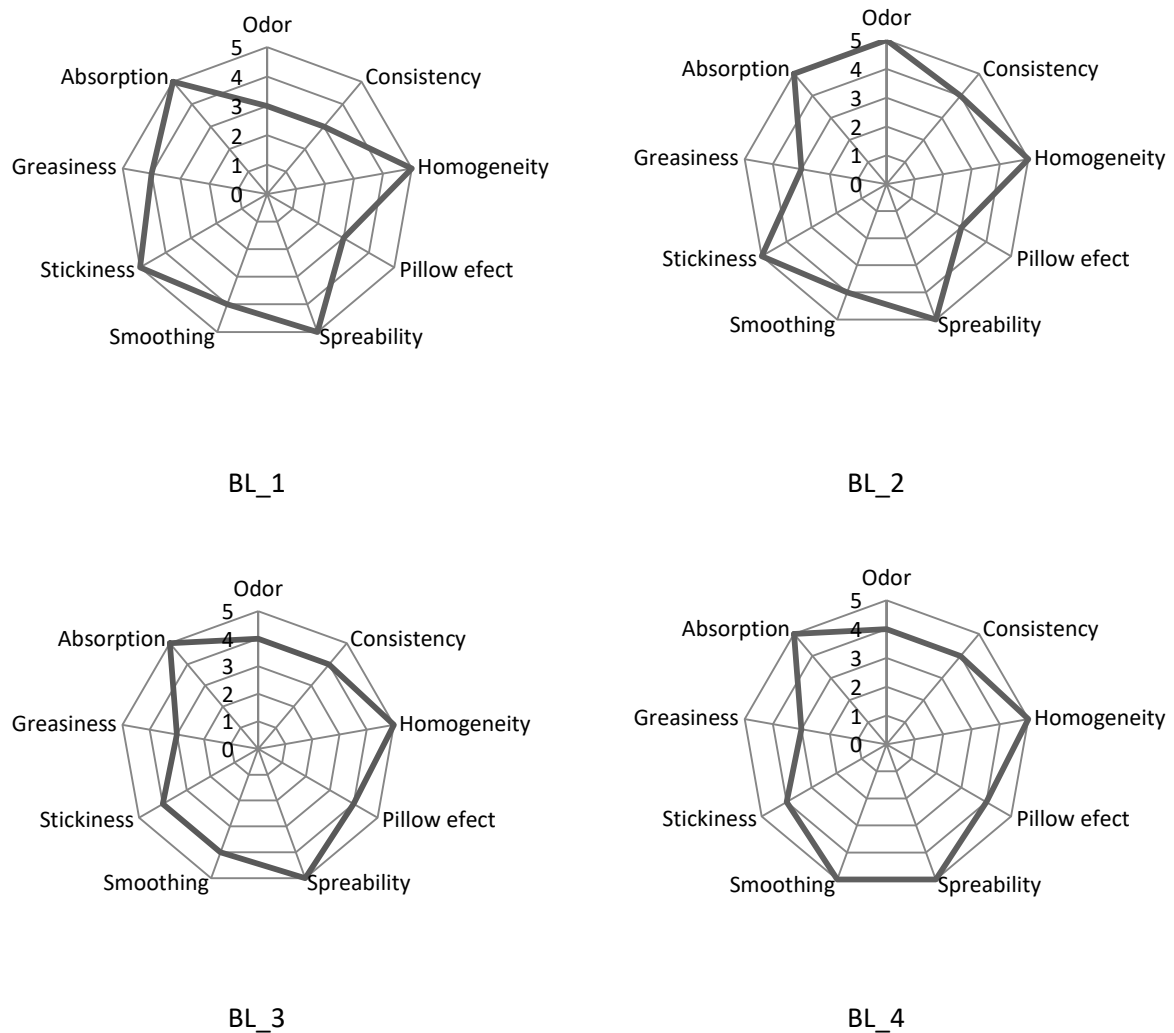


Fig. 6. Consumer assessment of sensory attractiveness for body lotions

Source: own study.

The highest rated parameters, out of a maximum number of points, were homogeneity, spreadability and absorption of all the body lotions tested. The lowest rated parameter was the pillow effect (3–4). Adding manuka honey to the formulations, according to the probands, improved the body lotions' fragrance and texture.

Summing up the scores, it was found that the prototypes were rated highly. The BL_2 lotion, in which the ratio of beeswax to manuka honey is 3.8:0.2, was rated most favorably, earning a total of 45 points out of a possible 50. BL_3 and BL_4 cosmetics received equally 43 points each. The lowest number of points was scored by BL_1 – without manuka honey (41 points).

Conclusions

The aim of the study was to develop formulations of body lotions with manuka honey and determine its effect on selected physicochemical and performance properties of such products.

After analyzing the results of the study, the following conclusions can be drawn:

- Manufactured prototypes of care lotions for dry skin show the stability necessary for emulsion products. The body lotions showed no signs of instability.
- It was found, there was no strict relationship between the composition of the beeswax mixture and manuka honey in the body lotions and the values of the yield point. The results oscillated between 39 and 52 Pa. It was determined from the study that the highest value was characterized by the BL_2 lotion, in which the ratio of beeswax to manuka honey was 3.8:0.2 and the lowest, indicating "ease" of application was the BL_1 formulation, which does not contain manuka honey in its composition. The yield point of BL_3 and BL_4 lotions was similar to the value obtained for BL_1.
- The highest value of hardness (14.5 g) and the lowest value of adhesion force (-7) were recorded for BL_2 lotion, in which the mass ratio of beeswax to manuka honey is 3.8:0.2. The other body lotions were characterized by lower values of hardness and higher values of adhesion force compared to BL_2 lotion. The results corresponded with those obtained for the yield point.
- After application of lotions containing manuka honey in the formulation, an increase in skin hydration and a decrease in transepidermal water loss were observed. The addition of manuka honey already at a low concentration (0.2%) has a beneficial effect on the skin. Therefore, the use of higher concentrations of this ingredient is not necessary.
- The body lotion prototypes performed favorably in the conducted consumer evaluation of sensory appeal. The highest scoring product is BL_2, with a ratio of beeswax to manuka honey of 3.8:0.2. Thus, it can be concluded that the higher yield point and higher hardness values compared to the other body lotions did not adversely affect the evaluation of sensory parameters.

Acknowledgments

The work performed under the project no. 3501/188/P titled: "Application of innovative raw materials of natural and synthetic origin in care and support of treatment of skin diseases in various clinical conditions". Project financed by the Ministry of Education and Science.

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ANALYSIS OF THE QUALITY OF BATH COSMETICS WITH THE ADDITION OF GOAT'S MILK

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DOI: 10.56091/CTQS.Qual-15

Abstract

Substances from which bath products are made are very often allergenic, therefore sensitive skin, which is prone to allergic changes, is not always able to avoid the negative effects of using such products. In this case, self-made products become a good alternative, which will contain only such ingredients that there will be no doubt that they are dangerous for the skin.

Goat's milk is rich in minerals and vitamins. It is one of the products with the highest amount of calcium in its composition. In addition to calcium, goat's milk also contains vitamins such as: C, D, E, B12, sodium, potassium, phosphorus, iron, zinc, selenium, PP, folic acid, copper and manganese. In the cosmetics industry, goat's milk is used in body, face and hair care products. Preparations based on this natural ingredient have a strong moisturizing and softening effect.

The aim of this study was to analyze the quality of selected preparations for baths with the addition of goat's milk, evaluated on the basis of utility tests and consumer opinions. Commercial preparations and preparations made with the use of several ingredients at home were compared. The study was also intended to assess the properties of DIY (Do It Yourself) products.

An organoleptic evaluation of such products was carried out, as well as an assessment of their performance properties, and consumers were asked for their opinion on the use and operation of such preparations.

Keywords: goat milk, bath cosmetics, useful properties, DIY cosmetics, consumer preferences

Introduction

Modern cosmetology appreciates various raw materials of animal, plant and mineral origin, which are a rich source of many biologically active substances. One of the reasons for using them in the production of cosmetics is the constantly growing demand for naturalness. Consumers increasingly prefer natural ingredients in cosmetics, which is connected with the ever-growing number of preparations containing components such as honey and bee products, aloe or goat's milk. Such additives are particularly desirable in preparations for daily hygiene.

The most important thing when choosing bath products is their appropriate selection for the skin and composition. The analysis of the composition of cosmetics should be a priority when using cosmetics,

because it is from the list of substances available on the label that the consumer is able to read what effect he can expect from a given product. It is also worth paying attention to whether the cosmetic in its composition does not contain strong preservatives that can irritate the skin.

The aim of this study was to analyze the quality of selected preparations for baths with the addition of goat's milk, evaluated on the basis of utility tests and consumer opinions. Commercial preparations and preparations made with the use of several ingredients at home were compared. The study was also intended to assess the properties of DIY (Do It Yourself) products.

Goat milk properties and application

Goat's milk is a natural product of animal origin, considered an alternative to cow's milk due to the lower amount of lactose in its composition (local farmer.pl). In addition to its beneficial health properties, it has also been known and appreciated as a cosmetic product for centuries. Already in antiquity, a well-known figure, i.e. Cleopatra, used it with the benefits of this product by bathing in it, as well as in honey and almonds, in order to soften their skin (Szewczyk & Młynarczyk, 2015).

The richness of minerals and vitamins makes this product have a very positive effect both on health and directly in contact with the skin. Goat's milk is known with a rich source of vitamins and trace elements. It is one of the products with the highest amount of calcium in its composition (Table 1). In addition to calcium, goat's milk also contains vitamins such as: C, D, E, B12, sodium, potassium, phosphorus, iron, zinc, selenium, PP, folic acid, copper and manganese (Pastuszka et al., 2014).

Goat milk health benefits:

- helps lower blood cholesterol,
- has a good effect on the work of the heart,
- reduces the risk of anemia,
- protects against the development of osteoporosis,
- allows the absorption of vitamins such as iron and copper,
- has a positive effect on the condition of the bones,
- protects against cancer,
- minimizes the likelihood of neurodegenerative diseases (zdrowie.tvn.pl).

Care properties of goat's milk:

- hydration,
- softness,
- clarification,

- smoothing,
- nutrition,
- flexibility,
- elasticity,
- protection against harmful radicals (Koniewicz & Bela, 2018).

Tab. 1. Comparison of the amount of minerals in 100 grams of goat, sheep and cow milk

Mineral (mg)	Goat's milk	Sheep's milk	Cow's milk
Calcium	134	193	122
Phosphorus	121	158	119
Magnesium	16	18	12
Potassium	181	136	152
Sodium	41	44	58
Chlorine	150	160	100
Sulfur	28	29	32
Iron	0.07	0.08	0.08
Copper	0.05	0.04	0.06
Manganese	0.032	0.007	0.02
Zinc	0.56	0.57	0.53
Iodine	0.022	0.02	0.021
Selenium (µg)	1.33	1.0	0.96

Source: www.spozywczytechnologie.pl.

In the food industry, goat's milk is considered an easily digestible product that is easily absorbed by the human body. The unique nutritional and functional properties of goat milk proteins are noteworthy (Van Leeuwen et al., 2020), including higher digestibility, buffering capacity and alkalinity compared to cow's milk (Saikia et al., 2022). Goat milk proteins have a different ratio of caseins and whey proteins than cow milk, resulting in different functional properties (Qin et al., 2021). Therefore, goat milk proteins are promising for use in various food products, especially for people sensitive to cow's milk (Csapóné Riskó & Csapó, 2019).

Goat's milk is most widely used in the category of dairy products. On store shelves you can see products such as cheese spreads, milk, yoghurts, butters, cottage cheese, salad cheeses or smoked cheeses. In addition, this product can be found in chips, various types of snacks or sweets (zywanie.medonet.pl). Goat milk can be used to create cosmetics with antioxidant and anti-

inflammatory properties and as baby formulas, which are very useful due to their good digestibility and the content of all substances necessary for kids (Voloshyna et al., 2021).

In the cosmetics industry, goat's milk is used in body, face and hair care products. Preparations based on this natural ingredient have a strong moisturizing and softening effect.

In the case of hair care, goat's milk is perfect for damaged and dry hair and it is in the case of this type of hair that you can notice the greatest effects. Hair masks with the addition of this ingredient will smooth and give your hair shine. It is also worth noting that products such as shampoos, conditioners and masks have a positive effect on combing hair and restore their healthy appearance (Śliwa et al., 2011).

Face products with the addition of goat's milk are most often: cream, mask, washing gel, cleansing scrub, concentrate, peeling or make-up remover and toner. The largest range of care products with the addition of goat's milk is offered for body skin care. This category includes products such as: shower gels, body milks, balms, butters, hand masks and fingernails, both bar and float soaps, as well as bath balls, salts and powders.

Goat milk is also used in professional beauty salons. Treatments with the addition of goat's milk are considered a luxurious and royal feast for the body and spirit. It is worth noting that the treatment with the addition of this nutritional ingredient is often recommended for pregnant women, because it has, among other things, softening properties, which can be very helpful in the case of stretch marks on the skin (art of cosmetology.pl).

Cosmetics with the addition of goat's milk

The largest selection of cosmetics based on goat's milk are products for bathing. This category includes preparations such as: bath bombs, lotions, shower gels with a rich, milky consistency, bath powders or salts with the addition of goat's milk. In addition to bath products, goat milk is also found in products such as body lotions, butters and scrubs.

In the case of facial care, goat's milk has found its application, among others in face creams and masks. These products are characterized by a softening, nourishing and deeply moisturizing effect. Preferred cosmetics are especially for dry skin, as well as for delicate and sensitive skin.

Goat milk is also used in hair care cosmetics such as shampoos and masks. These preparations will have a strengthening and smoothing effect on the hair. Cosmetics with the addition of goat's milk are very good for the care of dry hair, which is characterized by a lack of shine and roughness (Boniakowska et al., 2016).

One of the main advantages of cosmetics with the addition of goat's milk is their versatility. Cosmetics that contain goat's milk are intended for face, body and hair care. Most of these types of products that have appropriate markings can be used by all family members, including children and babies. Another advantage of products with the addition of goat's milk is a deeply moisturizing and softening effect. Goat's milk due to the fact that it contains, among other things, vitamins A and E, helps to maintain the youthful appearance of the skin and maintain proper hydration of the epidermis. In addition to moisturizing properties, cosmetics with the addition of goat's milk also have smoothing, nourishing and increasing skin elasticity properties. In addition, it protects the milk from the harmful effects of free radicals, and also eliminates the feeling of tension and dryness of the skin. Cosmetics with the addition of this ingredient are suitable for daily care, both in the morning and in the evening. Another important advantage of products with the addition of goat's milk is the fact that these cosmetics can be used by people with different complexions, because they tend to minimize the risk of irritation. Thanks to this, they can be used by people with normal, mixed, oily, dry, sensitive or problematic skin types. Goat's milk has properties that restore the skin's natural pH, which means that it also regulates the secretion of sebum.

Study of bath cosmetics with goat's milk

The aim of the study was to evaluate selected features of products with the addition of goat's milk. Organoleptic, physicochemical and functional parameters of bath preparations were analyzed. Commercial products were compared with home-made (DIY) formulations. The task was not limited only to the assessment of basic parameters, but also the quality of products, with particular emphasis on the quality of own products.

Materials

The tested products (made in Poland) were purchased in online stores named: www.ajeden.pl, www.naturalnemydla.pl, www.cocolita.pl, www.kosmetykiimpuls.pl, www.sklepbialysaibaba.pl, in autumn 2022.

The research material consisted of six bath products with the addition of goat's milk:

1. Ajeden Rose bath hemisphere with the addition of goat milk, 65 g, 7,00 zł;
2. LAVEA Bath ball with essential oils, 60 g, 12,00 zł;
3. LAVEA Goat milk bath powder, 250 g, 37,99 zł;
4. HAGI Goat milk bath powder, 400 g, 49,00 zł;
5. APIS Relaxing bath salt with honey, propolis and goat milk, 650 g, 18,00 zł;
6. HAGI Sea salt with goat milk, 400 g, 49,00 zł;

7. Own product – bath bomb (composition: Goat's milk powder, Sodium hydroxide, Citric acid, Tapioca starch, Cocoa butter, White clay)
8. Own product – bath powder (composition: Tapioca starch, Goat milk powder, Fragrance oil, Dried cornflower flowers, White clay)
9. Own product – bath salt (composition: Dead Sea Salt, Epsom Salt, Goat Milk Powder, Sweet Almond Oil, Jojoba Oil, White Clay).

Methods, results and discussion

Organoleptic examination

The appearance and color were assessed visually by observing the products in daylight, while the smell of the tested cosmetic was assessed immediately after opening the product from the packaging and pouring it onto a laboratory vessel. The tests were carried out in laboratory conditions at a temperature of approx. 20°C. The results of the analyzes are a subjective assessment of selected parameters of the analyzed bath products. The assessment was made by a team of 3 people.

Tab. 2. Organoleptic evaluation of selected preparations with the addition of goat's milk

Product	Appearance	Color	Smell
1	Compact mass, aesthetic appearance	Creamy	Rose-milky
2	Compact mass, aesthetic appearance	White	Rose-milky
3	Uniform consistency	Creamy	Milk
4	Uniform consistency	White	Milk
5	irregular shaped crystals	Light-orange	Honey and milk
6	irregular shaped crystals	Pale pink	Milk
7	Compact mass, aesthetic appearance	White	Milk
8	Uniform consistency	Creamy	Milk
9	Irregular shaped crystals	Light-orange	Milk

Source: own study based on Korzeniowski 2006.

All preparations had a characteristic milky smell immediately after opening the package and during the test. In the case of bath bombs, it was found that the products did not differ in consistency. Both ready-made and self-prepared products had a compact mass and aesthetic appearance. In the case of bath salts, each product has crystals of irregular shape and a dominant milky smell, while bath

powders were characterized by a uniform consistency, without lumps, a creamy color and a characteristic milky smell (Table 2).

pH

The pH value of cosmetics is one of the most important parameters, both from the point of view of the functioning of the skin itself and the preparation of the cosmetic formula. In order for a cosmetic to show the proper effect, preservatives or some thickeners, as well as some active substances, require a certain pH. (Bender, 2011). The skin is considered to have a pH value (assumed for normal skin) slightly acidic – within 4.5–5.5. The value of the pH factor depends primarily on the type of skin. The more oily the skin is, the lower the pH, i.e. more acidic. The pH of oily skin is usually in the range of 4–5, while for combination skin it is usually around 4.5. When it comes to dry or mature skin, the pH is closer to neutral and is between 6 and 7 (Bender, 2011).

Each of the tested bath products had a pH in the range of 6–7.5. The highest pH value had self-made products, i.e. bath salt with goat's milk with a pH of 7.5, bath powder with goat's milk with a pH of 7.4, and a bath ball with goat's milk with a pH of 6.94. The rose bath hemisphere with the addition of Ajeden goat milk and the HAGI goat milk bath powder had a pH of 6.77. Then there is a bath bomb with LAVEA essential oils with pH = 6.54, sea salt with HAGI goat milk pH = 6.52 and LAVEA goat milk bath powder 6.51. The lowest pH value was found in the relaxing bath salt with honey, propolis and APIS goat milk – 6.30 (Table 5). It can be seen that all finished products had an acidic pH, while the pH of self-made products was in the range of 7–7.5 (neutral, towards alkaline). A significant increase in the pH of the skin can cause, among others: drying, damage, the skin becomes less resistant to external factors, such as UV radiation, fungi or bacteria.

Transepidermal water loss

Small amounts of the tested preparations were applied to the skin of the forearm and then a tewameter was applied perpendicularly to the areas with the products applied and by pressing the button on the probe three times all measurements were made. The correct result is up to 25 g/m²*h, and the value read above 30 g/m²*h indicates an incorrect epidermal barrier. The measurement was made on dry skin, immediately after the application of the products, 15 minutes after, 30 minutes after and 45 minutes after the application of the tested cosmetics. The study involved 10 people aged 25–30 with mixed skin type.

The average value of transepidermal water loss from all measurements, which was measured with a tewameter before application of the products, was 7.92 [g/m²*h]. Immediately after application, the degree of water loss in each of the tested cosmetics significantly increased. The value of transepidermal water loss after 60 minutes decreased in seven cases (Table 3), i.e.: Ajeden rose bath ball

with goat milk (1.5) [g/m²*h], bath ball with LAVEA essential oils (-1) [g /m²*h], HAGI goat milk bath powder (-0.2) [g/m²*h], relaxing bath salt with honey, propolis and APIS goat milk (-0.4) [g/m²*h]], own product – bath bomb with goat's milk (-1.3) [g/m²*h], own product – bath powder with goat's milk (-1.3) [g/m²*h], own product – bath salt with goat milk (-0.5) [g/m²*h]. The smallest degree of water loss was demonstrated in the case of the rose bath hemisphere with the addition of Ajeden goat milk, and the highest in the case of LAVEA goat milk bath powder. Among the analyzed products, the rose bath hemisphere with the addition of Ajeden goat milk turned out to be the best, and the LAVEA goat milk bath powder turned out to be the worst.

Tab. 3. Water loss before and after one hour application

Product	Skin before application [g/m ² *h]	Skin one hour after application [g/m ² *h]	Difference after 1 hour after application [g/m ² *h]
1	8.9	7.4	-1.5
2	8.8	8.1	-1
3	8.1	8.4	+0.3
4	8.5	8.3	-0.2
5	7.9	7.2	-0.4
6	7.3	7.5	+0.2
7	7.1	5.8	-1.3
8	7.4	6.1	-1.3
9	7.2	6.7	-0.5

Source: own study.

Skin hydration

The degree of hydration of the epidermis was tested using a corneometer and the measurement principle is based on the analysis of the electrical capacity of the skin, thanks to which it is easy to check the water content in the stratum corneum. The higher the measurement value, the better the level of hydration of the epidermis. Normal results range from 0 to 130 IU (conventional unit) (Kmiec, 2016).

Table 4 presents the hydration values of the epidermis before application of the product and 1 hour after application. The average value of epidermal hydration (from all measurements) measured with a corneometer before applying the products to the skin was 40.07 [IU]. Immediately after applying the tested products, the level of hydration of the epidermis increased significantly. The highest moisturizing value was achieved by APIS bath salt, while the lowest was achieved by self-made bath pow-

der. After 15 minutes, a decrease in the degree of hydration was observed in all products in relation to the measurement immediately after application to the skin, while in subsequent time measurements, i.e. 30 minutes and 60 minutes after application, the values remain at a very similar level.

Tab. 4. Degree of epidermis hydration before and after 1-hour application

Product	Skin before application [IU]	Skin one hour after application [IU]	Difference after 1 hour after application [IU]
1	39.1	41.9	2.8
2	37.9	41.4	3.5
3	39.9	41.9	2
4	40.1	43	2.9
5	40.2	40.1	-0.1
6	38.6	40.6	2
7	42.1	39	-3.1
8	40.9	38.9	-2
9	41.8	40.9	-0.9

Source: own study.

The difference in the hydration value after 1 hour from the application of the Ajeden bath hemisphere is (+2.8) [IU], LAVEA bath bomb (+3.5) [IU], LAVEA bath powder (+2) [IU], HAGI bath powder (+2.9) [IU], APIS bath salt (-0.1) [IU], HAGI bath salt (+2) [IU], own product – bath bomb (-3, 1) [IU], in-house product – bath powder (-2) [IU] and in-house product – bath salt (-0.9) [IU]. From the results, it can be concluded that for most of the tested products, the level of skin hydration was slightly higher than the level of hydration before application of the product. It can also be noticed that among all bath products, the best moisturizing properties had the LAVEA bath bomb, while the worst moisturizing properties were in the difference between 1 hour after application on the skin, a self-made product - a bath bomb showed. During the study, none of the analyzed products caused allergies.

Foaming properties

Table 5 shows the results of the foam column volume of 0.1% formulation solutions measured after preparation, 1 minute, 3 minutes and 5 minutes.

Tab. 5. Foaming ability of the tested bath products

Time	Foam volume [cm ³] for 0.5% concentration			
	After whipping the foam	1 min	3 min	5 min
1	69.52	56.52	45.50	31.93
2	70.93	57.37	40.69	30.52
3	45.50	6.78	4.24	3.11
4	51.43	3.85	2.46	2.12
5	1.70	1.57	1.32	1.17
6	51.43	2.85	1.99	1.16
7	65.0	31.07	16.96	16.39
8	2.26	25.72	23.17	12.15
9	16.96	19.56	15.26	14.3

Source: own study.

The greatest foam-forming ability, immediately after foam formation, was in the bath ball with essential oils LAVEA 70.93 cm³, then the rose bath ball with the addition of Ajeden goat's milk with the value of 69.52 cm³, and then the self-made product – bath ball with goat's milk 65 cm³. The lowest foam-forming ability immediately after foam production was found in HAGI sea salt with goat's milk 1.70 cm³ and a self-made product – bath powder with goat's milk with a value of 2.26 cm³.

The highest durability of the foam was achieved by: sea salt with goat's milk HAGI with the index value of 74.52% and a self-made product – bath salt with goat's milk with the index value of 73.11%. The product that showed the least persistent foam turned out to be relaxing bath salt with honey, propolis and APIS goat milk (40.7%), while the rest of the tested products showed foam stability in the range of 45–57%.

Testing performance parameters

The test consisted in the application and evaluation of the properties of selected bath products by the testers. Each of the people participating in the study (26 people aged 20–26 with mixed skin type) received nine samples of each of the tested bath products and applied them for 9 days – each day a different product.

The test bath products were provided to the volunteers along with the appropriate, separate questionnaires, which were completed after testing the product and the final results were presented on their basis. The questionnaires contained a table in which probants marked on a scale from 1 to 5

(where: 1 – I do not like it very much, 2 – I don't like it, 3 – I like it a bit, 4 – I like it, 5 – I like it a lot) answers to the questions and additional questions.

Tab. 6. Evaluation of performance parameters of selected products

Parameter	Product								
	1	2	3	4	5	6	7	8	9
smell	4.8	4.4	3.2	5.0	4.8	4.6	3.6	3.0	4.8
color	4.2	3.6	3.6	4.4	4.8	4.4	3.6	3.4	4.2
solubility	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0	5.0
skin smoothness	4.4	4.8	3.0	4.4	4.8	4.4	4.0	3.8	3.4
hydration	3.8	4.4	3.2	4.6	4.2	4.8	3.6	3.8	3.8
frothiness	3.6	3.2	1.4	1.2	4.8	3.6	4.4	3.6	3.6
overall rating	4.2	4.0	4.0	4.6	5.0	4.8	4.2	3.6	3.8

Source: own study.

According to the respondents (Table 6), the bath powder with goat milk HAGI had the best smell, while the bath powder made by yourself was the least liked. In the case of color, the testers particularly liked the bath salt with honey, propolis and APIS goat milk, while the color of their own bath powder was the least favorite. Each of the tested products completely dissolved in water. All products received 5 points, which means that the testers had no problems with dissolving the tested products. According to the respondents, the smoothest skin was after using the bath bomb with LAVEA essential oils and HAGI goat milk bath powder, while the least noticeable skin smoothness was noticeable in the case of LAVEA goat milk bath powder, receiving an average score of 3.0. For the level of hydration, the testers particularly liked sea salt with HAGI goat milk (4.8), while the lowest level of hydration was felt by the testers in the case of LAVEA goat milk bath powder, receiving an average score of 3.2. In the case of foaming bath products, the most preferred product by the testers turned out to be a relaxing bath salt with honey, propolis and APIS goat milk with an average score of 4.8. However, products such as HAGI and LAVEA bath powders foamed the least.

The research shows that, according to the respondents, salt products proved to be the best bath additives. The best overall rating was given to APIS and HAGI bath salts. The least liked by the respondents were products made by themselves, i.e. bath powder with goat's milk, receiving an average rating of 3.6, and bath salt with the addition of goat's milk, receiving an average rating of 3.8.

Conclusions

The main purpose of using cosmetics containing goat's milk is to moisturize and smooth the skin and hair. Regular use makes the skin silky smooth, soft, elastic, looks young and has a healthy color.

The conducted research allowed to compare selected features of ready-made and self-made bath products with the addition of goat's milk.

- Organoleptic examination confirmed a noticeable milky smell of all analyzed preparations.
- Bath bombs, both ready-made and self-prepared, had a uniform compact mass, bath salts had crystals of irregular shape, while the tested bath powders had a uniform consistency and cream color.
- The pH of the tested products was in the range of 6–7.5.
- The tewameter test confirmed that the best film on the skin is obtained after using bath bombs (results from the product composition), and the worst in the case of powders (the lowest degree of TEWL – product No. 1, the highest – No. 3).
- Analysis of the degree of epidermis hydration – the highest hydration value after an hour from application was obtained after using APIS bath salts, and the lowest – self-made powder.
- Tested products did not have adequate foaminess for the consumer. Foam is an important parameter for users. The highest ability to produce foam had the bath bomb with essential oils LAVEA 70.93 cm³, while the smallest sea salt with goat's milk HAGI 1.70 cm³ and a self-made product – bath powder with goat's milk with a value of 2.26 cm³.
- The testers preferred ready-made products more, which could be primarily due to the specific smell of goat's milk, particularly perceptible in freshly prepared preparations.
- In-house products also differed from finished products in terms of skin hydration and color acceptability.
- The main advantage of making your own bath product is the ability to personalize the product to your needs by adding the right amount of individual ingredients. As a result of a well-prepared recipe, you can obtain a product perfectly suited to the needs of the skin.

The most frequently mentioned advantages of natural products are: lack of allergic skin changes, irritation, contact dermatitis, etc. The interest in natural cosmetics is influenced by reports of possible hormonal disorders of the body as a result of using preparations containing a large amount of chemical compounds (Kozioł, 2016).

Focusing on ingredients from a reliable source and the ability to prepare them yourself (influence on the recipe, low price, pleasure of production) guarantee that the DIY cosmetics trend has a chance for further development. Consumers interested in ecology, and above all, caring about the quality of

life and the products they use, often collect information and experiment with cosmetics at home. As research shows, own cosmetics do not differ much in terms of usable quality from commercial ones. In the case of DIY products, you must not forget about their proper storage and short shelf life.

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THE INFLUENCE OF AZELAIC, SUCCINIC AND GALLIC ACIDS ON THE IRRITATING POTENTIAL OF SHOWER GELS

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DOI: 10.56091/CTQS.Qual-16

Abstract

Ease of use and versatility make shower gels the most popular group of body wash products in liquid form. Body wash products typically use surfactants in concentrations of 10–20%. Such a significant share of surfactants in the composition correlates with the occurrence of skin irritation. Nowadays, the challenge is to improve the quality of personal hygiene products, including body wash products, manifested by their lower irritating potential. This goal is most often attempted to be achieved by introducing plant extracts or active substances into the composition.

Irritations and disorders in the hydrolipidic layer of the skin are not the only unfavorable phenomena that a person struggles with. In recent years, there has been an increase in the incidence of acne, also in adulthood. The aim of this study was to determine the effect of azelaic acid, succinic acid and gallic acid on the irritating potential, the surface activity and wetting properties of model shower gels. The skin irritating potential was evaluated based on determination of the zein value and the increase in the pH level of the bovine serum albumin (BSA) solution while for the surface activity and wetting properties evaluation the Wilhelmy method was used. The results of zein test were below 200 mg N/100 ml which means that tested model shower gels could be classified as non-irritants.

Keywords: shower gels, cosmetics safety, quality of personal care products

Introduction

Commercially available shower gels are aqueous solutions of anionic surfactants. Typically, the formulation contains 10%-20% surfactants. Amphoteric and nonionic surfactants are usually added to improve the performance properties. In order for the product to meet the consumer's expectations, the composition usually also includes pH regulators, colorants, viscosity modifiers and fragrance compositions (Bujak et al., 2015; Wasilewski et al., 2022). The cosmetics market is currently dominated by several trends, one of them is the use of multifunctional ingredients in formulations that would act on many levels, e.g. plant extracts (Nizioł-Łukaszewska et al., 2020). Due to structure and adsorption properties, surfactants are responsible for the washing effect generated by shower gels, but their adsorption properties are also important in causing skin irritation (Nizioł-Łukaszewska et al.,

2017). The intensity of this effect depends on the type of surfactant, its concentration and contact time. There are data indicating that a higher irritating potential is noted for solutions with a concentration lower than CMC (critical micelle concentration) (Rhein & Simion, 1991; Rhein et al., 1986). The two most important mechanisms responsible for skin irritation are related to the interaction of surfactants with proteins or lipids of the stratum corneum. Due to the possibility of strong electrostatic interactions, the ionic surfactant is attributed a greater tendency to interact with the proteins of the stratum corneum. As a result of the interaction of surfactants with keratin proteins, the α -helix may be denatured. Further damage to the secondary and tertiary structure of proteins results in swelling of the stratum corneum and leaching of proteins from the skin structure. Sequence of these effects makes the skin easier to penetrate by next particles (Seweryn, 2018). It seems that currently the prevailing belief is that both surfactant monomers and micelles are capable of denaturing the proteins of the stratum corneum (Walters et al., 2012). Although originally the belief in the accuracy of the model of skin penetration by a surfactant monomer prevailed, today there are studies that argue with this theory (Lu & Moore, 2012; Moore et al., 2003). The irritating effect of surfactants is also related to the interaction of surfactants with lipids forming the intercellular cement of the stratum corneum. This phenomenon is associated with both the activity of non-aggregated molecules and micelles. It seems that monomers are able to adsorb and integrate into the liquid crystal structure of the stratum corneum, changing its permeability, which in turn may lead to further migration of monomers. On the other hand, as a result of contact of the skin with a surfactant solution whose concentration exceeds the CMC, lipids of the stratum corneum can solubilize and, as a result, they can also be washed out (Ananthapadmanabhan et al., 2004; Ananthapadmanabhan et al., 2013; Seweryn, 2018). The consequence of interaction with proteins and lipids is the possibility of skin toxicity. Surfactants, penetrating into the deeper layers of the epidermis, interact with the cytoplasm of keratinocytes, causing their permanent damage and ultimately cell death (Lee et al., 2000).

It seems that the easiest way to reduce the irritating potential of personal care products is a well-thought-out selection of surfactants in the composition and actions that increase the size of micelles and reduce the concentration of monomers. It was found that ionic surfactants, due to the possibility of generating hydrophobic and electrostatic interactions with the proteins of the stratum corneum, stimulate the formation of skin irritation more than non-ionic surfactants (Bromberg et al., 2017; Cserháti et al., 2002; Seweryn, 2018). Reducing the irritating potential can also be obtained by adding polymers. Interactions of various strengths are formed between surfactants and polymers, these are usually electrostatic, dipole-dipole and hydrophobic interactions as well as hydrogen bonds. The type of resulting interactions depends mainly on the molecular structure of the molecules, including the nature of the hydrophilic and hydrophobic group, but also on the charge density, conformation and

flexibility of the polymer chain (Penfold et al., 2006; Seweryn, 2018). It is believed that the formed large surfactant-polymer complexes reduce the irritating effect of surfactants, among others, as a result of slower surfactant dynamics (Fevola et al., 2010; Walters et al., 2012). Another approach developed for polyethylene oxide assumes that the polyethylene oxide chains wrap around the surfactant micelles and bind water, making the whole system more biocompatible (Walters et al., 2008). Sometimes, the reduction of the irritating effect can be achieved by adding refatting substances to the product, which will limit the access of monomers to the skin surface by creating an occlusive layer on the surface of the epidermis. Another popular approach involves the addition of hydrophobic plant extracts to the formulation. Surfactants present in the washing bath are then partly involved in the process of solubilization of hydrophobic molecules, thus reducing the concentration of surfactants that may come into direct contact with the skin (Wasilewski et al., 2016).

Azelaic, gallic and succinic acids are naturally occurring compounds with proven bioactive properties. Due to antioxidant, antimicrobial, anti-inflammatory and keratolytic activity, these acids are used in the production of preparations for acne skin, e.g. creams, gels or chemical peels (Manosroi et al., 2016; Reszke & Szepietowski, 2016).

Based on available literature data, following hypothesis were formulated:

- The addition of azelaic acid, gallic acid and succinic acid causes a decrease in the zein value and reduces changes in pH value in pH rise test with bovine serum albumin (BSA).
- The addition of azelaic acid, gallic acid and succinic acid causes a decrease in the contact angle and surface tension value.

The aim of this study was to determine the effect of azelaic acid, succinic acid and gallic acid on the irritating potential, the surface activity and wetting properties of model shower gels.

Materials and methods

Chemicals

Model shower gels were prepared using: Sodium Laureth Sulfate (Sulforokanol L225/1, PCC Group), Cocamidopropyl Betaine (Rokamina K30, PCC group), Sodium Chloride (Chempur), Citric Acid (Stanlab), Sodium Hydroxide (0.1 mol/dm³, 0.1 N, Chempur), Gallic Acid (Aktyn), Succinic Acid (Aktyn) and Azelaic Acid (Ecospa). Measurements of irritant potential were performed with: copper sulphate pentahydrate, potassium sulphate, sulfuric acid 98%, sulfuric acid (0.1 mol/dm³, 0.2 N), sodium hydroxide (0.1 mol/dm³, 0.1 N) (all purchased from Chempur), bovine serum albumin (BSA) and zein

from corn (both from Sigma-Aldrich). All chemical reagents used in the experiment were analytical grade.

Model shower gels preparing

According to the data given in Table 1, two commercial surfactants such as Sodium Laureth Sulfate and Cocamidopropyl Betaine were used to prepare model shower gels. The amount of Sodium Chloride (viscosity regulator) used in formulation depended on the desired viscosity, which was set at approx. 3500 mPa·s. The dynamic viscosity of the gels was measured using a Brookfield viscometer (DV2T, Brookfield AMETEK, Germany). Citric Acid and Sodium Hydroxide were used as pH regulators (to pH 5.5±0.1). The factor differentiating the composition of the analyzed shower gels was the type of used active ingredient, i.e. Succinic, Azelaic or Gallic Acid. A shower gel containing no active components was also prepared as reference sample.

Tab. 1. Formulation of the model shower gels

Component (INCI name)	Formulations of tested shower gels			
	Concentration (%)			
	SG+SA	SG+AA	SG+GA	SG
Sodium Laureth Sulfate (SLES)	9.5			
Cocamidopropyl Betaine (CAPB)	0.5			
Sodium Chloride	To dynamic viscosity approx. 3500 mPa·s			
Succinic Acid	0.5	0.0	0.0	0.0
Azelaic Acid	0.0	0.5	0.0	0.0
Gallic Acid	0.0	0.0	0.5	0.0
Citric Acid or Sodium Hydroxide	To pH approx.5.5			
Aqua	Ad 100			

Designations of the model shower gels due to the used active component (applied throughout the manuscript): SG+SA – Succinic Acid, SG+AA – Azelaic Acid, SG+GA – Gallic Acid, SG – shower gel containing no active components.

Source: authors' own work based on Bujak et al., 2015.

Preparation of model shower gels consisted in combining the above-mentioned components at the concentrations specified in Table 1. Sodium Laureth Sulfate and Cocamidopropyl Betaine were added to the measured amount of water. After mixing the contents, respectively, active ingredient and Sodium Chloride were introduced into the formulation. Finally, pH was adjusted with Citric Acid or Sodium Hydroxide (Bujak et al., 2015).

Determination of the skin irritation potential by zein test

Zein test was performed according to the method described by Nizioł-Łukaszewska et al. (2017a). 2 ± 0.05 g of zein from corn was added to 40 cm^3 of a 10% aqueous solution of the prepared shower gel. The mixture was shaken on a shaker with water bath (SWB 22N, Laboplay, Poland) at 35°C for 60 min, then cooled and centrifuged (M-Science, MPW, Poland) at 5000 rpm for 10 min.

The nitrogen content was determined by the Kjeldahl method. For this purpose, 15 cm^3 of 98% sulfuric acid, 0.35 ± 0.01 g of copper sulphate pentahydrate and 7.5 ± 0.01 g of potassium sulphate were added to 1 cm^3 of the centrifuged solution and mineralized using a Kjeldahl digestion apparatus (SpeedDigester K-425, Buchi, Switzerland). Then, the mineralizate was diluted with 20 cm^3 of distilled water and 12 cm^3 of 33% sodium hydroxide solution was added. Steam distillation of released ammonia was performed using an automated distillation unit (K-350, Buchi, Switzerland) into a receiver containing 5 cm^3 of 0.1 N sulfuric acid. The unbound sulfuric acid was titrated with 0.1 N sodium hydroxide using Tashiro solution as an indicator. The zein value (ZV) expressed as mg N/100 ml was calculated from the following equation:

$$ZV = (10 - V_1) \cdot 100 \cdot 0.7$$

V_1 – the volume (cm^3) of 0.1 N sodium hydroxide used for titration.

Determination of the skin irritation potential by pH rise test with bovine serum albumin (BSA)

pH rise test with bovine serum albumin (BSA) was performed according to the method described by Klimaszewska et al. (2019). Before the test, 2% aqueous solution of bovine serum albumin (BSA) was prepared. This solution was then mixed with a 10% aqueous solution of the prepared shower gel in a ratio of 1:1. The pH of solutions was earlier adjusted to 5.5 with 50% citric acid or 0.1 M sodium hydroxide. The mixtures were left to stand for 48 h after which the pH value was measured (pH-meter CP-551, Elmetron, Poland). The percentage increase in pH value was calculated according to following equation:

$$\Delta pH = \frac{pH_r - pH_0}{pH_0} \cdot 100\%$$

ΔpH – change in pH value (%)

pH_r – pH value of mixtures measured after 48 h,

pH_0 – pH value of 5.5, considered the pH value of the health human skin.

Statistical analysis

All results were expressed as mean values of three determinations. Moreover, standard deviations were calculated and a one-way analysis of variance (one-way ANOVA) was performed. The significant differences between mean values were analyzed using Tuckey's test at $\alpha=0.05$. For the analysis Statistica 12.0 software (StatSoft, Inc. 2013) was used.

Evaluation of the surface properties

In order to assess the surface properties, surface tension measurements were made using the Kruss K100 tensiometer (Germany). The test was carried out using the Wilhelmy method with the use of a platinum plate which was burnt in a flame several times during the analysis. 5% water solutions of prepared shower gels were tested.

Evaluation of the wetting properties

The wetting properties of the paraffin film surface were determined using a Kruss K100 tensiometer. 2.5% aqueous solutions of the obtained shower gels were used in the study, following the Wilhelmy method.

Results and discussion

The skin irritation potential of model shower gels was investigated by two methods: zein test and pH rise test with bovine serum albumin (BSA). The tested model shower gels differed in the type of active ingredient used, i.e. azelaic acid, gallic acid and succinic acid. Based on the obtained data, the effect of the active ingredients on irritation potential of shower gels was assessed. The results were analyzed in relation to the sample without the additives.

Literature data (Bujak et al., 2015; Sułek et al., 2018) show a specific classification of cosmetics that, based on the zein value, distinguishes non-irritants (<200 mg N/100 ml), moderately irritants (200 – 400 mg N/100 ml) and strong irritants (>400 mg N/100 ml) to the skin. As shown in Figure 1, zein values obtained in this experiment ranged from 140 mg N/100 ml to 196 mg N/100 ml which indicates that all tested shower gels could be classified as non-irritants. Nevertheless, shower gels containing Gallic Acid or Azelaic Acid as well as referenced shower gel shown zein values, only slightly lower than 200 mg N/100 ml, above which cosmetics are classified as moderately irritant. Moreover, we found that among the active components used in our study only succinic acid caused the reduction of the irritation potential of tested shower gel. The zein value obtained for this sample was 140 mg N/100 ml.

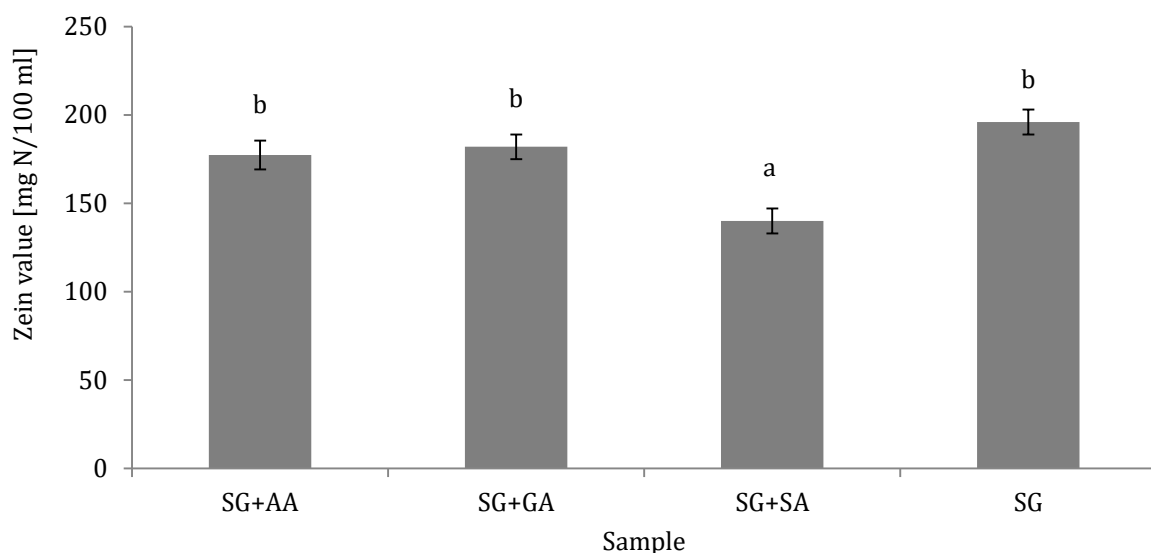


Fig. 1. Zein values of model shower gels

Mean values marked the different letter were significantly different at $\alpha = 0.05$

Source: authors' own studies.

According to the literature data, one of the key factors causing skin irritation are interactions between skin proteins and surfactants contained in the washing bath. Since the 1990s, one of the most widely used combinations of surfactants in body cleansers is the combination of Sodium Laureth Sulfate (SLES) and Cocamidopropyl Betaine (CAPB) as was the used in this study (Ananthapadmanabha, 2019). It has been proven (Klimaszewska et al., 2019) that anionic surfactants can interact via electrostatic forces with the proteins in the stratum corneum, which is manifested by enzyme inactivation, tissue swelling and removing of water-soluble low molecular weight components. Nevertheless, according to one of approaches, this applies primarily to surfactant molecules present in the solution in the form of monomers. As anionic surfactant, pure SLES exhibits relatively high irritation potential (Bujak et al., 2015) compared to other surfactants, but due to presence of an ethoxy group in the molecule it is milder surfactant than, e.g. Sodium Lauryl Sulfate (SLS) (Ananthapadmanabha, 2019). Moreover, addition of certain components may cause a decrease in the irritating potential of aqueous solutions of SLES, as was confirmed in different studies (e.g. Bujak et al. 2015; 2020; Klimaszewska, et al. 2019). For example, Cocamidopropyl Betaine by becoming incorporated into the structure of micelles created by anionic surfactants may increasing their size, act as a stabilizer and reduce the concentration of monomers in the solution that counteracting their interaction with skin proteins (Bujak et al., 2015).

The irritating potential of succinic acid as potential ingredient of cleaners has been studied by Hwang et al. (2022). The test of skin irritation in 3D-reconstructed human epidermis model shown that exposition to succinic acid at the concentration of 0.1 mol/dm^3 caused no significant decrease in cell

viability. The negative changes were observed at a concentration of 1.0 mol/dm^3 . In the same study, the authors, referring to secondary sources, described succinic acid as non-irritant to the skin. However, these studies concerned pure succinic acid, and there are no available data on the irritating potential of model preparations or commercial products containing this additive. On the other hand, described in the literature *in vivo* studies regarding the effect on the skin of selected model preparations containing gallic or azelaic acids indicated the possibility of local irritation occurring manifested by redness or minor inflammation (Manosroi et al., 2011, Reszke & Szepietowski, 2016).

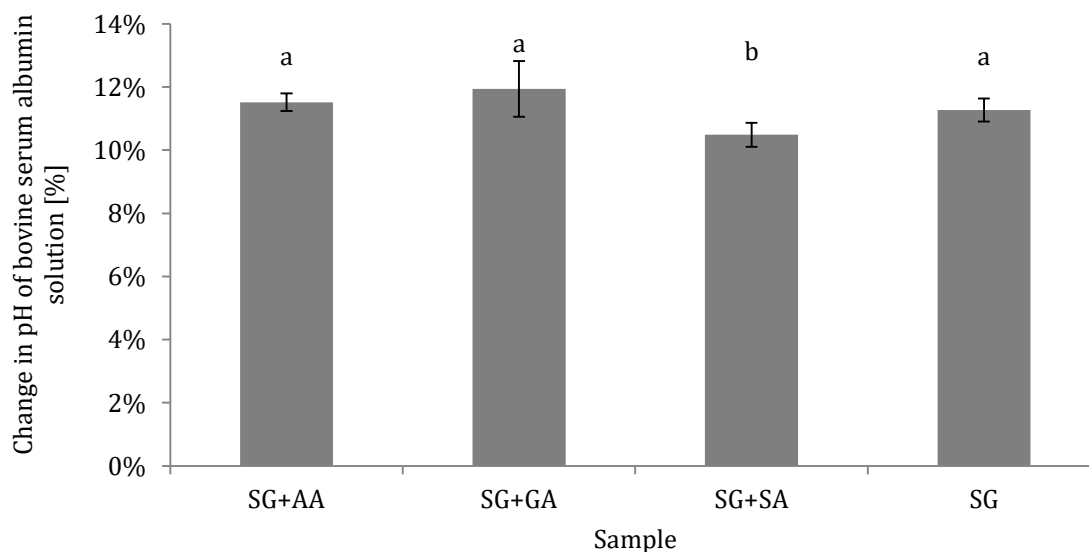


Fig. 2. Changes in pH of bovine serum albumin (BSA) and model shower gels mixtures

Mean values marked the different letter were significantly different at $\alpha = 0.05$

Source: authors' own studies.

pH rise test with bovine serum albumin (BSA) consisted of measuring the pH of a mixture of 10% aqueous shower gel solution and 2% aqueous bovine serum albumin (BSA) solution after 48 hours. Changes in the albumin structure as a result of denaturation under the influence of a surfactant leads to an increase in the pH of the solution. It is recognized that the higher the pH increase is, the greater the potential irritating effect that may be observed (Klimaszewska et al. 2019; Seweryn, 2018). As can be seen in Figure 2., the results of pH rise test with bovine serum albumin (BSA) are consistent with those of zein test. The lowest increase in pH by approx. 10.5% was observed in shower gel containing succinic acid, while the other shower gels contributed to a slightly higher increase in pH, which was at the level of 11.3 to 11.9% (these mean values were not significantly different at $\alpha = 0.05$) which confirms that succinic acid was the only active component used in this study able to reduction of irritation potential induced by SLES.

A parameter widely used to approximate wetting properties is the contact angle (θ_v). It is assumed that when its value is less than 90° the liquid wets the tested surface, a special case is when the angle is equal to zero, then the phenomenon of complete wetting of the surface occurs (Bruel et al. 2019). A paraffin film was used as the test surface because it is considered to sufficiently imitate human skin. In this study, a comparison of the contact angles obtained for water solutions of the analyzed shower gels containing 0.5% azelaic, gallic and succinic acids was performed. A slight decrease in the value of the contact angle was observed for gels containing added acids in comparison with the shower gel without additives (Table 2). Certainly, the formation of a more closely packed adsorption layer and thus the impact on the contact angle values is related to the elimination of the mutual repulsion of the anion heads, which is influenced by the presence of succinic, gallic and azelaic acids (Kwaśniewska & Kiewlicz, 2022).

Tab. 2. Values of surface tension and contact angles for solutions of the shower gel (SG) and gels containing Azelaic Acid (SG+AA), Gallic Acid (SG+GA) and Succinic Acid (SG+SA)

	SG+AA	SG+GA	SG+SA	SG
σ [mN/m]	31.61	31.24	31.15	31.61
θ_{Adv} [°]	65.75	63.86	62.68	67.63

Source: authors' own studies.

Reducing the surface tension of water is an essential factor in pollutant removal processes. In this work, the value of surface tensions of solutions of obtained shower gels with a concentration of 5% was compared. Although the value of the surface tension is influenced by a number of factors, including the presence of some organic compounds (Zieliński, 2021), the presence of azelaic, gallic and succinic acids in the tested compositions did not cause a significant change in the surface tension compared to the gel solution with the basic composition.

Conclusions

The study was attempted to determine the effect of azelaic acid, succinic acid and gallic acid, on the irritating potential, the surface properties and wetting properties of model shower gels. The results of zein test obtained in this study were below 200 mg N/100 ml which means that tested model shower gels could be classified as non-irritants. Moreover, based on zein test and pH rise test with bovine serum albumin (BSA), it was observed that the addition of succinic acid at the concentration of 0.5% may contribute to reduction of irritation potential compared to additive-free shower gel. This allowed for a partial positive verification of the first hypothesis. On the other hand, the addition of the analyzed acids did not significantly affect the wetting properties and surface activity. This proves

that the second hypothesis was verified negatively. The obtained results complement the current knowledge on the impact of the addition of selected active ingredients on properties of body wash products, but they do not completely exhaust the topic. An interesting aspect would be to conduct rheological tests of analogous systems.

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SHAPING THE QUALITY OF COSMETIC OILS FOR ATOPIC SKIN CARE

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DOI: 10.56091/CTQS.Qual-17

Abstract

Atopic dermatitis (AD) is an inflammatory disease with a chronic relapsing course. In AD, there is a defect in the functioning of the natural skin barrier, associated with increased transepidermal water loss. Dryness, intense itching and inflammation of the skin are characteristic features that occur in AD. The basic element of atopic skin care is the reconstruction of the epidermal barrier, involving the use of appropriate cosmetic and therapeutic preparations. The key raw materials used in the formulations of care products for atopic skin are emollients with occlusive properties, protecting against excessive evaporation of water from the deeper layers of the skin. In this study, an attempt was made to develop a formulation of cosmetic oils in gel form. Six ingredients acting as viscosity modifiers in the formulation and three base vegetable oils were selected for the study. The influence of the type of rheology modifier and vegetable oil on the functional and physicochemical properties of oils in gel form was demonstrated. It was found that the most favorable properties were characterized by the oil in gel form containing hydrogenated vegetable oil at a concentration of 5% as a rheology modifier.

Keywords: atopic dermatitis, cosmetic oils, quality, rheology modifiers, vegetable oils

Introduction

The key raw materials used in skin care formulations for atopic skin are emollients. These are hydrophobic raw materials with occlusive properties, causing the formation of a thin protective film on the epidermal surface. Emollients protect against excessive evaporation of water from the deeper layers of the skin, causing a reduction in Transepidermal Water Loss (TEWL). Raw materials with an occlusive effect include: esters, triglycerides, fatty alcohols, fatty acids, ceramides, cholesterol, vegetable and animal waxes, mineral oils, silicone oils (Jaworek et al., 2020; Kamińska, 2021; Welz-Kubiak & Reich, 2016). In connection with the growing trends of veganism in the world, vegetable oils play an important role in the market of cosmetic raw materials including emollients. According to Report Linker, the vegan cosmetics market is expected to grow by \$5872.81 million between 2023 and 2027, accelerating at a 6.5 percent compound annual growth rate (Dos Santos et al., 2023; Bozza, 2022). Vegetable oils are fats of liquid consistency extracted from various parts of oily plants, e.g. seeds,

fruits, seeds. Their properties depend on the percentage of individual saturated and unsaturated acids in the fat molecule (Zielińska & Nowak 2016). Vegetable oils are valued raw materials commonly used in the cosmetic or pharmaceutical industry, including in facial and body care preparations (Kulawik-Pióro et al. 2020). Emollients in cosmetic formulations can be a base or act as a complementary ingredient. For many years, the term "emollient" referred only to a raw material, being a component of a cosmetic formulation. Nowadays, we encounter emollients on the market as cosmetic products containing in their composition mainly "emollients" - hydrophobic ingredients and humectants. They are in the form of oil-in-water (O/W) or water-in-oil (W/O) emulsions, but can also be in the form of a mixture without an emulsifier, such as cosmetic oils (Kamińska, 2021; Welz-Kubiak & Reich, 2016).

Emollient preparations are an important part of the prevention and supportive treatment of many dermatoses. They can be used in monotherapy or as an adjunct to intensive treatment. One example of skin diseases in which emollients play a key role in prevention and treatment is atopic dermatitis (AD) (Loden, 2003; Penzer et al., 2012). It is an inflammatory disease with a chronic relapsing course. According to the World Health Organization (WHO), it is now the most common dermatosis in the world. In AD, there is a defect in the functioning of the natural skin barrier, associated with increased transepidermal water loss. Dryness, intense pain, itching and inflammation of the skin are characteristic features that occur in AD (Grobe et al., 2019; Luger et al., 2021; Millan & Mijas, 2017). Atopic dermatitis is also associated with sensory hypersensitivity (Engel-Yeger et al., 2011; Shani-Adir et al., 2009).

The basic element of atopic skin care is the reconstruction of the epidermal barrier, involving the use of appropriate cosmetic and therapeutic preparations. As a result of the use of emollient care preparations, the epidermis is regenerated and its functioning is improved. The skin becomes more elastic, protected against damage, cracking, peeling and penetration of undesirable exogenous substances (Jaworek et al., 2020; Kamińska, 2021; Nowicki, 2019; Welz-Kubiak & Reich 2016). Cosmetic products containing emollients, depending on their raw material composition, can have an antipruritic and anti-inflammatory effect on the skin through penetrating lubrication. Authors Narbutt et al. based on their own research showed that emollient preparations have a high therapeutic efficacy in the treatment of dry skin symptoms in the course of AD in children and adults. They found that during the application of emollient products there was an improvement in skin condition by increasing skin hydration and lubrication. The emollients also showed antipruritic effects (Narbutt et al., 2016). In the case of cosmetic products for atopic skin, it is indicated to design formulations with a simple composition, since the large number of raw materials in the formulation increases the risk of the

appearance of allergic reactions on the skin. Particular attention should be paid to the ingredients of fragrances and preservatives (Rudyk & Jurzak, 2012; Śliwa 2011).

In this study, an attempt was made to develop an emollient preparation as a three-component cosmetic oil in gel form. The choice of this type of cosmetic was dictated by the essential content in the formulation of emollients, so important in the prophylaxis and support of AD treatment, which has been confirmed by many scientific reports. In addition, the formulations will be selected in such a way as to obtain the gel form of the hydrophobic system. The commonly known form of cosmetic oil is characterized by low viscosity, which does not fully meet the consumer's expectations in terms of application. A similar study was conducted by Misiak et al. where raspberry seed and hemp seed oils were homogenized with the addition of silica or a mixture of caprylic-capric triacylglycerol with polyurethane-79 to obtain oleogels. Effective gel form was obtained for both oils obtained with a polymeric gelling agent or silica used at 4.5 and 6 wt. %, respectively (Misiak et al., 2021). On the other hand, Authors Balasubramanian et.al in their work showed that oleogels are characterized by simplicity of the technology of obtaining, high physical, chemical and mechanical stability and better in vivo efficacy, which makes them suitable for use as bases for topical preparations. In addition, the study hypothesized that the gel form of an emollient formulation could contribute to the sensory attractiveness of such products during use in AD patients (Balasubramanian et al., 2012). While the authors Engel-Yeger et al. found that AD patients showed higher sensory sensitivity than control subjects, mainly in the area of tactile modality (Engel-Yeger et al., 2011). In this study, the effects of the type and concentration of rheology modifier and vegetable oil on the usable and physicochemical properties of cosmetic oils in gel form were analyzed. A number of physicochemical and functional tests were performed for the developed gel forms of oils.

Materials and methods

Materials

The following raw materials were used to make a series of cosmetic olives in gel form: Hydrogenated Vegetable Oil (Hydrobase 57/63) by PROD'HYG; Silica (Aerosil) by Evonik; Aluminum Stearate by Strem Chemicals; Zinc Stearate by P.P.H. Stanlab; Cera Alba (Beeswax) by Ecospa; Cera Microcristallina (Microcrystalline Wax) by OQEMA; Helianthus Annuus (Sunflower) Seed Oil by Ecospa; Prunus Amygdalus Dulcis (Sweet Almond) Oil by Standard Sp. Ltd; Vitis Vinifera (Grape) Seed Oil by Ecospa; Tocopheryl Acetate (Vitamin E) by Ecospa.

Formulas preparation

Preparation of the care oils (Tab.2 and Tab.4) involved heating vegetable oil on a water bath to 70°C. Then, oils with a 5 wt.% content of silica, zinc stearate and aluminum stearate were dissolved using a mechanical stirrer for 10 minutes at 500 rpm. Samples with a 10 wt.% content of the above-mentioned raw materials were dissolved using a mechanical stirrer for 10 minutes at 700 rpm. On the other hand, oils with hydrogenated vegetable oil, beeswax and microcrystalline wax contents of 5 wt% and 10 wt% were dissolved on a water bath using a magnetic stirrer. After all components were dissolved, the samples were cooled to 30°C and vitamin E was added.

Stability

Stability testing was done by organoleptic, visual evaluation of cosmetic oil samples. The formulations were stored for a period of 6 weeks at 21°C. During the given time, any signs of instability of the products were observed and noted every 7 days.

Dynamic viscosity

The dynamic viscosity (η) of cosmetic oils was tested using a Brookfield DV-1+ viscosity meter. Measurements were carried out at a speed of 10rpm and at 21°C. The measurements were performed 5 times. The results shown in the graph are averaged values.

Yield point

The Yield point values of the tested cosmetic oils were determined using a Brookfield HADV III Ultra rheometer from Brookfield equipped with a set of paddle spindles (vanespindle). Measurements were carried out at a constant spindle speed of 1rpm. EZ-Yield Software was used to record the measurements and analyze them.

Consumer evaluation of sensory appeal

Consumer evaluation of the sensory appeal of the developed cosmetic oils in gel form was carried out in three categories: evaluation of the product during application, sensation after application, and evaluation of the product in the hand. For product evaluation during application, the following sensory parameters were analyzed: spreading, absorption, stickiness. In the case of post-application sensations, 0.5 ml of the test product was applied to the skin of the forearm then after 30 minutes the following sensory parameters were analyzed: smoothness and greasing. For the evaluation of the product in the hand, the following sensory parameters were analyzed: homogeneity, adhesion and consistency. Characterization of sensory parameters was presented in the works of Kulawik-Pióro et

al. (2020) and Szakiel & Turek (2019). The analysis was carried out using the scaling method in a group of ten (women aged 20–25). The preparations were evaluated based on a 5–point scale, which are shown in Table 1.

Tab. 1. Sensory parameters and scoring scale of consumer evaluation of sensory appeal

Evaluation of the product during application	Sensation after application	Evaluation of the product in the hand
After 10 laps	After 30 minutes	
Spreading 1. bad 2. insufficient 3. satisfactory 4. good 5. very good	Smoothing 1. None 2. low 3. medium 4. high 5. very high	Homogeneity 1. bad 2. insufficient 3. satisfactory 4. good 5. very good
Absorption 1. none 2. low 3. medium 4. high 5. very high	Greasing 1. very high 2. high 3. medium 4. low 5. none	Adhesion 1. none 2. low 3. medium 4. high 5. very high
Stickiness 1. very high 2. high 3. medium 4. low 5. none		Consistency 1. bad 2. insufficient 3. Sufficient 4. good 5. very good
Maximum number of points		
15	10	15

Source: own compilation based on Kulawik-Pióro et al. (2020) and Szakiel & Turek (2019).

Measurement Error

The points in the charts represent mean values from a series of three or five independent measurements. The t-distribution was used to calculate confidence limits for the mean values. Confidence intervals, which constitute a measuring error were determined for the confidence level of 0.90. Error values are presented in the Figures.

Results and discussion

The experimental part of the work was divided into two stages. In the first stage, the effect of the type and concentration of the rheology modifier on the physicochemical properties of cosmetic oils was studied. In the second stage, the effect of the type of vegetable oil on the physicochemical and usable properties of cosmetic oils in gel form was analyzed.

Effect of type and concentration of rheology modifier on physicochemical properties of cosmetic oil in gel form

The developed formulations of cosmetic oils (Tab.2) contain the following rheology modifiers: hydrogenated vegetable oil, silica, aluminum stearate, zinc stearate, beeswax, microcrystalline wax. Helianthus Annuus (Sunflower) Seed Oil was used as the base vegetable oil in the care oils. In addition, vitamin E (Tocopheryl Acetate) was used in the formulation.

Tab. 2. Formulations of cosmetic oils in gel form depending on the concentration and type of rheology modifier

INCI name	Concentration [wt.%]											
	S1_5 %	S2_5 %	S3_5 %	S4_5 %	S5_5 %	S6_5 %	S1_1 0%	S2_1 0%	S3_1 0%	S4_1 0%	S5_1 0%	S6_1 0%
Hydrogenated Vegetable Oil	5.0	-	-	-	-	-	10.0	-	-	-	-	-
Silica	-	5.0	-	-	-	-	-	10.0	-	-	-	-
Aluminium Stearate	-	-	5.0	-	-	-	-	-	10.0	-	-	-
Zinc Stearate	-	-	-	5.0	-	-	-	-	-	10.0	-	-
Cera Alba	-	-	-	-	5.0	-	-	-	-	-	10.0	-
Cera Microcrystallina	-	-	-	-	-	5.0	-	-	-	-	-	10.0
Helianthus Annuus (Sunflower) Seed Oil	Up to 100											
Tocopheryl Acetate	0.5											

Source: own study.

Preparation of the care oils involved heating sunflower oil on a water bath to 70°C. Then, oils with a 5wt.% content of silica, zinc stearate and aluminum stearate were dissolved using a mechanical stirrer for 10 minutes at 500 rpm. Samples with a 10wt.% content of the above-mentioned raw materials were dissolved using a mechanical stirrer for 10 minutes at 700 rpm. On the other hand, oils with hydrogenated vegetable oil, beeswax and microcrystalline wax contents of 5wt% and 10wt% were dissolved on a water bath using a magnetic stirrer. After all components were dissolved, the samples were cooled to 30°C and vitamin E was added.

The next stage of the work was to evaluate the stability of the produced cosmetic oils. The results of assessing the stability of oils in gel form based on sunflower oil and with 5wt.% and 10wt.% concentration rheology modifiers are shown in Table 3.

Tab. 3. Results of stability evaluation of oils in gel form based on sunflower oil and with 5wt.% and 10wt.% of rheology modifiers

Week	Stability											
	S1_5%	S2_5%	S3_5%	S4_5%	S5_5%	S6_5%	S1_10%	S2_10%	S3_10%	S4_10%	S5_10%	S6_10%
1	+	+	+	+	+	+	+	+	+	+	+	+
2	+	-	-	-	-	+	+	+	+	+	+	+
3	+	-	-	-	-	+	+	+	+	+	+	+
4	+	-	-	-	-	+	+	+	+	+	+	+
5	+	-	-	-	-	+	+	+	+	+	+	+
6	+	-	-	-	-	+	+	+	+	+	+	+

"+" stable preparations; "-" unstable preparations.

Source: own study.

Based on the results, the following cosmetic oils in gel form were found to be unstable: S2_5% with silica; S3_5% with aluminum stearate; S4_5% with zinc stearate; S5_5% with beeswax, in which signs of instability, delamination of the system, were observed after 14 days of storage at room temperature. The mentioned cosmetic oils due to their instability were excluded from further studies.

Figure 1 shows the results of dynamic viscosity of cosmetic oils in gel form depending on the concentration and type of rheology modifier.

Based on the results, it was found that the dynamic viscosity value for the produced oils in gel form varies depending on the concentration and the type of raw material used as a rheology modifier. The lowest viscosity value (456 mPa·s) was obtained for oil containing aluminum stearate at a concentration of 10wt.%. In contrast, the highest viscosity values (72867 mPa·s and 62700 mPa·s) are characterized by oils S1_5% and S5_10% in which hydrogenated vegetable oil and beeswax were used as a rheology modifier, respectively. A similar study was conducted by authors Borriello et al. (2021) whose research aimed to develop new oleogels based on pumpkin seed oil and natural waxes. They found that oleogels made from pumpkin seed oil and beeswax were weaker than those made from sunflower seeds. In addition, pumpkin seed oil-based oleogels structured with carnauba wax

showed higher viscoelastic properties, retained more oil and were harder than beeswax-based oleogels (Borriello et al., 2021).

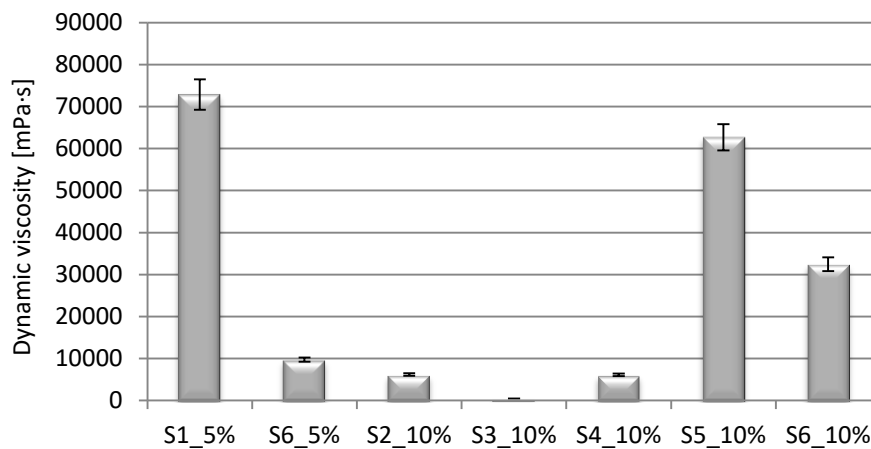


Fig. 1. Dynamic viscosity of cosmetic oils in gel form depending on the concentration and type of rheology modifier

Source: own study.

Based on the results of dynamic viscosity and stability studies, rheology modifiers were selected to produce oils in gel form characterized by viscosities in the lowest viscosity range (silica at 10wt.% concentration), intermediate viscosity range (microcrystalline wax at 10wt.% concentration) and high viscosity range (hydrogenated vegetable oil at 5wt.% concentration). The selected rheology modifiers will be further tested in Phase II of the experimental part to determine the effect of the type of vegetable oil on the properties of the oils in gel form.

Effect of the type of vegetable oil on the physicochemical and usable properties of cosmetic oils in gel form

The formulations developed (Tab.4) contain the following vegetable oils as cosmetic oil base substances: Helianthus Annuus (Sunflower) Seed Oil, Vitis Vinifera (Grape) Seed Oil, Prunus Amygdalus Dulcis (Sweet Almond) Oil. The formulations use rheology modifiers selected from Stage I: hydrogenated vegetable oil, silica, cera microcrystalline. In addition, vitamin E (Tocopheryl Acetate) was used in the formulation.

Tab. 4. Formulations of cosmetic oils in gel form depending on the type of vegetable oil used

INCI name	Concentration [wt.%]											
	HAS O	Rec_ S1	Rec_ S2	Rec_ S3	VVS O	Rec_ V1	Rec_ V2	Rec_ V3	PA- DO	Rec_ A1	Rec_ A2	Rec_ A3
Helianthus Annuus (Sunflower) Seed Oil	to 100				-	-	-	-	-	-	-	-
Vitis Vinifera (Grape) Seed Oil	-	-	-	-	to 100				-	-	-	-
Prunus Amygdalus Dulcis (Sweet Almond) Oil	-	-	-	-	-	-	-	-	to 100			
Hydrogenated Vegetable Oil	-	5.0	-	-	-	5.0	-	-	-	5.0	-	-
Silica	-	-	10.0	-	-	-	10.0	-	-	-	10.0	-
Cera Micro-crystallina	-	-		10.0	-	-		10.0	-	-		10.0
Tocopheryl Acetate	0.5											

Source: own study.

Tab. 5. Results of stability evaluation of cosmetic oils in gel form depending on the type of vegetable oil used

Week	Stability											
	HAS O	Rec_ S1	Rec_ S2	Rec_ S3	VVS O	Rec_ V1	Rec_ V2	Rec_ V3	PA- DO	Rec_ A1	Rec_ A2	Rec_ A3
1	+	+	+	+	+	+	+	+	+	+	+	+
2	+	+	+	+	+	+	+	+	+	+	+	+
3	+	+	+	+	+	+	+	+	+	+	+	+
4	+	+	+	+	+	+	+	+	+	+	+	+
5	+	+	+	+	+	+	+	+	+	+	+	+
6	+	+	+	+	+	+	+	+	+	+	+	+

Source: own study.

Table 5 shows the results of assessing the stability of oils in gel form based on Helianthus Annuus (Sunflower) Seed Oil, Vitis Vinifera (Grape) Seed Oil, Prunus Amygdalus Dulcis (Sweet Almond) Oil and with the selected rheology modifiers from stage I.

Summarizing the obtained results of the stability assessment, no adverse changes were found in the consistency, odor and homogeneity of the tested cosmetic oils. Photos of the developed and produced cosmetic oils are shown in Figure 2.

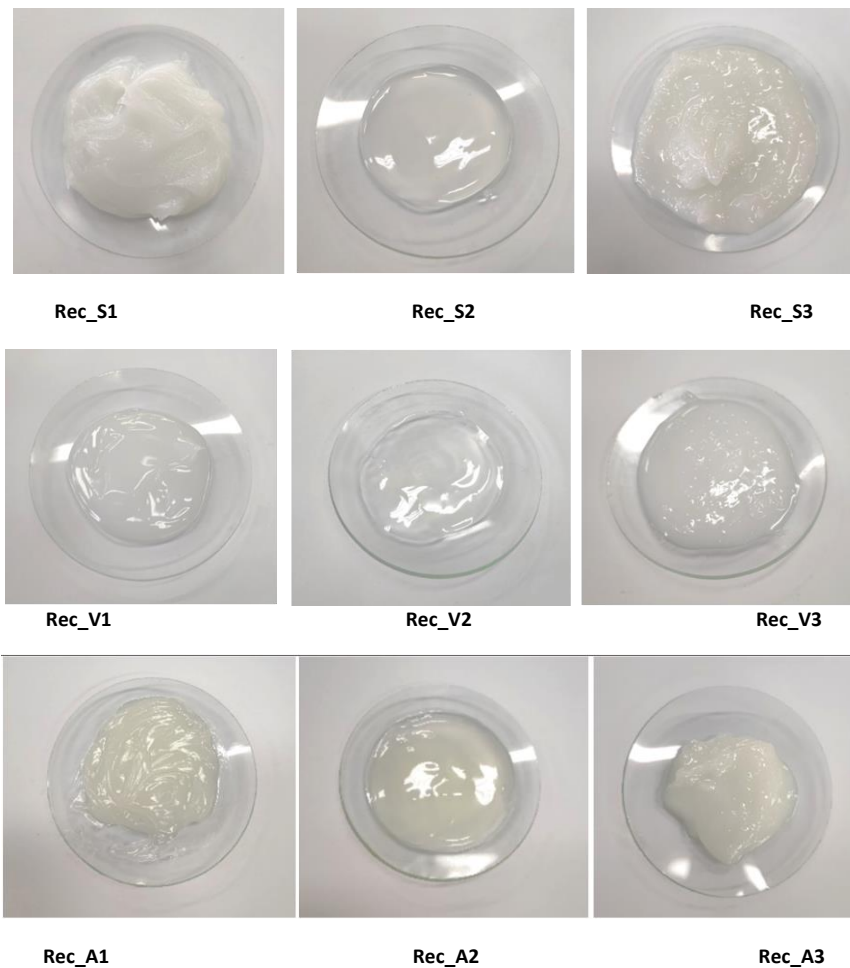


Fig. 2. Photos of developed and prepared cosmetic oils according to the formulations shown in Table 2

Source: own study.

Subsequently, the author's cosmetic oils were subjected to viscosity and yield point tests. The result of the study is shown in Figures 3 and 4.

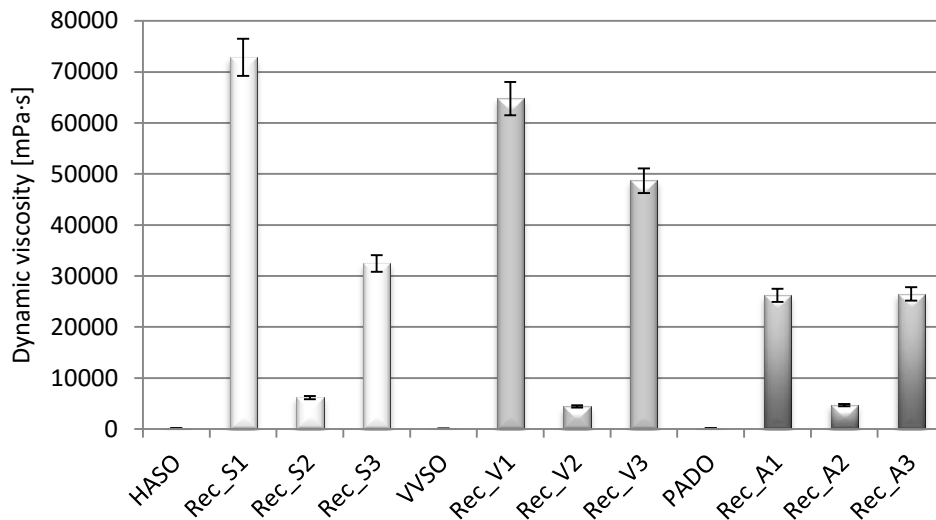


Fig. 3. Dynamic viscosity of cosmetic oils in gel form depending on the type of base vegetable oil
Source: own study.

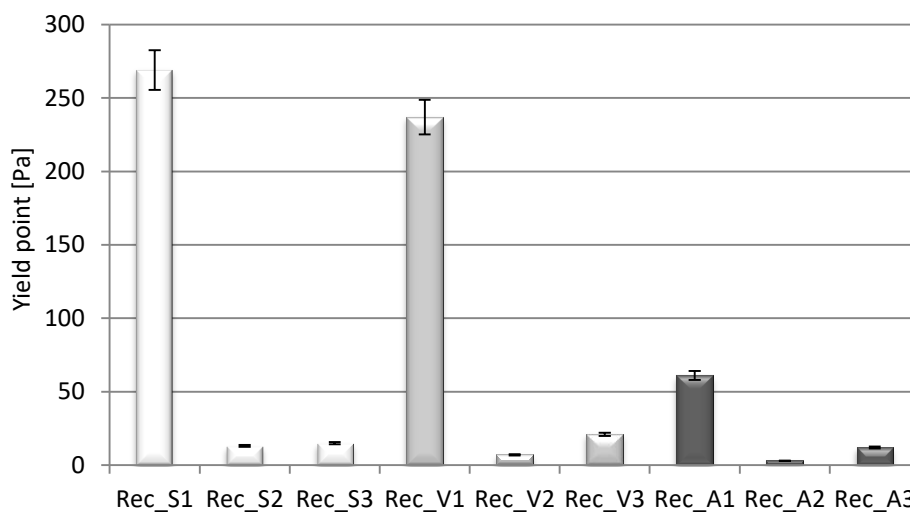


Fig. 4. Yield point of cosmetic oils in gel form depending on the type of base vegetable oil
Source: own study.

Based on the results obtained, it was found that the values of dynamic viscosity (η) of oils in gel form vary depending on the type of vegetable oil used. The lowest values were obtained for care oils based on *Prunus Amygdalus Dulcis* (Sweet Almond) Oil (A1, A2 and A3 series). For cosmetic oils based on *Helianthus Annuus* (Sunflower) Seed Oil and *Vitis Vinifera* (Grape) Seed Oil, viscosity results were obtained in similar ranges, from 6163 to 72867 mPa·s and from 4463 to 64767 mPa·s. The viscosity results of the base vegetable oils *Helianthus Annuus* Seed Oil (HASO), *Vitis Vinifera* Seed Oil (VVSO) and *Prunus Amygdalus Dulcis* Oil (PADO), which had low viscosities ranging from 140 to 210 mPa·s, were also presented to illustrate the obtained gel form. The authors Misiak et al. (2021) in their pa-

per on the production of oleogels based on natural oils also demonstrated the effect of the type of vegetable oil on the viscosity of oleogels. They found that oleogels based on hemp oil showed higher viscosity values compared to oleogels based on raspberry seed oil. In addition, they found that the viscosity of gels obtained with the addition of silica was significantly lower than those with the addition of a polymeric agent (Misiak et al., 2021).

The results obtained for the yield point (Fig. 4) of the tested cosmetic oils in gel form correlate significantly with the results of dynamic viscosity measurements (Fig. 3). Also in this case it was observed that the lowest values of the yield point were characterized by care oils based on the *Prunus Amygdalus Dulcis* (Sweet Almond) Oil (A1, A2 and A3 series). For cosmetic oils based on *Helianthus Annuus* (Sunflower) Seed Oil (HASO) and *Vitis Vinifera* (Grape) Seed Oil (VVSO), yield point results were obtained in similar ranges from 13 to 269 Pa and 7 to 237 Pa, respectively. The results of the yield point are important in selecting the optimal cosmetic packaging and dosage method. They also give information about the spreading efficiency of the product on the skin during use and the stability of the system. In the literature (Bekker et al., 2013; Kulawik-Pióro et al. 2019), it was shown that the product flow properties, established by rheological measurements, may be correlated with the empirically subjective assessment of skin feeling.

The final stage of the work was a consumer evaluation of the sensory appeal of the tested cosmetic oils in gel form. There are numerous scientific reports in the literature using sensory testing to evaluate cosmetic products, despite the possibility of using instrumental methods (Kulawik-Pióro et al. 2020; Kossowska & Zieliński, 2017). In their work, authors Fabbron-Appas et al. (2021) suggest that emollients are directly responsible for sensory properties such as spreading, absorption and touch on the skin. In addition, they can affect the viscosity and appearance of a skin care product (Fabbron-Appas, 2021). The results obtained are shown in Table 6.

Based on the results of the consumer evaluation of the sensory appeal of the tested cosmetic oils in three categories: evaluation of the product during application, sensation after application of the preparation, and evaluation of the product in the hand, it was shown that cosmetic oils in gel form containing hydrogenated vegetable oil as a rheology modifier received the highest total score (Rec_S1, Rec_V1, Rec_A1). According to the members of the sensory panel, the lowest scores were given to preparations based on *Vitis Vinifera* (Grape) Seed Oil and *Prunus Amygdalus Dulcis* (Sweet Almond) Oil, which contain cera microcrystalline as a rheology modifier (Rec_V3, Rec_A3).

Tab. 6. Consumer evaluation of sensory appeal of tested cosmetic oils in gel form

	Rec_S1	Rec_S2	Rec_S3	Rec_V1	Rec_V2	Rec_V3	Rec_A1	Rec_A2	Rec_A3
Evaluation during application, after 10 laps									
Spreading:	5/5	5/5	4/5	4/5	5/5	3/5	4/5	3/5	3/5
Absorption:	4/5	4/5	3/5	3/5	4/5	2/5	4/5	4/5	2/5
Stickiness:	3/5	3/5	2/5	3/5	3/5	2/5	3/5	3/5	2/5
Evaluation 30 minutes after application									
Smoothing:	5/5	3/5	4/5	5/5	2/5	3/5	5/5	3/5	4/5
Greasing:	3/5	3/5	2/5	3/5	4/5	1/5	4/5	4/5	2/5
Product evaluation in the hand									
Homogeneity:	5/5	5/5	3/5	5/5	5/5	3/5	5/5	5/5	3/5
Adhesion:	5/5	2/5	5/5	4/5	2/5	5/5	5/5	2/5	5/5
Consistency:	5/5	3/5	4/5	5/5	3/5	5/5	5/5	3/5	4/5
Total points	35/40	28/40	27/40	32/40	28/40	24/40	35/40	29/40	25/40

Source: own study.

Conclusions

In this study, an attempt was made to develop cosmetic oils as emollient preparations for atopic skin. The choice of this type of cosmetic was dictated by the essential content in the formulation of emollients, so important in the prophylaxis and support of AD treatment. In addition, an attempt was made to develop the formulations in such a way as to obtain a gel form of the hydrophobic system. To this end, a series of prototypes of care oils differing in the type and concentration of rheology modifiers and the type of base vegetable oils were prepared. Based on the results, it was found that cosmetic oils based on *Helianthus Annuus* (Sunflower) Seed Oil with 5wt.% rheology modifier: silica, aluminum stearate, zinc stearate and beeswax showed instability of the system. Rheology modifiers were selected for further study, which resulted in gel oils characterized by viscosities in the lowest range (silica at 10wt.% concentration), intermediate range (cera microcrystalline at 10wt.% concentration) and high viscosity range (hydrogenated vegetable oil at 5wt.% concentration). The effect of the type of base vegetable oil on the viscosity of cosmetic oils in gel form was then demonstrated. The viscosity results were in the following gradation: η - sunflower oil > η - grapeseed oil > η - almond oil. The results of testing the yield point of the tested cosmetic oils in gel form correlate with the results of measuring dynamic viscosity. Again, it was observed that yield point values decrease with the use of the following base oils in care oils: sunflower oil > grapeseed oil > almond oil. On the other hand, consumer evaluation of sensory appeal showed that the use of the rheology modifier hydro-

generated vegetable oil in cosmetic oil formulations improves the sensory properties of cosmetic oils. Thus, it can be concluded that the introduction of hydrogenated vegetable oil into the formulation of cosmetic oils at a concentration of 5wt.% has a significant impact on the functional and physicochemical characteristics of this type of products. The use of hydrogenated vegetable oil in the formulation of cosmetic oils allows the formation of a stable gel form of the hydrophobic system but also has a positive effect on the sensory evaluation of this type of products.

Acknowledgments

The work performed under the project no. 3501/188/P titled: "Application of innovative raw materials of natural and synthetic origin in care and support of treatment of skin diseases in various clinical conditions". Project financed by the Ministry of Education and Science.

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THE EFFECT OF SOLVENT TYPE ON THE USAGE PROPERTIES OF HAND CLEANERS

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DOI: 10.56091/CTQS.Qual-18

Abstract

The article evaluates the effect of the type of solvent on the performance properties of modern hand cleaners for heavily soiled. Formulations in the form of emulsions containing as hydrophobic solvents: strawberry seed oil, blackberry seed oil, methyl esters of fatty acids obtained on the basis of rapeseed oil were developed. On the basis of available literature data and own research, formulations were developed for 12 prototypes of hand cleaners containing 10, 12, 14, 16% by weight the above mentioned additives. The following were evaluated for the made products: stability, pH, viscosity, flow limit of the degree of skin hydration, transepidermal water loss (TEWL), dirt removal efficiency.

The obtained test results were related to those obtained for two commercial products. Application tests proved that the highest dirt-removing efficiency was characteristic of preparations containing in their composition rapeseed oil methyl esters in concentrations of 14 and 16% by weight. After washing off with water, they lubricated the skin, preventing it from drying out. On the other hand, an increase in the concentration of esters in the emulsions resulted in a decrease in the value of the yield stress, which had a beneficial effect on dispensing and spreading it on the skin surface. The addition of strawberry seed oils at concentrations of 14 and 16% by weight provided good removal efficiency relative to the commercial product, but caused a significant increase in viscosity values and yield stress relative to ester formulations and commercial products, resulting in poorer application properties. On the basis of the study, it can be concluded that the original formulations obtained were stable, had more favorable application properties relative to their market counterparts, and exhibited a skin care effect.

Keywords: handwashing agents, quality, solvents

Introduction

The high demands placed on handwashing agents inspire the search for new formulation solutions and challenge manufacturers of household chemicals. The oldest and best-known handwashing agent is traditional soap. And although its effectiveness has proven itself for thousands of years, the prevalence of its many unfavorable properties was the main reason for the appearance of synthetic detergents on the market. The irritating effect on the skin of some of them has led to a strong trend in recent years to reuse soaps as: emulsifiers, consistency regulators and additives to finished prod-

ucts such as laundry powders. This was mainly supported by: availability of raw materials, low price, ease of production, biodegradability (Białowąż et al., 2001; Dmytryshyn et al., 2004).

Products for cleaning the skin of the hands, even from difficult dirt, should be gentle preparations that do not penetrate into its deeper layers. Thus, they must not contain aggressive surfactants, which destroy the fat-lipid cover of the skin. This is a difficult task to achieve if we are dealing with the composition of formulations, intended for use in the public sector. Mild cleaning agents are not effective in contact with a range of contaminants of industrial origin, such as grease, fats, oils, paints, varnishes, soot. Therefore, it is necessary to strive for a compromise between cleaning effectiveness and gentle action on the skin. By using appropriate compositions of surfactants and combining them with additives that support the washing process, it is possible to obtain formulations that meet the expectations of even the most demanding groups of consumers. Commonly known and used additives are hydrophobic solvents. By introducing them into the formulation, we have two phases in the system: aqueous and fatty. In the presence of surfactants in such systems, emulsions will be formed (Sułek & Małyśa, 2010; Zieliński, 2021).

This paper presents attempts to develop formulations of emulsion pastes for washing heavily soiled hands based on oleic acid soaps, containing as solvents: strawberry seed oil, blackberry seed oil and methyl esters of fatty acids obtained from rapeseed oil. There are many arguments in favour of this form of preparation, among the most important are: the possibility of introducing a large number of surfactants, thereby increasing the efficiency of removing difficult dirt, and on the other hand, the large share of the oil phase, is designed to protect the skin from excessive drying. In addition, the proper selection of the emulsifying system, the main component of which is oleic soap, is designed to ensure the efficiency of dirt removal and the appropriate stability and consistency of the preparation. These are the most desirable properties of hand cleaning agents.

In the literature on the subject (Friis & Skagerlind, 2008; Hreczuch et al., 2000; Przondo 2007; Sułek & Klimaszewska, 2008; Sułek et al., 2008), agents for removing heavy soils from the skin of the hands are called: hand cleaners, hand cleansers, hand cleansing emulsions, hand cleansing pastes, hand-washing agents and these terms will be used interchangeably in the text of the article.

Materials and methods

Materials

The study material consisted of 12 original formulations in the form of hand wash emulsions containing as emulsifier and consistency ingredient INCI: Sodium Oleate at a concentration of 24% by weight, as auxiliary emulsifier ethoxylated with 7 moles of ethylene oxide lauryl alcohol – INCI: Laureth-7,

at a concentration of 7% by weight. The formulas also used hydrophobic solvents in concentrations of 10, 12, 14, 16% by weight: rapeseed oil fatty acid methyl esters (formulas sequentially 1–4), raspberry seed oil (formulas sequentially 5–8) and blackberry seed oil (formulas sequentially 9–12). Both raspberry seed oil and blackberry seed oil were obtained by supercritical carbon dioxide extraction (Rój et al., 2009).

A summary of the formulations of the original cleaning agents are shown in Table 1.

Tab. 1. Recipes of original hand cleaners

Formulation Ingredients (INCI)	% by weight											
	1	2	3	4	5	6	7	8	9	10	11	12
Sodium Oleate	24											
Rapeseed Fatty Acid Methyl Esters	10	12	14	16								
Strawberry Seed Oil					10	12	14	16				
Blackberry seed oil									10	12	14	16
Laureth-7	7											
Aqua	up to 100											
Preservative	0.2											

Source: own research.

Two commercial products that contained hydrophobic solvents in the formulation were used to compare performance with the original formulations, designated as TP1 and TP2. The price of commercial products was in the range of 70–90 PLN per liter.

Methods

Stability

Visual evaluation of emulsions consisted of observing their appearance, homogeneity and signs of instability, such as stratification, creaminess and coalescence, among others. It also allowed observation of the appearance of mold or other signs of microbial contamination present in the preparation. Visual evaluation of the prepared emulsions was carried out immediately after making them, and after 1 and 7 days (Marcinkowska et al., 2006).

Load temperature tests allowed visual evaluation of the stability of preparations stored alternately at elevated (40°C, 1 day) and reduced (5°C, 1 day) temperatures. They were carried out in an ST-68 type hothouse and an Amica refrigerator. The test lasted 8 days (4 full cycles) (Sułek & Klimaszewska, 2008).

pH

The pH was measured using a CP-411 waterproof pH meter, which has a combination pH-metric electrode. The test was conducted at room temperature. Performing the measurement involves immersing the electrode in the test sample. Measurements, of each preparation were made three times, the results in the table are the arithmetic mean of the measurements (Tomaszkiewicz- Potępa & Mitoraj, 2007).

Viscosity

Dynamic viscosity coefficient measurements were carried out using a Brookfield viscosity meter type HA DV III Ultra. Measurements were performed at 21°C at a spindle speed of 5 rpm using Helipath type spindles (Klimaszewska et al., 2017).

Yield stress

The values of the yield stress of the tested cosmetic masks were determined using a Brookfield HA DV III Ultra viscosity meter equipped with a set of vane spindles. Measurements were carried out at constant spindle speeds of 0.1, 0.5, 1 and 5 rpm. The flow limit was the minimum value of shear stress above which body flow occurred. EZ-Yield Software was used to record the measurements and analyze them (Klimaszewska et. al., 2016).

Degree of skin moisturization

The degree of skin moisturization was measured using a Corneometer type CM 825. The electrical capacity of the skin, which depends on the water content of the epidermis, was measured. The scale of the instrument ranged from 1–120 a.u. The measurement was carried out at 20°C with 50% humidity. Measure area of 4 × 4 cm was marked on the surface of the forearm. Then 0.5 g of each emulsion was applied, spread with a metal spatula and after 10 min the preparation was removed with a cotton pad. Skin moisturization tests were performed with a suitable probe before application (control test) and 2 h after application (Klimaszewska et al., 2016).

Transepidermal water loss

Transepidermal water loss was examined using a Tewameter TM 300. Tests were performed at designated locations on the skin of the forearm before application of the product samples, 10 minute after application, and 2 hours after application of the samples to the skin. Three measurements were made for each sample so the final result was the arithmetic average of three independent measurement series (Klimaszewska et al., 2016).

Dirt removal efficiency

The dirt removal efficiency of the tested formulations was evaluated by the ability of the formulation to remove model dirt from the skin of the hands in the form of semi-synthetic motor oil and universal lithium plastic grease. For this purpose, about 2 grams of dirt was applied to the hands, spread and left for two minutes, then the cleaning product was applied in the amount of 4 grams spread in the hands then rinsed with tap water. The degree of dirt removal was visually assessed on a two-point scale, where 0 points means that the product did not remove the dirt, 1 point – the product removed the dirt partially, 2 points – the product removed the dirt completely (Sulek & Matysa, 2010).

Result and discussion

Stability

Evaluating the stability over time of an emulsion is the most important quality parameter during storage and use. This stability refers to the ability of an emulsion to maintain a homogeneous combination of aqueous and oil phases for a specified period of time without either phase precipitating, separating, or changing its appearance or consistency.

The results of stress tests (under elevated and reduced temperature) showed full stability of all tested compositions. No signs of microbiological and physicochemical instability were observed, and it was found that all handwashing prototypes were characterized by satisfactory stability over time.

pH

Measuring the pH of hand sanitizers is an extremely important aspect in their quality control. Too high pH values of the preparation could violate the hydrolipidic shield of the skin and irritate it. Measured pH values for all tested samples, were in the range of 7–8, so the pH of the preparations was equalized with a 3% citric acid solution to a value of about 5.5.

Viscosity

Viscosity (η) of hand cleaners is a very important parameter from the point of view of their performance, especially dosage and application to the skin surface. Viscosity is derived from its composition, mainly viscosity regulators, as well as components that perform other functions in the product, such as hydrophobic solvents. The effect of the type of hydrophobic solvent on the dynamic viscosity of the original and commercial products was evaluated. The results of η measurements are shown in Figure 1.

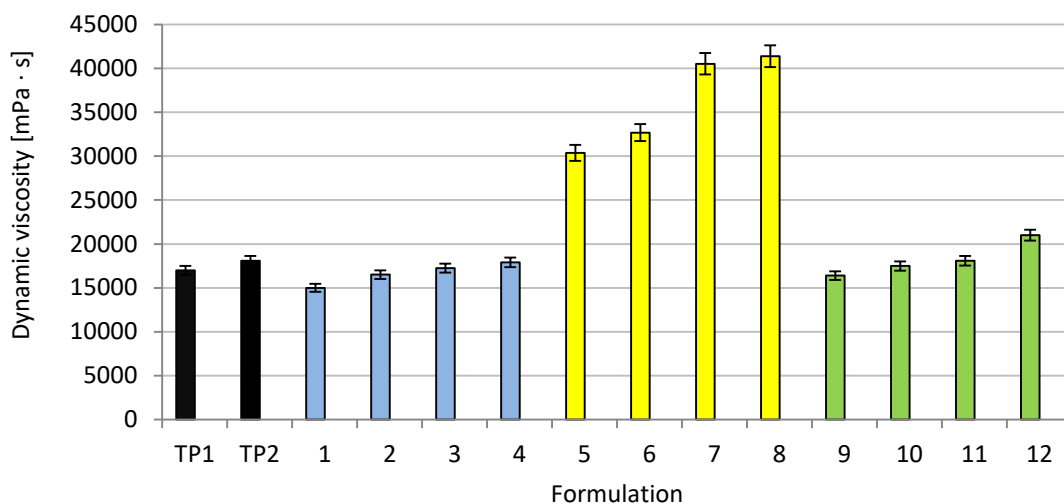


Fig. 1. Dynamic viscosity of trade products (TP1, TP2) and original formulations (1–12)

Source: own research.

The effects of fatty acid methyl esters, strawberry seed oil and blackberry seed oil on the dynamic viscosity of the original hand wash products were studied. Two commercial products, TP1 and TP2, were used as reference points in the evaluation. For the commercial products, the measured viscosity values oscillated in the range of 17000 –18000 mPa·s. For the obtained washing agents containing fatty acid methyl esters from rapeseed oil and blackberry seed oil in concentrations of 12–16 wt% in the formulation, viscosity values similar to their market counterparts were obtained. On the other hand, undoubtedly, for formulations containing strawberry seed oil as a hydrophobic solvent, the viscosity values were much higher in the range of about 30 000 to 41 000 mPa·s. The obtained relatively high viscosity values of formulations with strawberry seed oil may hinder the spreading of the agent on the hand surface during washing. Therefore, the next step was to test the yield stress.

Yield stress

The yield stress has a major impact on the application properties of the product. Too high a value of the flow limit makes it difficult to spread the product on the skin, while too low a value causes the product to run off. The yield stress is a parameter that determines the application properties of hand cleaners and their dosage from the package. The results of the study are shown in Figure 2.

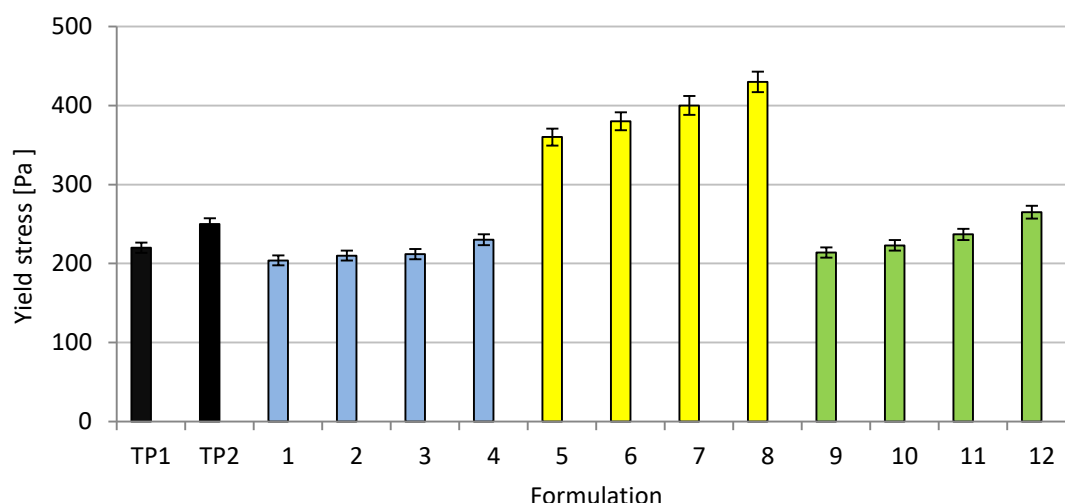


Fig. 2. Yield stress of trade products (TP1, TP2) and original formulations (1–12)

Source: own research.

The yield stress tests carried out closely correlate with viscosity tests. Similarly, hand wash products containing rapeseed oil methyl esters and blackberry seed oil as hydrophobic solvents showed yield stress values close to those of commercial products, i.e. from 200 to 265 Pa. In contrast, in the case of washing agents containing strawberry seed oil, much higher yield stress values close to 360–430 Pa were achieved. The results presented in the study may indicate a more difficult application of the product to the hands from the package and worse distribution of the product on the skin surface during washing.

Skin moisturization

Hand cleaners, especially those designed to remove heavy dirt, dry out the skin after cleaning, compromising its hydrolipidic film. Therefore, an important test is to assess the degree of skin moisturization after washing. The results of the research are illustrated in Figure 3.

Tests of the degree of moisturization of the skin after washing were carried out 10 minutes and 2 hours after the application of original preparations and commercial products. The effect of applied hydrophobic solvents on the studied parameter was evaluated. The reference point in the evaluation was the so-called "control point," i.e. the area on the skin to which no product was applied. The study showed that the skin was most dried out by commercial formulations. The difference in skin moisturization values from 10 minutes to 2 hours after washing off the product was about 20 units. For all formulations developed, the differences in skin hydration at 10 minutes and at 2 hours after washing off the product differed by a few units. This demonstrates the ability to keep a high level of skin moisturization after washing and the skin care properties.

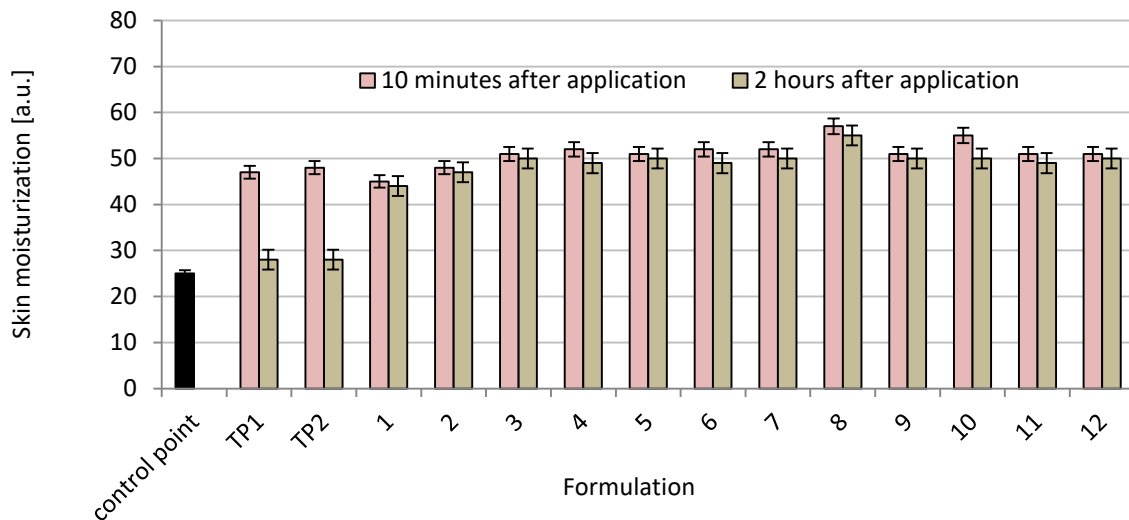


Fig. 3. Skin moisturization of trade products (TP1, TP2) and original formulations (1–12)

Source: own research.

Transepidermal water loss

Transepidermal water loss (TEWL) is a complementary test to skin moisturization. The higher the values of this parameter after washing, the more dried the skin and the use of additional skin care products is required to replenish naturally occurring lipids in the skin. TEWL measurements are shown in Figure 4.

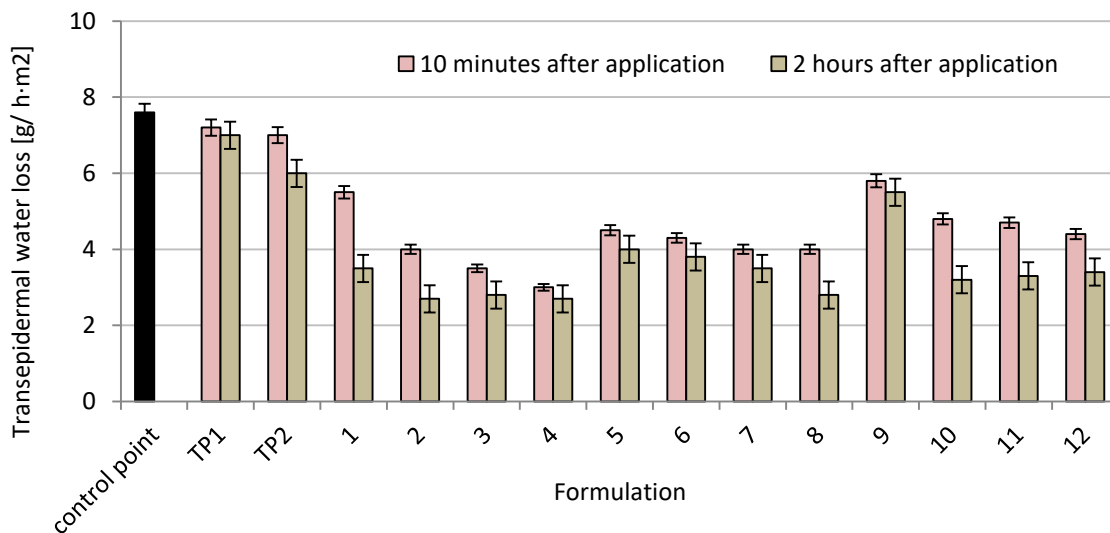


Fig. 4. Transepidermal water loss after use of trade products (TP1, TP2) and original formulations (1–12)

Source: own research.

The TEWL tests performed correlate well with skin hydration results. The highest TEWL values (6–7.6 g/h·m²) were recorded for the control point (skin before application) and for market products.

Subsequently, TEWL values decreased for formulations containing blackberry seed oil, strawberry seed oil, and rapeseed oil methyl esters, respectively. It is noteworthy that the lowest TEWL values of formulation 4 $-2.7 \text{ g/h}\cdot\text{m}^2$ were achieved when esters were used, which indicates the ability of these products to retain water in the epidermis. It can also be noted that an increase in the concentration of hydrophobic solvents affects the reduction of TEWL values 10 minutes after washing. It is also very significant that in the time interval from 10 minutes to 2 hours after washing, the difference in TEWL values is small.

Dirt removal efficiency

A high efficiency in removing dirt is the most important feature of modern cleaning agents. Cleaning is an indispensable treatment in keeping proper hygiene. It prevents microorganism extension. The basic purpose of cleaning is mechanical removal of dirt (usually with complex chemical structure), e.g. grease, engine oil. During the cleaning process, solvents contained in the cleaning agents penetrate the fatty soils. The effect of the type and concentration of hydrophobic solvents on the removal efficiency of two types of dirt: engine oil and universal lithium grease was evaluated. The formulations were evaluated on a three-point scale, where 0 points meant no removal of the soiling, 1 point – partial removal of the soiling and 2 points – complete removal of the soiling. The results of the tests are shown in Figure 5.

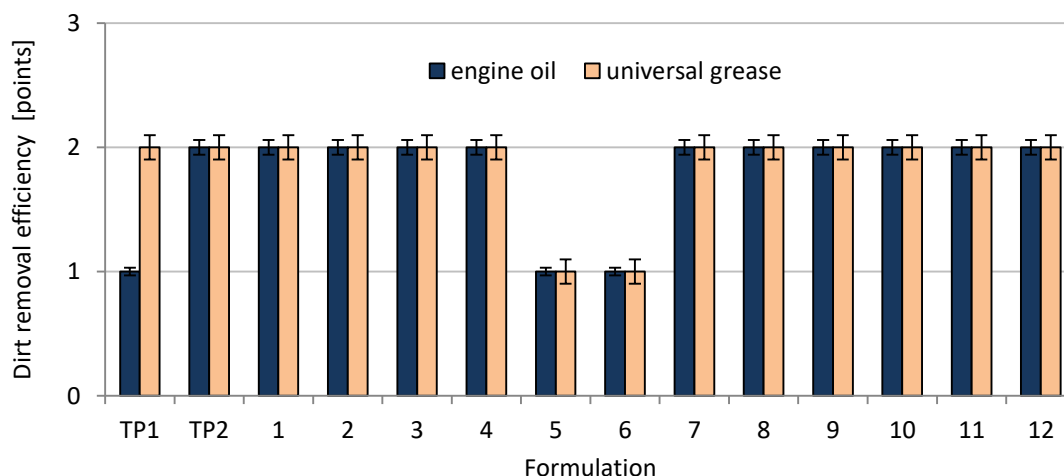


Fig. 5. Dirt removal efficiency after use of trade products (TP1, TP2) and original formulations (1–12)
Source: own research.

The obtained data showed that formulations containing rapeseed oil methyl esters (formulations 1–4), as well as containing blackberry seed oil (formulations 9–12), showed the best ability to remove engine oil and universal grease. The commercial formulation removed the soiling from the engine oil

only partially, while formulations containing blackberry seed oil at concentrations of 10 and 12 by weight (formulations 5, 6) only partially removed both types of soiling.

Conclusions

The result of the research was the development of 12 formulations of original agents for removing heavy soils from hand surfaces. Stable formulations were obtained, characterized by functional properties comparable to their commercial counterparts. It was shown that prototypes containing rapeseed oil methyl esters and blackberry seed oil exhibited the desired viscosity and yield stress to ensure adequate application properties.

It has also been shown that the developed products do not dry out the skin and maintain reduced TEWL for up to 2 hours after washing off with water relative to the control point and commercial products. Cleaning emulsions with rapeseed oil methyl esters and blackberry oil were the most effective in removing dirt. However, in the case of formulations with strawberry seed oil, too high viscosity and flow limit disqualified these samples due to inferior application properties.

The developed formulations of prototypes of emulsion-type heavy-duty soils removers and the evaluation of their properties can provide a valuable compendium of knowledge for practitioners. It can also provide a rationale for further research into the use of various hydrophobic solvents in such products.

Acknowledgments

The article has been prepared under Project no. 3086/35/P “Development of formulations and technologies for the manufacture of innovative cosmetics, pharmacy supplies, household and industrial chemicals” financed by University of Radom.

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QUALITY EVALUATION OF FACIAL CLEANSING GELS WITH SURFACTIN-RICH DIGESTATE SOLVENT EXTRACT. PART I – FUNCTIONALITY

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DOI: 10.56091/CTQS.Qual-19

Abstract

The results of a study on the quality assessment of prototype facial cleansing gels prepared from developed original recipes are presented. Raw materials of natural origin were used in gels preparation, and the concentration of solvent digestate extract was a variable in the formulation. The extract, according to the manufacturer's declaration, was rich in surfactin and was used in this study at concentrations of 20; 40 and 80% by weight. In this part of the work, studies for the obtained gels prototypes were performed to evaluate their functionality. Stability, viscosity, foaming properties, detergency properties and color were analyzed. The results were compared with those obtained for the reference sample, which was a gel prototype that did not contain the digestate extract.

All prototypes of face cleansing gels showed physicochemical stability. The introduction of digestate extract resulted in a decrease in viscosity values compared to the reference composition. However, the obtained values did not exclude the use the evaluated cosmetics as face cleansing gels. In the case of assessing the foaming properties of the product prototypes, it was shown that the value of foaming ability and foam stability index decreased with an increase in the concentration of biotechnology-derived raw material in the sample. Colorimetric evaluation indicated that the extract introduced into the gels affected the change in their color, and the change might be noticeable by the consumer. The obtained values of hydrophobic dirt removal efficiency after contact with the gels' solutions indicated that the introduction of the extract in the gels' prototypes improved their detergent properties compared to the reference sample.

The performed research showed that it is possible to obtain facial cleansing gels with a significant amount of the digestate extract providing satisfactory performance in terms of functionality.

Keywords: cosmetics, extract, functionality, surfactin, quality, facial cleansing gels

Introduction

Facial cleansing gels are cosmetics, with their main function to help clean the facial skin. Their specific application enforces the appropriate selection of the composition of these products to remove various types of dirt. On the face surface there may be dirt naturally formed on the skin (sebum,

sweat, dust), as well as makeup residues. The composition of such cosmetics must, therefore, be selected to ensure the proper functionality of the product in terms of cleansing action, while at the same time maintaining the other characteristics of the product required by consumers, including stability, rheological properties or foaming properties. In terms of composition, these cosmetics are usually aqueous solutions of anionic surfactants, enhanced with compounds from the group of non-ionic and amphoteric surfactants and various additives such as humectants, preservatives, dyes, plant extracts or pH regulators. The viscosity of the product is regulated by the use of polymeric rheology modifiers and, less frequently, by the introduction of sodium chloride (Bujak, et al. 2018).

The European cosmetics market, estimated at 76.7 billion euros at retail prices in 2020, is still growing and is the world's largest market for cosmetic products (Nowak et al., 2022). Development trends in the area of the cosmetics industry are mainly related to the increasing interest of consumers in cosmetics based on natural raw materials and coincide with the implementation of sustainability policy goals by manufacturers to an increasing extent (Amberg & Fogarassy, 2019; Bom et al., 2019; Kolling et al., 2022). Natural cosmetics and their dedicated raw materials are increasingly an object of scientific research (Majchrzak et al., 2022; Goyal & Jerold, 2021; Savic et al., 2021; Dini & Laneri, 2021). Therefore, cosmetics-related work is focusing on the use of raw materials of plant origin, including plant extracts (Savic et al., 2021), surfactants of natural origin (Seweryn & Bujak, 2018; Klimaszewska et al., 2022) and materials obtained through biotechnological processes (Bezerra et al., 2018). Studies on final cosmetic formulations based on raw materials of natural origin are also emerging.

One of the more interesting trends in the development of cosmetics is the use of plant raw materials and ingredients obtained through biotechnological processes, including fermentation of plant material (Majchrzak et al., 2022; Ziemlewska et al., 2021). Bioferments are ingredients extracted, typically from plant material, through a fermentation process using appropriate strains of microorganisms. The fermented material often constitutes waste from other industrial processes, such as the food industry. Suitably selected bacterial strains and process parameters allow the production of beneficial compounds for cosmetics, such as proteins, ceramides, amino acids and antioxidants. Ultimately, products containing such raw materials show increased biological effectiveness and bioavailability (Domżał-Kędzia et al., 2019; Majchrzak et al., 2022; Mathur et al., 2020;). A number of properties of bioferments of interest in terms of cosmetic applications have been identified in the literature. They can exhibit antiaging (Bae et al., 2012), antioxidant (Lin et al., 2022), moisturizing (Ziemlewska et al., 2021) and antirritant (Domżał-Kędzia et al., 2019) properties. An interesting group of raw materials obtained by biotechnological processes in terms of use in cosmetics are biosurfactants (Bjerk et al., 2021; Jahan et al., 2020). Besides their ability to reduce interfacial tension, they also exhibit antioxi-

dant activity (Kumar, 2021) and improve the bacterial microflora of the skin (Adu et al., 2020). Particularly high promises are pinned on the use of surfactin in cosmetics. This compound is highly effective in lowering interfacial tension and has low critical micellar concentration (Ganesan & Rangarajan 2021). Previous work revealed that it exhibits reduced irritation and low toxicity to the skin compared to synthetic surfactants (Fei et al. 2020). The antimicrobial effect of surfactin has also been proven (Chen et al., 2022).

In this work, a surfactin-rich ferment extract is proposed for use in facial cleansing gels. The use of fermentation extracts contemplates a mixture of ingredients, particularly important from a cosmetic point of view. The composition of such raw material can exhibit preservative, antioxidant or anti-wrinkle effects in the cosmetic systems (Majchrzak et al., 2022; Lee et al., 2020). These properties are derived from appropriately selected microorganisms and parameters of the fermentation process. The use of *Bacillus subtilis* bacteria in the fermentation process offers the possibility of obtaining raw materials with collagen (Hsu & Chiang, 2009) and hyaluronic acid synthesis-stimulating effects (Jia et al. 2013). In addition, studies were presented in which this bacterium may exhibit the ability to synthesize biopolymers, the levan (Domżał-Kędzia et al., 2023), and biosurfactants, such as surfactin (Lewińska et al., 2023). Levan has been confirmed for its beneficial cosmetic properties, including antioxidant (Bouallegue et al., 2020) and skin moisturizing (Lewińska et al., 2021). The use of post-ferment extracts as a fully natural, unprocessed by chemical synthesis source of skin care and anti-irritant ingredients along with surfactants (biosurfactants) can fulfill the policy demands of sustainable development in the cosmetics industry.

The purpose of this study was to evaluate the application potential of surfactin-rich postferment extract in facial cleansing gels. The digestate extract was obtained by fermentation with the *Bacillus subtilis* bacterial strain. Through the appropriate selection of the fermentation process conditions, the obtained raw material contained a high level of surfactin. Mineral salts, proteins or fatty acids were also present in the extract composition. A detailed description of the extract preparation and an analysis confirming the presence of significant amounts of surfactin in the obtained material were presented in another work of the Authors (Seweryn et al., 2023). For the purpose of this work, formulations of face cleansing gels, with variable concentrations of the surfactin-rich extract, were developed, and based on them, prototypes of cosmetics were prepared. As a reference sample in this study, a face cleansing gels prototype without the extract in its composition was applied. Compounds approved for use in natural cosmetics were selected to create prototype gels. For the developed products, studies to assess their quality related to functionality (stability, rheological properties, foaming properties, detergent properties and colorimetric evaluation) were carried out.

Materials and methods

Raw materials

Materials approved for use in natural cosmetics in accordance with COSMOS standards were used to produce prototype face cleansing gels. The list of ingredients used is shown in Table 1.

Tab. 1. The list of ingredients used to prepare prototype face cleansing gels

Name According to INCI*	Trade name	Supplier
Sodium Coco Sulfate	SULFOPON® 1216	BASF
Cocamidopropyl Betaine	Dehyton K	BASF
Coco Glucoside	Plantacare 818	BASF
<i>Bacillus subtilis</i> Ferment Extract, Propanediol, Pentylene Glycol, Surfactin	EPS3	InventionBio
Glycerin	Organic Glycerin	Laboratoires Prod'Hyg
Sodium Benzoate and Potassium Sorbate	KEM BS	AKEMA
Xanthan Gum	Cosphaderm X34	Cosphatec GmbH
Lactic Acid	Lactic Acid	WarChem

* International Nomenclature of Cosmetic Ingredients

Source: own study.

Facial cleansing gels formulations and manufacturing

Model formulations of facial cleansing gels differing in the content of surfactin-rich digestate extract were developed (Table 2).

Tab. 2. Facial cleansing gels formulations

Name According to INCI	Concentration [% w/w]			
	FG_0	FG_1	FG_2	FG_3
Sodium Coco Sulfate	4.00			
Cocamidopropyl Betaine	2.00			
Coco Glucoside	0.50			
Glycerin	1.50			
<i>Bacillus subtilis</i> Ferment Extract, Propanediol, Surfactin	0.00	20.00	40.00	80.00
Xanthan Gum	0.25			
Sodium Benzoate and Potassium Sorbate	0.50			
Lactic Acid	To pH 5.5			
Aqua	To 100			

Source: own study.

In the process of manufacturing cosmetic prototypes, a homogenizing mixer type MZUTL 5 (Urliński, Poland) with a capacity of 5 L was used. In the first step, Sodium Coco Sulfate was dissolved in water at 95°C. Then, after it was completely dissolved, the next components were added with continuous stirring (50 rpm) in the order given in the formulation (up to and including glycerin). The system was stirred until a clear solution was obtained. In the next step, after lowering the temperature of the system to about 40°C, the digestate extract and preservative were introduced. In the next step, Xanthan Gum was added by introducing it through dispensing on the surface of the stirring mixture. The solution was stirred until a clear, homogeneous product was obtained. Then, the pH was adjusted to a value of 5.5. The finished cosmetic samples were left for 24 hours at room temperature until the system was completely vented, and then they were subjected to further studies.

Stability

Stability was evaluated under laboratory storage conditions. Additionally, the stability of the cosmetics was evaluated using a mechanical loading test. The exact methodology of the study was described in the work of Seweryn et al. (2023). In addition, the turbidity of the cosmetic prototype samples was measured. A detailed description of the methodology was presented in the works of Wasilewski et al. (2018).

Viscosity

The dependence of viscosity on shear rate for prototype cleansing products was determined by testing according to own methodology. More details on the testing method are available in Wasilewski's work (Wasilewski et al., 2018). The study presents dynamic viscosity values at shear rates of 1 s^{-1} and 50 s^{-1} .

Foaming properties

Foaming properties were evaluated on the basis of own methodology, based on the standard: EN 12728: 2001. A detailed description of the research method is included in Wasilewski's and Seweryn works (Seweryn & Bujak, 2018; Wasilewski et al., 2018).

Evaluation of detergent properties

The detergent properties were evaluated based on the methodology described in the US patent No. 4904359 (Pancheri et al., 1990). The methodology is described by Wasilewski in detail in his work (Wasilewski et al., 2022).

Determination of the color parameters

Cosmetic samples with post-ferment extracts were tested at room temperature, 48 h after their preparation. The exact methodology of the study was presented in work of Wasilewski et al. (2022).

Statistical analysis

For turbidity, detergent properties, foaming ability, foam stability and viscosity data were reported as a mean \pm standard deviation (SD) with 3 replicates for each sample (n=3). For comparing the statistical significance of values, ANOVA and a Tukey HSD post hoc test were applied. Differences were considered significant with the p-value was <0.05 . The Statistica ver. 10 software (StatSoft, Tulsa, OK, USA) was used for calculations.

Results and discussion

Stability

The use of raw materials of plant origin in cosmetics declared as natural, risks the appearance of symptoms of instability during storage. This is because both raw materials of this type and, consequently, finished products made from such materials are susceptible to various aging processes. Symptoms of changes occurring in the cosmetic may appear as turbidity (Fonseca-Santos et al., 2015). Neither in the visual assessment nor in the loading test performed for the developed facial gels was observed symptoms of instability occurring in the form of solution turbidity or precipitation of solid fragments. The samples appeared to be fully stable. Moreover, in order to confirm long-term stability, two series of turbidity measurements were carried out after a period of one month. The obtained results are shown in Figure 1.

The turbidity values obtained for the prototype gel samples tested were in the range of values 22 – 37 NTU. Prototypes containing the digestate extract show higher turbidity compared to the reference sample FG_0. The effect of extract concentration on the parameter under study is noticeable. The highest turbidity was detected for the gel containing the highest analyzed concentration of surfactin digestate extract (FG_3). The obtained relatively low values of the evaluated parameter indicated the full clarity of cosmetics. Comparable values were obtained for the corresponding samples after a one-month storage period. This may prove the stability of the tested cosmetic prototypes. The maintenance of the transparency of the prototypes over time indicates the stability of the systems. No precipitation or sedimentation occurred in the samples, which could translate into an increase in the turbidity of the cosmetics. Obtaining a trend toward a slight decrease in turbidity values

after a month of storage of the gels compared to the values obtained after 24 hours could result from complete venting of the system.

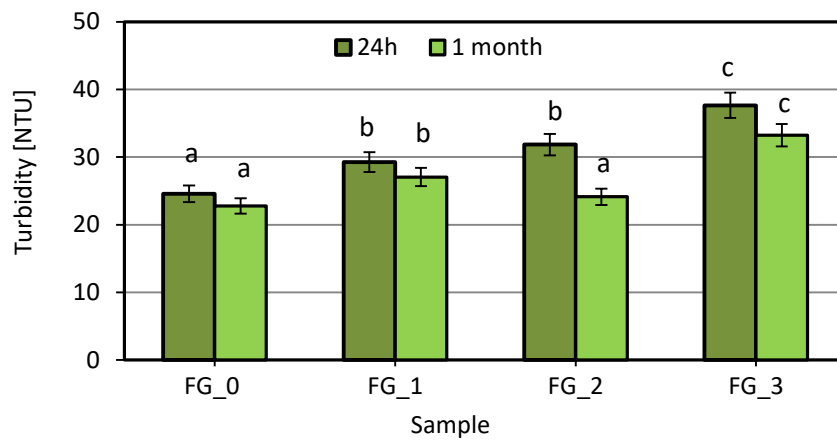


Fig. 1. Turbidity of the facial cleansing gels after 24 hours and one month after preparation
The superscripts a, b and c above the graph column indicate significant ($p < 0.05$) differences between sample FG_0 and samples FG_1, FG_2, FG_3 in terms of turbidity
Source: own study.

Viscosity

The basic physicochemical parameter related to the evaluation of the functionality of cleansing cosmetics is viscosity. This parameter provides information on the consistency of the product, which is an important indicator of the quality of cosmetics assessed by consumers. For the group of products discussed in this work, the term "gel" does not refer to their physicochemical form, but to the product as it is offered to the consumer. Obtaining the appropriate gel form is achieved through the use of appropriate viscosity modifiers and results from the composition of the product (Gallegos & Franco, 1999; Wasilewski et al., 2018). In the case of the product prototypes studied, appropriate viscosity values were required, reflecting a variety of important properties, such as easiness of dispensing the product from the package, distribution on the skin surface, and its mixing with water during the cleansing process. Determination of viscosity values enables to predict the behavior of a product during the consumer evaluation performed during the decision to buy a product (Chiari et al., 2012; Davies & Amin, 2020). Table 3 shows viscosity values obtained for prototype face cleansing gels at specified shear rates of 1 s^{-1} and 50 s^{-1} . The viscosity determination at low shear rate values allows to predict the product's behavior during dispensing from the package. At higher shear rate values, information is obtained about the behavior of the product during squeezing or shaking it out of the package (Wasilewski et al., 2018).

Tab. 3. Viscosity values of prototypes facial cleansing gels at shear rates of 1 s^{-1} and 50 s^{-1}

Sample identification	Dynamic viscosity value [mPa·s]			
	1 s^{-1}	SD	50 s^{-1}	SD
FG_0	9660	+/-110	1650	+/-50
FG_1	3670	+/-70	370	+/-20
FG_2	1520	+/-50	370	+/-20
FG_3	530	+/-30	180	+/-10

Source: own study.

For both shear rates analyzed, a rapid decrease in dynamic viscosity (530 mPa·s at 1 s^{-1} and 180 mPa·s at 50 s^{-1} for FG_3) was observed as the concentration of the digestate extract increased in the prototype face cleansing gels. The viscosity decreases observed in the prototype products may be related to the appearance of solvents (propylene glycol and pentylene glycol) in the system, which are included in the extract, as well as other compounds in their composition (including mineral salts). The analyzed cosmetic prototypes constitute an aqueous solution of surfactants. Solvents introduced into the system modify the bulk phase structure of the surfactant solution. Modification of the structure of the bulk phase affects the obtained viscosity values. The resulting effects were so strong that the cosmetic prototypes showed decreases in the viscosity parameter with increasing extract concentration, despite the use of a polymeric viscosity modifier. In addition, a decrease in dynamic viscosity values was observed with an increase in shear rate. This indicates the susceptibility of cosmetics to thinning under the influence of the imparted force. Such a feature is advantageous in terms of application of the cosmetic from the package and its distribution on the skin. The decrease in viscosity of the gel under the influence of the imparted force is expected in the situation of spreading on the surface of the facial skin, as it allows the cosmetic to be easily applied during the cleansing process. Moreover, facial cleansing gels are usually packaged in bags in which the application of the cosmetic is carried out by applying the appropriate hand pressure on the package. The proper decrease in viscosity under applying pressure allows the gel to flow freely out of the package.

Foaming Properties

An important indicator of the quality of cleaning cosmetics, including face cleansing gels, is their foaming properties. Consumers wrongly identify a foaming ability with the effectiveness of the product, while in fact this parameter has no effect on the product's performance or washing properties. It is required for hygiene products to generate a dense and velvety foam during washing. However, the ability to generate foam and its stability should be at a level that enables easy removal of the product

from the skin. An evaluation of the foaming properties of the produced prototypes of facial cleansing gels was carried out. The obtained results of foaming ability and foam stability are shown in Figure 2.

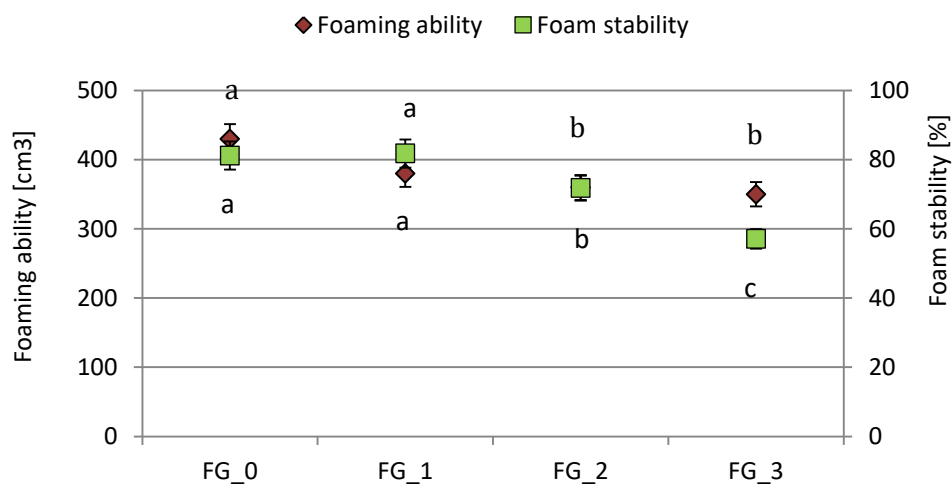


Fig. 2. Foaming ability and foam stability of prototypes facial cleansing gels

The superscripts a, b and c above and below to points on the graph indicate significant ($p < 0.05$) differences between sample FG_0 and samples FG_1, FG_2, FG_3 for foaming ability and foam stability

Source: own study.

The developed cosmetic prototypes generated foam at the value of 350 – 430 cm³. Compared to the reference composition FG_0, a decrease in the evaluated parameter was observed with increasing concentration of the extract added to the composition. The lowest foaming capacity was observed for the prototype cosmetic FG_3 with 80% (w/w) of the extract. A similar correlation was determined when evaluating the stability of the foam formed, though the greatest decrease in foam stability (up to 57%) was observed for the FG_3 composition. The differences in the evaluated parameters compared to the FG_0 prototype are probably due to the complex composition of the digestate extract. Despite the supply of an additional surfactant (surfactin) to the system, a deterioration of the evaluated properties was noted. Surfactin is known for its good foaming properties (Fei et al. 2020) and it was expected that the introduction of an extract with this compound would result in an improvement in the foaming properties of the cosmetic. However, other ingredients present in the extract besides surfactin, such as mineral salts, hydrophobic components or hydrophilic solvents (propylene glycol and pentylene glycol), may negatively affect the cosmetic's foam ability and its stability (Petkova et al., 2020; Hill & Eastoe, 2017).

Evaluation of detergent properties

The results of the evaluation of the detergent properties of the face cleansing gels are shown in Figure 3.

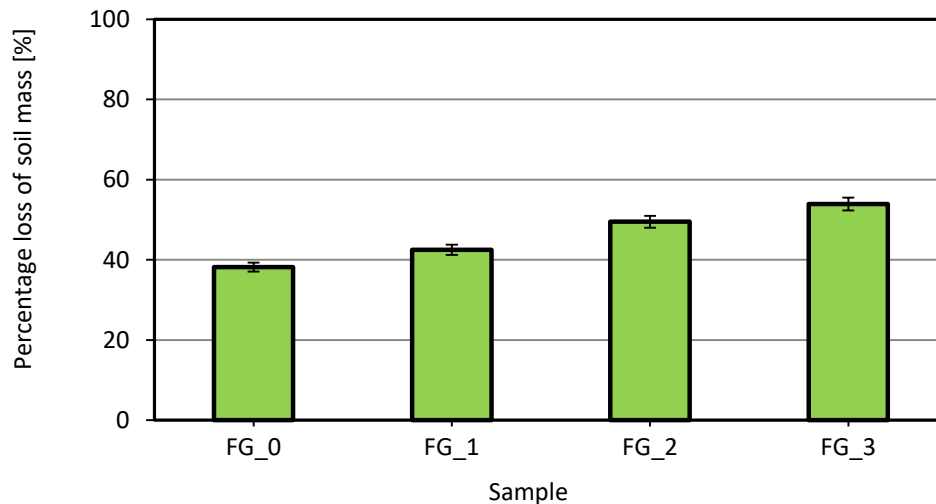


Fig. 3. The evaluation of detergent properties for facial cleansing gels

The superscripts a, b and c above the graph column indicate significant ($p < 0.05$) differences between sample FG_0 and samples FG_1, FG_2, FG_3 for the value of the parameter evaluated

Source: own study.

The addition of digestate extract affects the detergent properties of the tested prototypes of face cleansing gels. Losses of hydrophobic dirt mass after contact with the analyzed gels at the level of 38–53% were obtained. An increase in dirt mass loss after contact with the solutions of the tested cosmetics was observed with an increase in the concentration of extract in the cosmetics prototypes. The increase in detergent activity of the prototypes may be due to the introduction of additional compounds – surfactin, showing surface activity, and solvents.

Determination of the color parameters

For consumers of hygienic cosmetics, an important parameter for assessing quality is their visual impression (Amberg & Fogarassy, 2019). For this reason, conventional washing cosmetics are enriched with specially selected dyes that give them the appropriate color to encourage the consumer to buy the cosmetic (Guerra et al., 2018). The dyes used in cosmetics are usually synthetic raw materials of synthetic origin, which are generating increasing controversy due to emerging information questioning their safety of use (Borowska & Brzóška, 2015; Guerra et al., 2018). In the case of cosmetics declared as natural, usually no dyes are applied, and the color of the product results only from the natural compounds used in their manufacture (Bujak et al., 2022). No dyes were used in the facial

cleansing gels developed in this study, so it was particularly important to determine how the use of post-ferment extract would affect the appearance of the prepared prototypes. The results of the colorimetric evaluation of the face cleansing gel prototypes are shown in Table 4.

Tab. 4. Spectrophotometric data of the cosmetics prototypes

Sample	L*	a*	b*	ΔE Cosmetic with Digestate Extract /Reference Sample
FG_0	12.09	-0.07	4.23	-
FG_1	22.65	-0.36	6.92	10.01
FG_2	28.02	-0.51	9.54	16.80
FG_3	30.08	0.07	10.35	22.82

Source: own study.

It was proven that the addition of surfactin-rich digestate extract changes the coloration of cosmetics compared to the FG_0 reference sample. Cosmetics without extract had the lowest L* value. As the extract concentration increased, the L* value became higher, indicating that the products became darker. The parameters a* and b* also varied with the addition of the extract, with a* changing to indicate receiving more red products and b more yellow. The cosmetic prototypes changed from colorless to colored products. The calculated ΔE parameter indicated a strong coloration of the products, with the highest ΔE value obtained for FG_3 cosmetics with the highest concentration of surfactin-rich extract. The relatively high ΔE values (all above a value of 5) indicated that the color change can be perceived with the naked eye by an inexperienced observer (Wan et al. 2019). Color differences between the cosmetic prototypes were clearly evident, with ΔE values of 10.01; 16.80 and 22.82 for FG_1, FG_2 and FG_3, respectively.

Conclusions

The results of work on the development of functional facial cleansing gels using surfactin-rich digestate extract were presented. The obtained prototypes of gels showed full stability and transparency during storage. Based on the viscosity values determined for the gels at given values of shear rate, it was shown that all samples were susceptible to thinning, which guarantees their easy dispensing and distribution on the skin surface. The study of the evaluation of detergent properties showed an increase in the evaluated parameter with increasing concentration of the digestate extract. Decreases in the parameters of the foaming properties were observed for the prototypes. With the increase of the raw material of biotechnological origin in the gels, their foaming ability and foam stability decreased. Prototypes of cosmetics, in which the extract from the ferment was used, revealed a characteristic coloration derived from the raw material used.

Acknowledgments

The authors are thankful for the financial support provided for these studies by the National Centre for Research and Development, Poland. Grant No. POIR.01.01.01-00-1433/19.

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QUALITY EVALUATION OF FACIAL CLEANSING GELS WITH SURFACTIN-RICH DIGESTATE SOLVENT EXTRACT. PART II – SAFETY OF USE

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DOI: 10.56091/CTQS.Qual-20

Abstract

This part of the work presents the results of a study on the evaluation of the quality of prototypes of facial cleansing gels using surfactin-rich digestate extract, in terms of safety of use. The assessment was performed for cosmetics in which the raw material of biotechnological origin was used in concentrations of 20; 40 and 80% w/w. Studies related to the safety of cosmetics in terms of their irritant and drying effects were performed, including zein number determination and bovine albumin test. A study of the ability to emulsify fat was also conducted. The results were compared and contrasted with that obtained for the reference composition, which was a gel prototype not containing digestate extract.

The zein number value determined for the facial cleansing gel prototypes indicated that all the cosmetic compositions produced did not reveal skin irritation. It was observed that as the concentration of the extract in the sample increased, the zein number value decreased. A similar trend was obtained for the results of test with bovine albumin. Thus, it was shown that through replacing water, which is the solvent in the cleansing gel, with the digestate extract, the irritating effect of the cosmetic was reduced in terms of its interaction with epidermal proteins.

In the study of evaluating the ability of cosmetic prototypes to emulsify fats, an increase in the mass of hydrophobic substance possible to emulsify with increasing concentrations of digestate extract in the samples was observed. This was probably the result of the introduction of an additional ingredient into the composition that showed surface activity – surfactin. Thus, the introduction of the extract may guarantee an improvement in the efficiency of the washing process of the cleansing cosmetic, since emulsification of fatty soiling is one of its key elements. On the other hand, the high ability to emulsify fats can contribute to excessive removal of valuable lipids from the epidermis, resulting in considerable drying of the skin after the washing process.

Keywords: cosmetics, extract, safety, skin irritation, quality, facial cleansing gels.

Introduction

Facial cleansing gels are physicochemically most often aqueous solutions of anionic surfactants (with the addition of compounds from the non-ionic and amphoteric group) enriched with various addi-

tives (preservatives, dyes, plant extracts or pH regulators). The gel form is usually obtained by introducing a polymeric viscosity modifier, such as carbomers, xanthan gum, or, less frequently, sodium chloride. The main function of cleansing cosmetics is to remove dirt from the skin surface, and in the case of the discussed group of cosmetics, the surface of facial skin (Barel et al., 2014; Bujak et al. 2018).

Recently, the driving force for the development of the hygiene cosmetics industry (including facial cleansing gels) is related to the environmental concerns. Consumers, who are becoming increasingly environmentally conscious, are looking for products based on renewable raw materials, natural in origin, and at the same time safe to use and functional. For this reason, an increasing number of cosmetics manufacturers are offering natural cosmetics for sale, declaring the implementation of sustainability policies in their company (Amberg and Fogarassy, 2019; Bom et al., 2019; Kolling et al., 2022). In the literature, these types of cosmetics are referred to as biocosmetics. They are defined as products made from natural ingredients derived from plants, animals, microbes, enzymes, insects and organic crops that are free of pesticides and chemical fertilizers. Their naturalness is often confirmed by appropriate certifications, including Ecocert, Cosmébio, NaTrue, USDA Organic, BDIH (Goyal & Jerold, 2021).

The growth potential of the natural cosmetics sector is enormous. The market has seen explosive growth in 2018–2021, and according to analytical reports related to cosmetic products, the global market for organic and natural cosmetics is expected to reach \$25.11 billion by 2024 (Goyal & Jerold, 2021). With the increase in demand for natural and greener products, sustainability concerns are being raised in terms of the use of raw materials obtained from nature and their rational use. For example, the production of oat, barley or wheat extracts used in personal care products raises worries about water pollution (Almendinger et al., 2020; Hwnag et al., 2020), while the production of palm oil-based raw materials may contribute to massive deforestation and threaten plant and animal diversity. Sustainability is multidimensional and must take into account all economic, environmental and social aspects (Bom et al., 2019; Żuchowski & Paździor, 2022).

A company should demonstrate a holistic approach to the environmental performance offered by its products in terms of raw material sourcing, formulation and manufacturing processes, product testing, final packaging, and finally marketing strategies. A company's sustainable policy should analyze all processing steps with regard to resource management, energy use, global impact, waste management, waste consumption, quality control and ultimately lowering the carbon footprint (Goyal & Jerold, 2021). Bom et al. (2019) point out, however, that of all the stages, the design and manufacturing of a cosmetic deserves the most attention. Creating cosmetics based on natural raw materials can

be quite challenging due to possible instability, emerging aesthetic limitations of the product and the inability to achieve the desired quality characteristics, including issues related to the safety of using the cosmetic (Bom et al., 2019).

Cleansing cosmetics are designed to act as an additional source of nutrients for human skin, improve skin barrier function, inhibit the growth of pathogenic microorganisms, and simultaneously cleanse and moisturize skin surfaces (Bouslimani et al. 2019; Heinrich et al., 2014; Purnamawati et al., 2017; Yamaguchi et al., 2017). The use of raw materials of plant origin, including plant extracts (Savic et al., 2021; Sikora et al., 2015), surfactants of natural origin (Klimaszewska et al., 2022; Seweryn & Bujak, 2018) and raw materials obtained through biotechnological processes (Bezerra et al., 2018; Ferreira et al., 2017; Manga et al., 2021) is the subject of intensive research.

Due to the large range of possibilities, a promising group of raw materials seems to be those obtained by biotechnological processes. Raw materials such as biopolymers (Domżał-Kędzia et al., 2019), surfactants (Manga et al., 2021), vitamins (Averianova et al., 2020) or bioferments (Majchrzak et al., 2022) are produced by this method. The fermentation of plant material can yield bioferments. The raw materials, obtained with the participation of appropriate bacterial strains from plant material, can contain a number of ingredients of interest for cosmetics, including proteins, ceramides, amino acids and antioxidants (Domżał-Kędzia et al., 2019; Majchrzak et al., 2022; Mathur et al., 2020; Ziemlewska et al., 2021). Anti-aging (Bae et al., 2012), antioxidant (Lin et al., 2022), moisturizing (Ziemlewska et al., 2021) and anti-irritant (Domżał-Kędzia et al., 2019) effects have been demonstrated for them. Great interest is being paid to biosurfactants (Bjerk et al., 2021; Jahan et al. 2020) which are produced as secondary metabolites by bacteria, yeast and fungi (Manga et al., 2021). Biosurfactants are natural compounds that exhibit surface activity, effectively reduce interfacial tension, exhibit antioxidant activity (Kumar, 2021) and improve the bacterial microflora of the skin (Adu et al., 2020). An example of a biosurfactant is surfactin. It has low critical micellar concentration values and is highly effective in lowering interfacial tension (Ganesan & Rangarajan, 2021). Compared to synthetic surfactants, surfactin exhibits limited skin irritation and low toxicity (Fei et al., 2020). It also has antibacterial properties (Chen et al., 2022).

The subject of this work was to evaluate the safety of facial cleansing gels containing solvent-based surfactin-rich digestate extract. In the research work, the use of the extract in facial cleansing gels was proposed. Scientific literature revealed that bioferments have a variety of beneficial properties in terms of application in cosmetics. They exhibit preservative, antioxidant or anti-aging effects (Lee et al., 2020; Majchrzak et al., 2022). Under appropriate conditions of the fermentation process, a raw material rich in surfactin, mineral salts, proteins and fatty acids was obtained. The description

of obtaining the extract and studies on its composition are presented in another work of the Authors (Seweryn et al., 2023). In the presented work, in the first stage, formulas were developed, based on which prototypes of face cleansing gels were prepared, where the concentration of the surfactin-rich extract was a variable. A sample without the extract in the formulation was the reference in the study.

Only ingredients approved for application in natural cosmetics were used in the product development. For the prototypes of facial cleansing gels, studies to evaluate their safety of use in terms of skin effects were carried out. Irritant evaluation tests were performed regarding the effect on epidermal proteins (zein test, bovine albumin test), and the influence of the manufactured compositions on epidermal lipids and its barrier functions was evaluated (ability to emulsify fatty dirt, measurements of transepidermal water loss – TEWL).

Materials and methods

Raw materials

For the preparation of the facial cleansing gel prototypes, the following COSMOS-compliant raw materials were used: Sodium Coco Sulfate (trade name: Sulfopon 1216 G; supplier: BASF, Ludwigshafen, Germany), Cocamidopropyl Betaine (trade name: Deghyton K; supplier: BASF, Ludwigshafen, Germany), Coco Glucoside (trade name: Plantacare 818; supplier: BASF, Ludwigshafen, Germany), Glycerin (trade name: Organic Glycerin, supplier: Laboratoires Prod'Hyg, Houdan, France), Lactic Acid (trade name: lactic acid 88%, supplier: WarChem, Warsaw, Poland), Bacillus Subtilis Ferment Extract, Propanediol, Surfactin (supplier: InventionBio, Bydgoszcz, Poland), Xanthan Gum (trade name: Cosphaderm X34; supplier: Cosphatec GmbH, Hamburg, Germany), distilled water.

Facial cleansing gels formulations and manufacturing

Formulas were developed, based on which prototypes of facial cleansing gels were produced. The composition of the products was as follows 4% w/w Sodium Coco Sulfate; 2% w/w Cocamidopropyl Betaine; 1.5% w/w Coco Glucoside; 0.5% w/w Glycerin; 0.25% w/w Xanthan Gum as a viscosity modifier; 0.5% w/w mixture of Sodium Benzoate and Potassium Sorbate as a preservative, Lactic Acid as a pH adjuster and water. The variable in the compositions was a solvent-based surfactin-rich ferment extract (Bacillus Subtilis Ferment Extract, propanediol, surfactin), which was used in concentrations of: 20 (FG_1); 40 (FG_2) and 80 (FG_3) % w/w, replacing water. A prototype containing no extract (FG_0) was the reference composition in the study. Cosmetic prototypes were prepared using a homogenizing mixer type MZUTL 5 (Urliński, Poland) with a capacity of 5 L. After completely dissolving Sodium Coco Sulfate in water at 95°C with continuous stirring (50 rpm), raw materials were

added in the order listed above (up to and including glycerin). The solution was stirred until clear. The digest extract and preservative were then introduced, previously lowering the temperature of the system to about 40°C. In the last step, Xanthan Gum was introduced by dispensing it on the surface of the stirring liquid. The mixture was stirred until a clear, homogeneous solution was obtained. The pH was adjusted to a value of 5.5. The prepared samples of prototype face cleansing gels were tested 24 hours after their preparation.

Zein value

Irritant potential of the facial cleansing gel was measured using zein number test. The detailed methodology of the study was described by Seweryn and Bujak (Seweryn & Bujak, 2018).

pH rise test with bovine albumin serum (BSA)

The test was based on measuring the degree of protein denaturation by determining the pH level of the BSA solution in the solution of the studied cosmetic. The greater its increase, the stronger the skin irritation effect produced by the product concerned. The results were expressed as a percent increase in the pH value in relation to the level defined for normal human skin (pH = 5.5). The test methodology was described in more detail by Bujak et al. (2015).

Ability to emulsify fatty soils

The ability to emulsify fatty soils was evaluated in tests in accordance with PN-C-77003. The test methodology was described by Seweryn and Bujak (2018).

Measurement of transepidermal water loss (TEWL) of skin after washing

Transepidermal water loss was measured using a Tewameter TM 300 device (Courage-Khazaka, Germany) connected to a Cutometer MPA 580 adapter. The study was conducted 1 hour, 2 hours and 3 hours after application of the cosmetic prototypes on the skin. The testing methodology was described by Wasilewski et al. (2016).

Statistical analysis

For turbidity, detergent properties, foaming ability, foaming stability factor and viscosity data were reported as a mean \pm standard deviation (SD), with 3 replicates for each sample (n=3). For comparing the statistical significance of values, ANOVA and a Tukey HSD post hoc test were applied. Differences were considered significant with the p-value was <0.05. The Statistica ver. 10 software (StatSoft, Tulsa, OK, USA) was used for calculations.

Results and discussion

Cosmetics based on raw materials of natural origin are assumed to be fully safe for the consumer. The use of raw materials straight from nature, with as little processing as possible, is supposed to ensure that a fully skin-friendly product is obtained (Dini & Laneri, 2021; Fonseca-Santos et al., 2015). The mandatory analysis in the European Union in the form of a safety card for cosmetics placed on the market, including those produced from raw materials of natural origin, is designed to ensure the high safety of the products offered on the market (Pauwels & Rogiers, 2010). Nevertheless, there are cases of negative effects of the cosmetic on the user, especially on the skin surface. In the case of cleansing cosmetics, skin irritation may occur as a result of contact between the cleansing bath formed with the cosmetic and the skin surface (Seweryn et al., 2018). The use of raw materials of natural origin in a cosmetic does not guarantee a final non-irritating product. Particularly in the case of the use of anionic surfactants in the formulation, whether or not a final cleansing cosmetic will exhibit an irritant effect is determined by the structure of the surfactants used (Seweryn, 2018). As an example, Sodium Coco Sulfate, despite its proven naturalness and approval for use in natural cosmetics, exhibits similar irritant effects to its petrochemical counterpart Sodium Laureth Sulfate (Bujak et al., 2019).

In this work, cosmetic prototypes were evaluated for their irritant effects. The products were analyzed on two stages. First, an evaluation was made for the possibility of irritation resulting from interactions with the epidermal building proteins that are the main cause of irritation symptoms in the form of redness, burning or itching of the skin. The prototypes were then evaluated in terms of their interaction with the lipids found in the epidermis and the interference of the washing baths with the barrier functions of the skin. Irritation resulting from interaction with proteins was evaluated by the zein number assay and the bovine albumin test. In both of these tests, the interaction of a given cosmetic with a model protein (zein or albumin) is assessed instrumentally, and the result indicates the level of irritant effect of the tested product (Wasilewski et al., 2022). The obtained values of zein number are shown in Figure 1.

The zein number values obtained for the tested cosmetic prototypes are within a fairly wide value range of 20–120 mg N/100 ml. In the case of the reference sample, the obtained results, according to the literature (Pezron et al., 1996), indicate a slight irritant effect (120 mg N/100 ml). In the case of the remaining facial gels, significant decreases in the evaluated parameter were observed, with the largest decrease to a value of 20 mg N/100 ml obtained for the FG_3 prototype, with the highest analyzed concentration of the post-ferment extract. The data obtained indicate that these prototypes, in accordance with literature data (Pezron et al., 1996), do not exhibit skin irritation. Support-

ing the results of the zein test is the bovine albumin assay. The results of this study are shown in Figure 2.

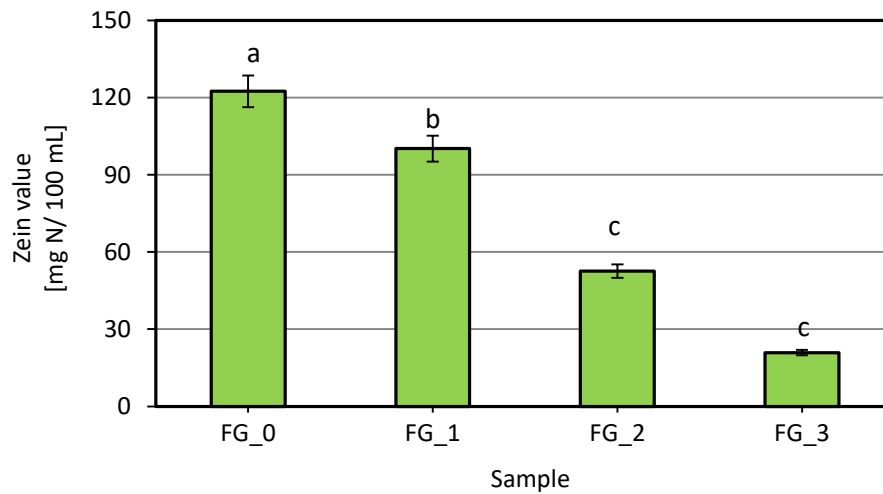


Fig. 1. Zein value of the facial cleansing gels with the digestate extract

The superscripts a, b and c above the graph column indicate significant ($p < 0.05$) differences between sample FG_0 and samples FG_1, FG_2, FG_3 for zein value

Source: own study.

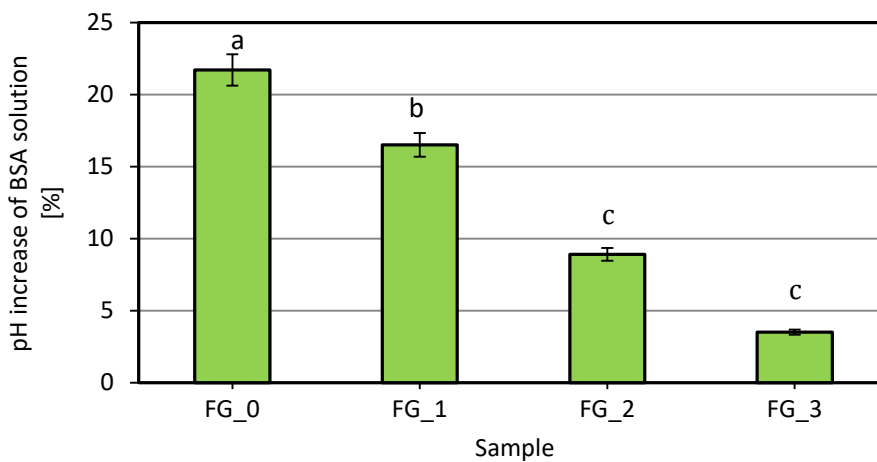


Fig. 2. Changes in pH of the mixture of bovine serum albumin solutions (BSA) and facial cleansing gels

The superscripts a, b and c above the graph column indicate significant ($p < 0.05$) differences between sample FG_0 and samples FG_1, FG_2, FG_3 for changes in pH

Source: own study.

The results of the albumin test were consistent with the results of the zein number determination (Figure 1). The highest increase in the pH of the solution was obtained for the reference composition FG_0, indicating its highest irritant effect. For the other prototypes, the increase in pH above the value of 5.5 assumed for healthy human skin was characterized by increasingly lower values. Such

results indicate that the introduction of surfactin-rich digestate extract reduces skin irritation in terms of negative effects on skin-building proteins of the epidermis.

The obtained results indicating a reduction in irritant effects may be due to the presence of a biosurfactant in the extract as well as the complexity of the extract composition. In the literature, an anti-irritant effect has been demonstrated for surfactin (Fei et al., 2020). Such an effect was also confirmed for other components of the extract such as mineral salts (Ferreira et al., 2010), hydrophobic substances (Wasilewski et al., 2016) or macromolecular compounds (Bujak et al., 2018).

Effects of cosmetic prototypes on epidermal lipids and their barrier function

Cosmetics undergo rigorous quality assessment by the consumer. In the case of cosmetics intended for washing, in addition to adequate performance related to the product's function, it is expected that the product will not cause excessive drying of the skin after the washing process. Dryness of the skin after the application of washing cosmetics is associated with too high removal of fatty components from the surface of the skin. This effect is closely related to the washing process and the ability of the washing bath to emulsify and solubilize hydrophobic components from the skin. Particularly dangerous are the extraction of components of the intercellular cement of the stratum corneum, changes in the pH of the skin due to disruption of the bacterial flora, and disturbances in the area of the water and lipid layer and components of the natural moisturizing factor (Morris et al., 2022). As a result of such changes, the epidermal barrier may be disrupted, which appears as excessive water loss from the epidermis and dryness. Measurements of TEWL from the epidermis allow to assess the state of the skin in terms of its barrier function. The higher the value of the evaluated parameter, the higher the probability of skin dryness (Uehara et al., 2023). For the analyzed prototypes of washing cosmetics, relevant TEWL measurements of the skin after the washing process were conducted. The results are shown in Figure 3.

Measurements of TEWL for the skin after the washing process using the analyzed cosmetic prototypes show that the tested samples do not affect the condition of the epidermal barrier. For all analyzed preparations, similar TEWL values within 4 g/m²/h were obtained. They were slightly lower than the values determined for the skin before the washing process. There was no effect of the addition of the extract or its concentration on the value of the determined parameter. Although a surfactin-rich extract was introduced into the cosmetic, no increase in TEWL values was observed, which could be due to the appearance of an additional ingredient in the composition of the cosmetic that exhibits surface activity (surfactin derived from the surfactin extract). The study showed a nearly neutral effect on the skin surface of the analyzed prototype cleansing cosmetics.

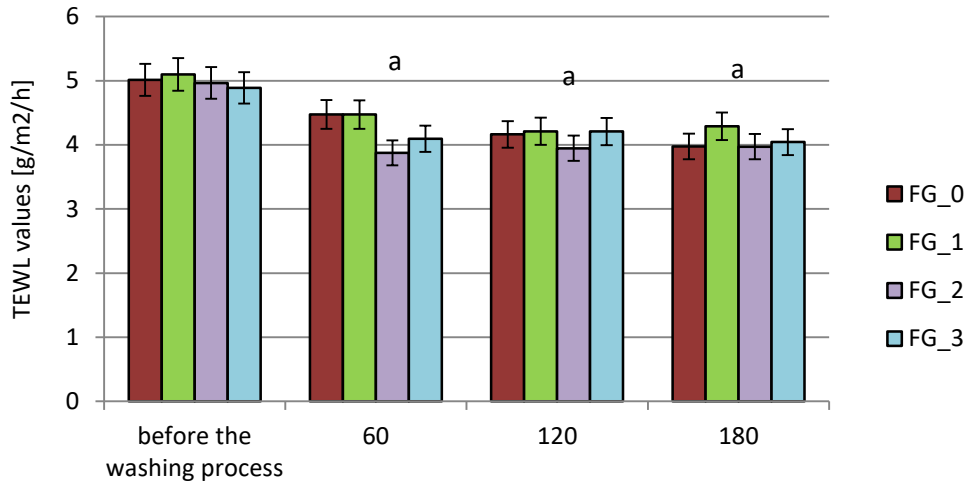


Fig. 3. Influence of prototypes facial cleansing gels containing digestate extract on TEWL
 The superscripts a above the graph columns indicate significant ($p < 0.05$) differences between sample FG_0 and samples FG_1, FG_2, FG_3 for TEWL
 Source: own study.

In the next stage of the study, the ability of the cosmetic prototypes to emulsify fatty soil was evaluated. The results are shown in Figure 4.

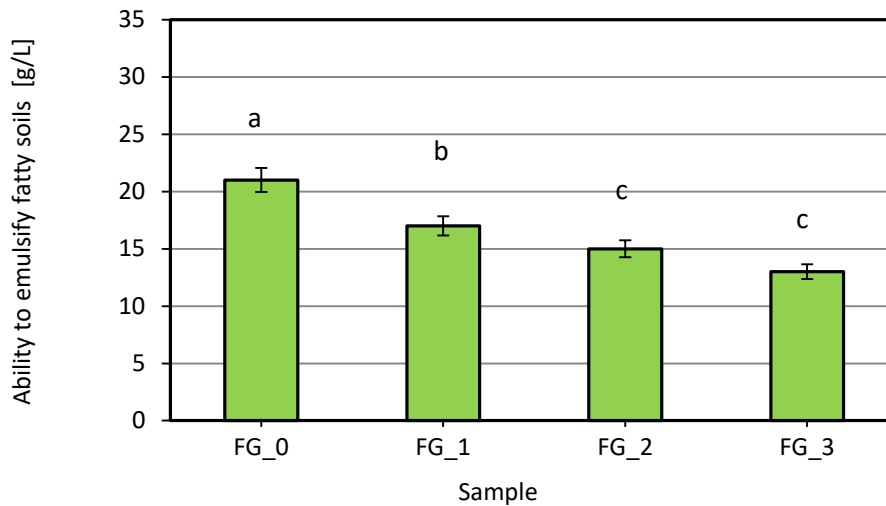


Fig. 4. Ability to emulsify fatty soils determined for facial cleansing gels containing digestate extract
 The superscripts a, b and c above the graph column indicate significant ($p < 0.05$) differences between sample FG_0 and samples FG_1, FG_2, FG_3 for the value of the parameter evaluated
 Source: own study.

The evaluated parameter can provide information about the functionality of the product, as well as safety in terms of potential negative effects on the skin. Emulsification is a partial step in the washing process and determines the effective removal of dirt from the surface (Seweryn & Bujak, 2018).

Determining the value of the dirt mass that can be emulsified by a given cosmetic allows to assess its washing effect. However, too high values of this parameter may indicate strong negative extraction of lipids from the epidermis. This can lead to leaching of valuable lipid components from the skin and, moreover, to depletion and destruction of the intercellular cement of the stratum corneum, resulting in disruption of the skin's barrier functions. As a result, an increase in TEWL and skin dryness is observed (Mijaljica et al., 2022). The determined values of the studied parameter are in the range of 21–13 g/L. The highest value of the evaluated parameter was determined for the FG_0 sample, while the lowest value was determined for the FG_3 prototype with the highest analyzed concentration of the digest extract. The results clearly indicate that the introduction of post-ferment extract into the composition of a cosmetic leads to a decrease in the ability to emulsify fats. This may affect the cosmetic's performance related to its intended use (cleansing action). However, according to the preference of consumers focused on the search for cosmetics gentle to the skin, the extract lowering the ability of the system to emulsify hydrophobic substances makes the analyzed prototypes of gels softer in contact with the skin. This is confirmed by studies on TEWL after application of cosmetic prototypes to the skin (Figure3), where no effect of the tested compositions on epidermal barrier functions was observed.

Conclusions

In this part of the work, prototypes of facial cleansing gels containing a solvent-based surfactin-rich digestate extract were evaluated for their safety of use in terms of skin effects. The cosmetic prototypes prepared on the basis of the original formulations were tested in terms of skin irritation by determining the zein number and performing a bovine albumin test. Moreover, the gels were evaluated for their effects on lipids and epidermal barrier functions by determining TEWL after cosmetics contact with the skin, and by testing the ability to emulsify fats. The results of the zein number determination and the bovine albumin test indicate that the digest extract introduced into the gels effectively reduces their irritant effects. Compared to the reference composition, prototypes containing digestate extracts showed low zein number values and low pH increments in solution with bovine albumin. The effect of extract concentration on the resulting tendency to decrease irritant activity was observed. In the case of TEWL measurements, there was no significant effect of the tested compositions on epidermal barrier function. The results of fat emulsifying ability indicate that the analyzed extract reduces the effectiveness of cosmetics in this regard. This is important from the consumer's point of view, since on the one hand it reduces the functionality of the product in terms of cleansing action, while at the same time it increases its safety by making it more gentle to the skin in terms of drying action.

Acknowledgments

The authors are grateful for the financial support provided for this research by the National Center for Research and Development, Poland. Grant No. POIR.01.01.01-00-1433/19.

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SELECTED PROPERTIES AND EMULSION STABILITY OF NEW FACIAL SERUM WITH HYALURONIC ACID

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DOI: 10.56091/CTQS.Qual-21

Abstract

Nowadays, physical appearance has become an important determinant of social standing. The fashion for being eternally beautiful and young does not pass, and almost everyone professes the cult of taking care of one's appearance. Manufacturers flood the market with cosmetics with various effects: anti-ageing, beautifying, slimming or moisturising, and of course many, many others. Customers in the flood of products are looking for newer, more effective ones that will improve their appearance. The present study evaluates selected parameters of quality and stability of serums containing different concentrations of hyaluronic acid. The research allowed us to determine the behaviour of the cosmetic mass, to evaluate the functional properties, and its stability under various temperature conditions.

Keywords: quality, serum, hyaluronic acid, stability of product

Introduction

The serum is one of the modern forms of cosmetic product, available in both the skincare and dermatological cosmetic markets. This product can be of various consistencies, and due to its highly potent action, it is becoming an essential element in the care of the skin of the face, scalp or body. Serum is a form of cosmetic, which is characterized by a high concentration of biologically active complexes, much higher than in creams. It has a liquid or semi-liquid consistency, which facilitates optimal absorption by the skin. The active ingredients are responsible for the potent action of the preparation and most often target a specific skin problem, such as improving colour, smoothing, firming or regenerating the epidermis (Dylewska-Grzelakowska, 2011; Kozak-Chaber, 2019). The application of the serum is the next stage of skin care. It is recommended to apply it immediately after cleansing the skin, but before a moisturizing treatment, such as applying a cream. It should not be used as the only part of skincare, as used alone it will not do its job. Serum belongs to cosmetic products composed of small molecules, which makes it easier for the product to penetrate the deeper layers of the skin. It can be said that serums are the opposite of creams, as such moisturizers con-

sist of larger molecules and moisturize only the outer layer of the skin. Therefore, to increase the effectiveness of both serums and creams, they should be used together (Mahto, 2019). Hyaluronic acid (HA) belongs to the group of natural polysaccharides, i.e. it is a polysaccharide. This acid is a biopolymer with a high, yet variable molecular weight. Hyaluronic acid molecules have a left-handed helix structure stabilised by hydrogen bridges. Typically in living organisms, it is in the form of a sodium salt (sodium hyaluronate) (Sionkowska, 2019; Wasiluk, 2019). Hyaluronic acid can be extracted from rooster combs, shark skin or the vitreous body of cattle; for example, 5kg of combs yields approximately 5–8g of acid. With the increase in demand for hyaluronic acid, it is increasingly being produced by biotechnological methods that use streptococci cultured on a molasses medium. Unlike hyaluronic acid extracted from animal tissues, the one obtained by microbiological methods is easy to purify and does not carry the risk of interspecies infection. However, there is a risk of mutation in bacterial strains, and the possibility of producing various toxins or immunogens. Therefore, in medicine, where hyaluronic acid is injected directly into the body, cockle comb acid is used (Lukas, 2014; Placek, 2009; Sionkowska, 2019).

Hyaluronic acid has many important and valuable properties that determine its numerous applications (Sionkowska, 2019). Products that have hyaluronic acid in their composition come in many pharmaceutical forms and are registered as medicinal products, medical devices and cosmetics (Olejnik et al., 2012).

Hygroscopicity, or the ability to bind large amounts of water, is considered the greatest advantage of hyaluronic acid. Hyaluronic acid also regulates osmotic pressure and the transport of raw materials and metabolites. In an aqueous environment, hyaluronic acid molecules can increase their volume 1000-fold, and in this way can form a permanent network stabilized by hydrogen bonds. One molecule can bind from about 200 to 500 water molecules, depending on its molar mass (1 g of acid retains about 6 litres of water) (Olejnik et al, 2012; Sarbak et al, 2013; Sionkowska, 2019).

Hyaluronic acid has a wide range of applications, not only in many fields of medicine but also in cosmetology. The main branches of medicine where it is used are urology, general surgery and orthopaedics. It is also of great importance in aesthetic cosmetology. Currently, hyaluronic acid is the most popular substance used by dermatologists and aesthetic physicians, especially for regenerating, rebuilding and smoothing skin and filling in wrinkles or furrows, it is also used to add volume to specific parts of the face or body (Kucia, 2017). Hyaluronic acid and its salt (sodium hyaluronate) are active ingredients in many cosmetics for various purposes. Due to its properties, this compound can be found in moisturising, protective or anti-ageing products. It is mainly used in face, neck and eye care products, e.g. masks, creams, toners and serums. It can also be found in many full-body cosmet-

ics, mainly anti-cellulite and stretch mark products (Kucia, 2017; Olejnik et al., 2012). The benefits of hyaluronic acid in cosmetics only apply to the skin surface. Hyaluronic acid has a high molecular weight, which means that it does not penetrate deep into the epidermis, but remains on the surface forming a protective layer that shields the stratum corneum and participates in the formation of biological barriers, which is why the skin becomes smoother and well-hydrated after using products with hyaluronic acid by reducing transepidermal water loss. In many cosmetic products, hyaluronic acid acts as a surface humectant, i.e. an ingredient that maintains moisture in the cosmetic formulation and prevents it from drying out (Kucia, 2017; Olejnik et al., 2012).

Nowadays, when a great variety of substances and compounds with various effects are available on the cosmetic market, manufacturers have to pay attention to many factors to launch a new product, which above all must be safe and stable. Due to the wide variety of cosmetic forms, it is difficult to define a specific method of stability testing, everything depends on the type of product, the way it is used and applied, the type of packaging, composition or storage and transport conditions.

It is important to define the meaning of the word stability, according to Petsitis and Kipper: “The term product stability means microbiological stability on the one hand, and stability during storage at different temperatures on the other. Colloquially, the period during which a product is intended for use is called shelf life” (Pytrus-Sędkak, 2007, pp. 111).

Conducting stability tests is intended to help manufacturers evaluate their product for shelf life, that is, how the original parameters of a cosmetic compound will change over time. The important element is to ensure that the product has the correct properties over its shelf life, under certain storage conditions during normal use (Ozga, 2012). To ensure the microbiological stability of products, preservative treatments are applied or preservatives are added. The most common preservatives are chemicals that are intended to prevent spoilage and protect the user from damage to health. In many cases, the addition of a preservative is necessary because microbiological safety must be ensured throughout use (Pytrus-Sędkak, 2007). Stability during storage at different temperatures involves holding the products under test at 3–5°C, 40°C and at room temperature of about 22°C. It is also possible to perform so-called stress (rapid) tests, which are performed to test the sensitivity of substances contained in a cosmetic product to changing conditions. Samples are tested alternately at high and low temperatures. Stress tests are designed to check whether a cosmetic can withstand extreme conditions, since in normal use, during the day, the temperature can vary between 0, 20, and 40°C. If, for example, phase separation, crystallization or sedimentation occurs during testing, or there are changes in consistency, colour or odour then we cannot consider the product stable. In this case, changes must be made to the formulation. Testing times can vary, the minimum period is con-

sidered to be from 3 months up to the full shelf life. The following parameters are considered important for the quality of the product: pH value, viscosity, odour, and colour appearance. These properties are checked during testing at specific intervals. Typically, tests are conducted at an interval of 2 weeks to observe changes (Ozga, 2012; Pytrus-Sędkak, 2007).

Experimental

The study aimed to assess the effect of the concentration of the active substance – hyaluronic acid present in the cosmetic preparation – on selected parameters of the cosmetic serum. A formulation of facial moisturising serums was developed, which contained different concentrations of hyaluronic acid and sodium salt in their compositions. This was followed by a comparative analysis of the functional properties and an examination of the formulations in terms of their storage stability.

The scope of the research included:

- pH measurement,
- sensory analysis of serum,
- moisture measurement in the epidermis using a corneometer,
- measurement of water loss from the epidermis using a tewameter (TEWL),
- infrared (IR) spectroscopic measurements.

Research material

In the first part of the study, a moisturising serum formulation was developed, the composition of which is shown in Table 1. Three products were prepared, each differing in the concentration of hyaluronic acid sodium salt.

Tab. 1. Moisturising serum formulation

Ingredients (Name, INCI, Company)	Composition [%]	Ingredient properties
Lactic acid 80% INCI: <i>Lactic acid</i>	5.0	Lactic acid has a moisturizing and softening effect on the skin. In the right concentration, it is also used as an antimicrobial, which makes it support the action of preservatives in cosmetics. It is referred to as a so-called alpha-hydroxy acid. Its low molecular weight makes it more deeply absorbed by the skin (ecospa.co.uk/lactic-acid-80).
Geranium hydrolate (INCI: Pelargonium X Asperum Flower Water)	69.3	The product is very intensively moisturizing and smoothing. When used in daily care, it minimizes the harmful effects of external factors. It deeply hydrates and nourishes the skin. This hydrolate is obtained from the Pelargonium x asperum plant by steam distillation (ecospa.pl/hydrolat-geranium-ecological).

Selected sodium salt of hyaluronic acid (1.5%, 2%, 3%) (INCI: Sodium Hyaluronate)	15.0	Tri-particle hyaluronic acids 1.5% and 3% consist of three types of acids of different molecular weights in corresponding concentrations. Each performs a specific function. Low-molecular-weight acids of the SLMW (super low molecular weight) and LMW (low molecular weight) types increase hydration at the level of the inner layers of the skin and, through their moisturizing effect, enable faster penetration of active substances. High molecular weight (HMW) hyaluronic acid is designed to hold moisture in the skin. Triple hyaluronic acids are designed to enhance the skin's protective properties and maintain adequate moisture levels (ecospa.co.uk/triple-hyaluronic-acid-3-solution). Hyaluronic acid 2% consists of two types of acid: a low-molecular type SLMW and a high-molecular type HMW. It reaches both the superficial and inner layers of the skin while maintaining maximum water-binding capacity in the skin. It ensures an adequate level of skin hydration (esent.pl/en/p/Hyaluronic-acid-solution-2-volume-30-ml).
Broccoli seed oil (INCI: Brassica Oleracea Italica (Broccoli) Seed Oil)	10.0	Broccoli seed oil is a natural alternative to silicone in many cosmetics. The oil is rich in vitamins A and K and also contains potassium, iron and calcium. As a result, our skin becomes more moisturised, regenerated, smooth and pleasant to the touch (ecospa.co.uk/oil-of-broccoli-seeds).
Eco preservative	0.7	Eco preservative consists of four active ingredients: benzyl alcohol, salicylic acid, glycerine, and sorbic acid. It does not contain parabens, formaldehyde, or isothiazolinones. Effective against Gram+, Gram- bacteria, fungi and moulds. The preservative is in the form of a colourless liquid, almost odourless (ecospa.co.uk/eco-liquid-preservative).

Source: own study.

Methods

The tests were carried out weekly for 8 weeks for all four types of lipsticks, stored under various conditions (at a temperature of about 10°C, room temperature, at 35°C and in variable conditions (so-called stress tests): one week at ca. -5°C and another one week at 35°C.

Measurement of the hydration degree of the corneal layer

The hydration degree of the corneal layer of skin was examined using Corneometer® CM 825 (Courage-Khazaka Electronic GmbH, Köln, Germany). The used probes were connected to the Multi Probe Adapter System. The way it operates is based on the measurement of the electrical capacity of skin, thanks to it, the content of water in the stratum corneum of the skin can be checked. The more water contains the stratum corneum, the better the current will flow. The higher the measurement value, the better the skin hydration. Obtained results of measurements are in the range of 0–130 units, where one unit is 0.02 mg of water per 1 cm² of stratum corneum. Measurements were carried out 15 minutes after, and 30 minutes after application.

Measurement of water loss from the epidermis

The transepidermal water loss (TEWL) is associated with the secretion of sweat by sweat glands and the evaporation of water through the epidermis. It is carried out to assess the condition of the skin barrier function. That was measured using the Tewameter® TM 300 (Courage-Khazaka Electronic GmbH, Köln, Germany). The used probes were connected to the Multi Probe Adapter System. Higher TEWL values correspond to lower moisture, and thus, the efficiency of the skin barrier function is reduced. Measurements were performed 15 minutes and 30 minutes after application.

Infrared spectroscopy (IR)

The study consisted of the measurement of base oil spectra and basic infrared (MIR) lipids. The spectra were recorded in the range of 4000–600 cm^{-1} (4700 FTIR spectrophotometer Jasco, Japan), using the method of attenuated total reflection (ATR). All spectra were the average of 16 interferograms. The resolution was 4 cm^{-1} .

pH measurement

The pH was measured for 1% aqueous solutions of each serum type. The determination consisted of reading the pH value from a digital pH meter with a CP-551 glass electrode, which had previously been calibrated with a suitable buffer solution of pH=4. After immersion of the pH meter's measuring electrode, the reading was taken only after the measuring system had stabilized, i.e. when no significant changes in the pH value were observed on the display (BN-74/6140-08/04).

Sensory analysis

Sensory evaluation consists of measuring and assessing the properties of a product through the senses, during use. These tests are carried out both during the development of new formulations and the confirmation of product quality. They characterize the cosmetic product in terms that are closest to everyday sensations when applying the cosmetic to the skin. They have a decisive influence on the acceptance of the product by the consumer. Sensory evaluation of the prepared preparations was carried out according to the guidelines in Table 2, immediately after serum formulation and 4 weeks after formulation. The preparations were evaluated according to a scoring scale from 1 to 5 (1 – worst, 5 best). The study group consisted of 10 subjects – six women and four men, aged 20–25 years (Kulawik-Pióro, 2017).

Tab. 2 Development of a sensory analysis form

Parameters	Scale				
	5	4	3	2	1
Odour	Very pleasant	pleasant	so-so odour	not very pleasant	Unpleasant
Consistency	very light	light	light medium	heavy	very heavy
Application	very easy to apply	easy to apply	medium to apply	difficult to apply	very difficult to apply
Absorption/greasiness	absorbs very well / no film	well absorber/ slightly oily	medium/moderately oily	hardly absorbs	very absorbing / leaves
Cushion effect/ stickiness	very high	high	moderate low	hardly	any
Adhesion/smoothing	very good	good	so-so	very not very good	no adhesion / does not smoothing
Homogeneity	homogeneous	bubbles air	fine lumps	lumps	delaminates

Source: Plocica et al., 2014.

Sensory evaluation of all serum types stored at different temperatures

All serum types stored at room temperature, 10°C, 35°C and under stress conditions (alternating between fridge and greenhouse) were observed and evaluated for 4 weeks. The effect of different temperature conditions on emulsion quality was evaluated. Regardless of hyaluronic acid concentration, the formulations behaved similarly during the 4-week observations.

Results and discussion

Results of pH measurement

pH measurements were made for 1% solutions of each type of serum, stored under different temperature conditions, i.e. room temperature of about 22°C, 10°C, 35°C and stress (variable) conditions. Determinations of pH values were carried out immediately after the serum was made (the so-called zero week) and then weekly for 4 weeks. The results obtained are summarized in Table 3. The pH value of the samples stored at about 22°C, during the 4-week measurements, was in the range of 3.86–4.04. All the tested products immediately after manufacture had very similar pH values. Slight changes could be observed in the following weeks. The pH value of the 1.5% hyaluronic acid serum immediately after manufacture was equal to 3.96, and in the 1st and 2nd weeks it decreased by 0.08 and 0.1, respectively. In the 3rd week, the value increased again, to reach a pH of 4 in the 4th week. The 2% hyaluronic acid serum from the moment of manufacture, where the pH was 3.97, until the 3rd week recorded fluctuations in values of only 0.01 and 0.03. By week 4, the pH had risen to a value of 4.04. The 3% hyaluronic acid serum had a pH of 3.94 after manufacture. By week 2, the value

was decreasing, and by weeks 3 and 4 it had increased by 0.13 and 0.14, respectively. At the last measurement, the pH value of this serum was 4.01.

Tab.3. Results of pH measurement

Hyaluronic acid serum	Week	0	I	II	III	IV
1.5%	10°	3.95	3.96	3.99	4.01	4.03
	22°	3.96	3.88	3.86	3.94	4.0
	35°	3.96	4.02	3.99	4.04	4.04
	variable conditions	3.96	3.9	3.97	4	3.97
2.0%	10°	3.97	3.98	3.99	4.04	4.08
	22°	3.97	3.96	3.94	3.94	4.04
	35°	3.97	4.07	3.89	3.92	3.95
	variable conditions	3.97	3.99	4.08	4.36	4.21
3.0%	10°	3.94	3.99	4.12	4.16	4.18
	22°	3.94	3.91	3.9	4.03	4.01
	35°	3.94	4.01	4.17	4.06	4.07
	variable conditions	3.94	4.05	3.98	4.11	4.09

Source: own study.

During the storage of all types of serums at 10°C, their pH value increased from week to week. In the case of the 1.5% and 2% hyaluronic acid serums, the increase was slight. The pH value of the 1.5% serum immediately after manufacture was 3.95, and after 4 weeks was 4.03, while the pH of the 2% serum at week zero was 3.97, and at the end of the measurements was 4.08. For the 3% hyaluronic acid serum, the greatest increase in pH value was observed. Immediately after manufacture it was 3.94, and within 4 weeks it had risen to 4.18.

Storing all serums in a greenhouse, at 35°C, caused small but numerous fluctuations in the pH value, from the time of manufacture. The 1.5% hyaluronic acid serum experienced the smallest fluctuations in pH value. In week 1, the value increased from 3.96 to 4.02, while already in week 2 it decreased to a value of 3.99. In weeks 3 and 4, the pH value was equal to 4.04. The serum with a concentration of 2% hyaluronic acid in week 1 recorded a pH increase of 0.1, and in week 2 the value decreased by 0.18. In the following week, it increased again and after 4 weeks the pH value was 3.95. The pH value of the 3% hyaluronic acid serum increased by 0.07 after 1 week and was 4.01, while in week 2 the

value increased to 4.17 and decreased in the following weeks. In the 4th week of measurements, the pH value was equal to 4.07.

The pH value of the stored samples under stress conditions fluctuated numerous times during the 4-week measurements. The pH values of the 1.5% and 3% hyaluronic acid serum were highly variable from week to week. If there was an increase in the pH value in one week, there was a decrease in the next week. In the end, the value in the 4th week of measurements of the 1.5% serum was 3.97, and that of the 3% serum was 4.09. The pH value of the 2% hyaluronic acid-added serum increased until the 3rd week of measurements, where in the 3rd week the value was 4.36, 0.39 more than immediately after the formulation. In the 4th week of measurements, there was a decrease in the pH value, which eventually reached 4.21.

Sensory evaluation of all serums stored at room temperature

Sensory evaluation was carried out according to the guidelines in Table 4, immediately after serum manufacture and 4 weeks after manufacture. The preparations were evaluated according to a point scale from 1 to 5 (1 – worst, 5 best). The study group consisted of 10 subjects – 6 women and 4 men, aged 20–25 years. To better illustrate the sensory evaluation of the preparations produced and the differences that occurred between them. Analyzing the results of the sensory evaluation, it can be seen that the highest-rated parameters for all types of serums were primarily texture and spreading. Their ratings were in the range of 4–5 points. The formulations produced also had their weaknesses. The lowest-rated parameters were odour, adhesion, stickiness and cushion effect. Their ratings were in the 1–3 point range. Odour was rated worst, and the spreadability of the preparations was rated best. All serums in the overall evaluation had similar values. The serum with 3% hyaluronic acid was rated best. According to the respondents, it distinguished itself from the other types by its texture, distribution on the skin, uniformity of the emulsion and smoothing effect on the skin. The serum showed a low level of greasiness and oiliness of the epidermis. The average rating of the serum according to the respondents was 3.39 points. The 2% hyaluronic acid serum was rated at 3.14 points. This formulation was only minimally distinguished by its adhesion from other types of serums. Respondents considered its weakest parameter, compared to other serums, to be the cushion effect. This formulation absorbed just as well as the 3% hyaluronic acid serum. The serum with 1.5% hyaluronic acid was rated the worst. Its overall score was 2.98 points. The weakest rated parameters are stickiness, smoothness, adhesion, consistency, uniformity and absorption of the product into the skin. This serum shows the highest skin lubricating abilities.

Sensory evaluation of all types of serum stored at different temperatures

All types of serum stored at room temperature, 10°C, 35°C and under stress conditions (alternately in a refrigerator and a heat box) were observed and evaluated for 4 weeks. The effect of different temperature conditions on emulsion quality was evaluated. The formulations, regardless of the concentration of hyaluronic acid, behaved very similarly during the 4-week observations. Any changes and observations are presented in Table 4.

Tab. 4. Observation of changes in all types of serum stored in different conditions

Temperature	Observations	Comments
22°C	3 weeks after manufacture, small particles/bubbles appeared, with slight turbidity of emulsion	The smell of broccoli oil and lactic acid (unpleasant odour) increased from week to week
10°C	The consistency was denser, which increased the adhesion of the formulation, high clarity of the formulation	Low temperature had a beneficial effect on the fragrance, the intensity of geranium hydrolate (floral fragrance) was strengthened
35°C	After 2 weeks, small air bubbles, very large and visible turbidity of the solution	Intense, unpleasant, very noticeable smell of broccoli oil and lactic acid, long-lasting on the skin
different conditions	After 3 weeks, numerous lumps, air bubbles, and turbidity in the solution	Storage in the greenhouse enhanced the unpleasant odour of broccoli oil and lactic acid

Source: own study.

Results of IR spectrophotometric analysis of all types of serum stored at different temperatures and under stressful conditions

When the spectra were analyzed, the presence of a band at 3447 cm^{-1} was noted, which is attributed to the stretching region of the -OH and -NH groups. The band at about 1634 cm^{-1} corresponds to the amide carbonyl, and the band at 1420 cm^{-1} can be attributed to COO⁻-stretching, which relates to the acid group of the hyaluronic acid molecule. The absorption band at 1019 cm^{-1} is attributed to the stretching of the -OH group bond. The stretching region of the -COOH proton group is observed at 1735 and 1255 cm^{-1} (Silverstein, R. M. 1969).

Figures 1–4 present the obtained spectra of the different types of serum stored at room temperature of about 22°C, at 10°C and 35°C, and under alternating conditions: one week at 10°C and one week at 35°C, for 4 weeks.

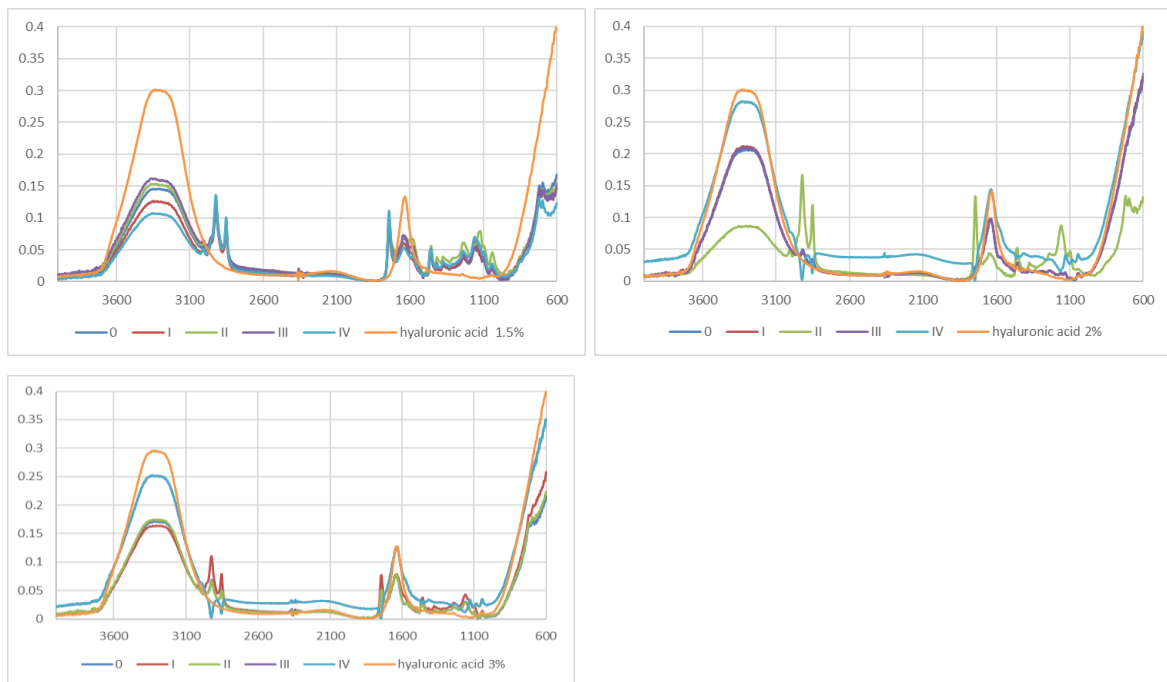


Fig. 1. The IR spectrum of a serum containing 1.5%, 2%, and 3% hyaluronic acid which has been stored at a temperature of 22°C with the access light
Source: own study.

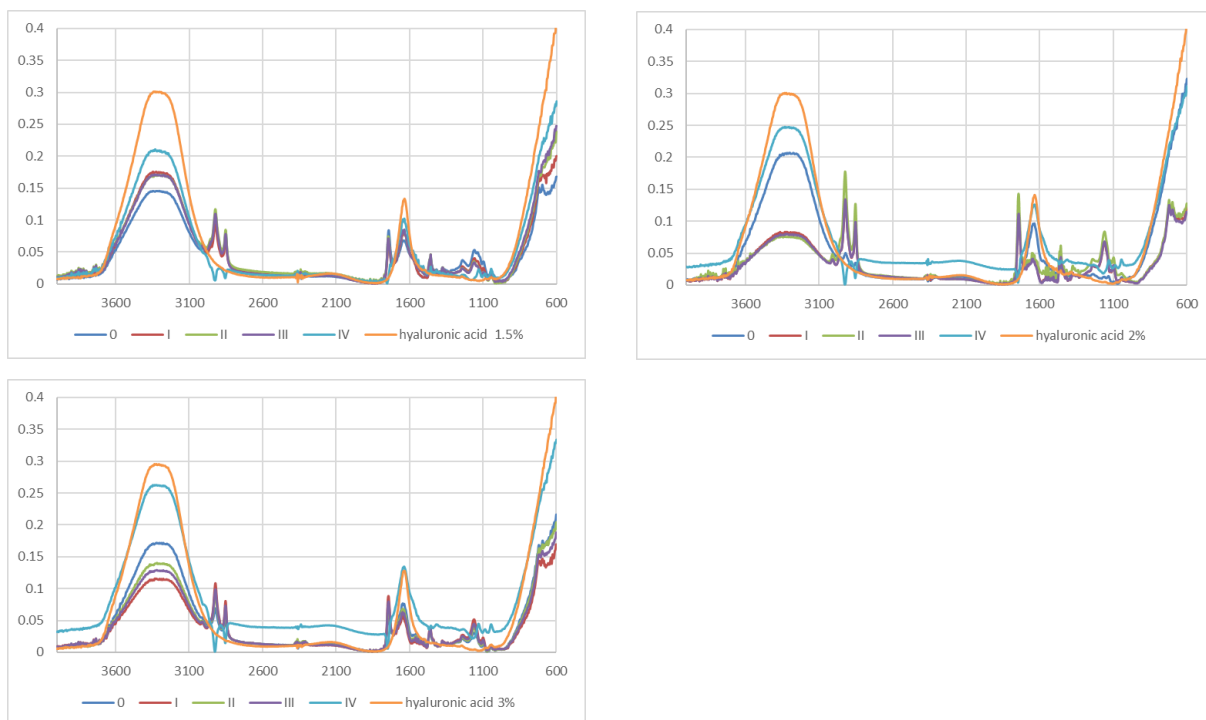


Fig. 2. The IR spectrum of a serum containing 1.5%, 2%, and 3% hyaluronic acid which has been stored at a temperature of 10°C
Source: own study.

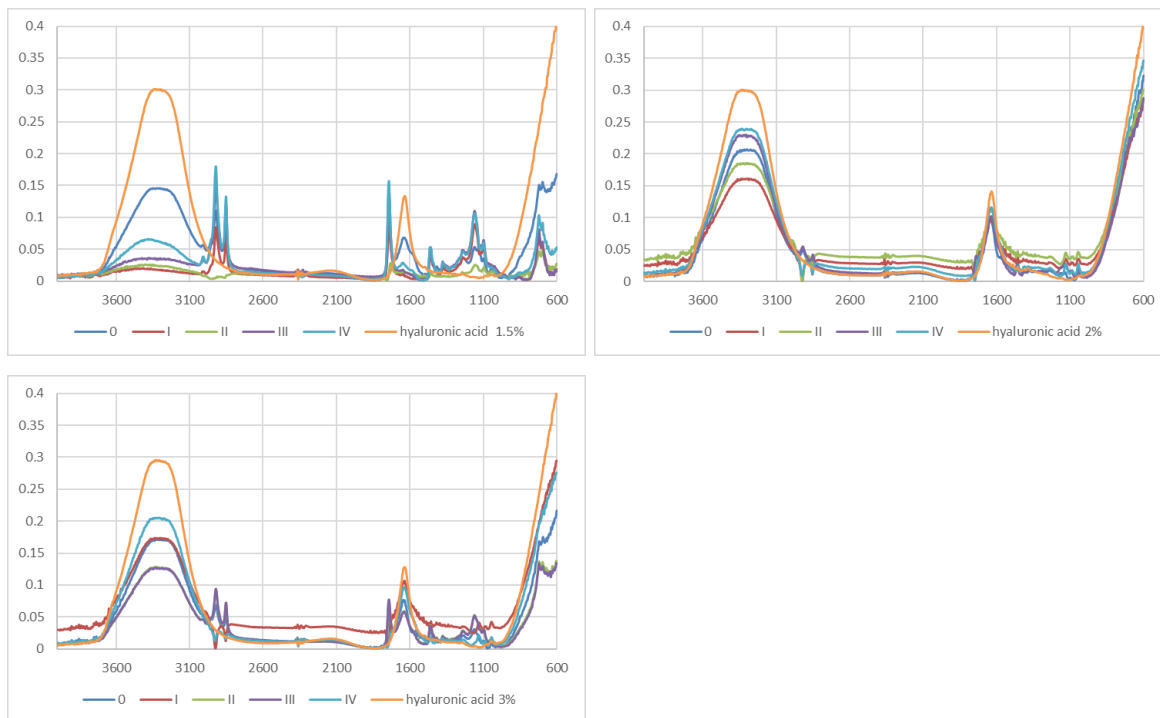


Fig. 3. The IR spectrum of a serum containing 1.5%, 2%, and 3% hyaluronic acid which has been stored at a temperature of 35°C

Source: own study.

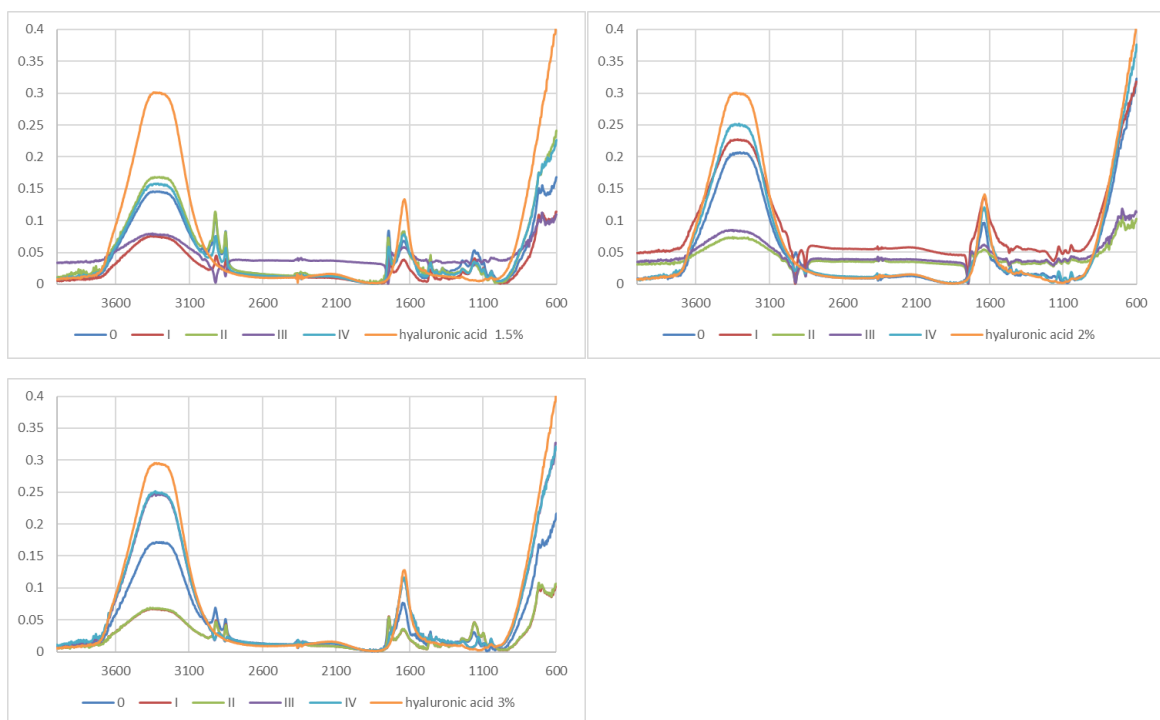


Fig. 4. The IR spectrum of a serum containing 1.5%, 2% and 3% hyaluronic acid stored under different conditions

Source: own study.

Results of measurement of the hydration degree of the corneal layer of the skin and transepidermal water loss from the skin

The degree of epidermal hydration was measured using a corneometer. The measurement was carried out at specific time intervals, i.e. before the serum was applied to the skin and immediately after the serum was applied to the skin, 15min and 30min after application. The test was conducted immediately after the serum was made. All formulations produced had the highest degree of hydration immediately after application, this was because the applied product did not absorb enough, and the more water in the stratum corneum of the skin, the better the current flows, which is equivalent to a high degree of hydration of the epidermis (Figure 5).

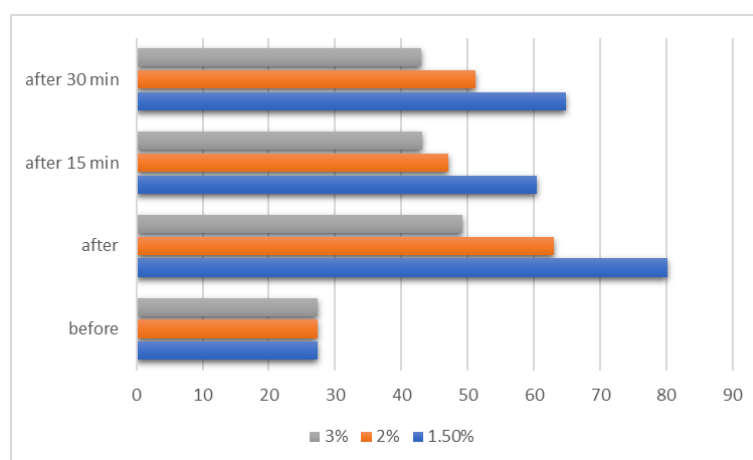


Fig. 5. Results of measurement of the hydration degree of the corneal layer of the skin treated with a serum containing 1.5%, 2% and 3% hyaluronic acid

Source: own study.

A tewameter was used to measure the degree of water loss from the epidermis. The test was carried out simultaneously with the examination of the degree of epidermal hydration, so the course of performing the determination looked the same, and the time intervals between measurements did not change. The results of the degree of water loss from the epidermis are given in units of grams per square meter per hour ($\text{g}/\text{m}^2\text{h}$). The lower the TEWL value, the more water the skin retains and is more hydrated (Figure 6).

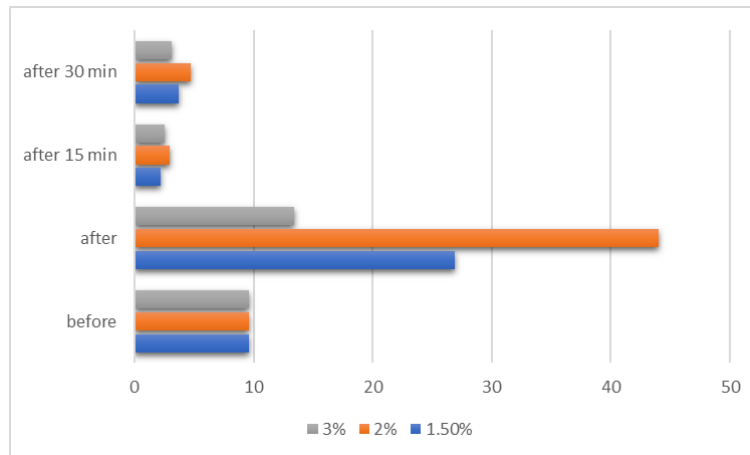


Fig. 6. Results of measurement of the transepidermal water loss from the skin treated with a serum containing 1.5%, 2% and 3% hyaluronic acid

Source: own study.

The degree of water loss from the epidermis 30 minutes after application is greater than before application. In each of the weeks, regardless of the concentration of hyaluronic acid in the serum, the highest degree of water loss from the epidermis is observed immediately after application. These results are related to the absorption of the product, which in turn is related to the rapid evaporation of water from the skin surface.

Conclusions

The tests conducted made it possible to compare and check how different concentrations of the same active substance in a cosmetic product affect its functional properties and stability under varying conditions. The sensory evaluation determined which serum type had the best handling properties and how these changed during the 4-week test cycle under different temperature conditions. The serum with 3% hyaluronic acid had the best properties, both immediately after manufacture and 4 weeks after manufacture. All serums were rated with an average score of 3 (out of a possible 5). The worst-rated parameter was the smell, which became more intense week by week, especially when the products were stored at 35°C. The smell of broccoli oil and lactic acid increased, which caused a very unpleasant sensation. In contrast, parameters such as texture and spreading of the product on the skin scored best. Storing the manufactured products in the refrigerator had the most favourable effect on the functional properties, especially the smell and consistency of the product. All the cheeses produced immediately after manufacture had a very similar pH value, which averaged 4. The pH of the preparations produced is acidic, lower than the natural pH of the skin (about 5–5.5). This pH of the solution indicates its bacteriostatic effect, and with prolonged use, it may also have a weak keratolytic effect, i.e. loosen the keratin connections on the skin enough for the epidermis to exfoliate, and reduce the tendency to form blackheads (Noszczyk, 2012). The different storage condi-

tions of the serums produced had no significant effect on the changes in pH values. The IR spectroscopic study examined how many transformations take place during the 4-week test cycle in each of the serums produced. The spectra obtained during the study do not allow a clear conclusion to be made as to which serum is the least stable product, how the concentration of the active substance influences this stability and where the greatest changes occur.

Regardless of the storage conditions, each serum produced has a beneficial effect on the epidermis by increasing the level of hydration. Closely related to epidermal hydration is transepidermal water loss. The lower the water loss from the epidermis, the better the protective barrier provided by the cosmetic product. Of the three types of serums, the serum with a concentration of 1.5% hyaluronic acid had the best moisturising and epidermal water retention properties.

The cosmetic products produced were characterised by an acidic pH. Cosmetics with such a pH are mainly intended for oily skin. The serum was intended to be a product that mainly moisturises the skin, but not a bacteriostatic or exfoliating product. When refining the formulation, care should be taken to ensure that the pH of the manufactured product is higher, between 4.5 and 5.

Assessing the results obtained, which are very comparable for each type of serum, we can conclude that a higher concentration of a biologically active substance is not a guarantee of higher quality, both in terms of stability under different storage conditions and in terms of functional properties.

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BN-74/6140-08/04 *Cosmetic and perfumery products, Industry standard*

Part III

Consumer perspective & quality issues

UNDERSTANDING THE CONSUMER BEHAVIOUR OF GENERATION X AND Y REGARDING FUNCTIONAL FOODS IN SLOVAKIA

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DOI: 10.56091/CTQS.Qual-22

Abstract

In recent years, a notable shift in dietary preferences and health awareness of consumers has caused a rising interest in functional foods. Functional foods have become a significant worldwide trend in nutrition as consumers attempt to maintain healthier lifestyles and enhance their general well-being. The objective of the study is to evaluate the consumer behaviour of Generation X and Y regarding functional foods in Slovakia. The research is based on primary data obtained from Slovak respondents of Generation X (born between 1965–1980) and Generation Y (born between 1981–1996). In total 159 answers were obtained from participants of Generation X and 133 from Generation Y during the months of April and May 2023 in the Slovak Republic. The main barriers why consumers do not consume functional foods are primarily due to the taste and higher prices compared to conventional food. The most critical factors which would encourage consumers to consume functional food in terms of importance are recommendations from a doctor, improvement of health and prevention of health risks. The new trend of functional foods was evaluated as the least significant determinant for motivating respondents to consume functional foods. Based on the data, the commonly consumed functional foods of Generation X include mainly dairy products with lower fat, sugar content, enriched with vitamins and minerals, protein and energy bars. Functional food consumption preferences differ across generations. Generation Y individuals primarily consume dairy products with probiotic cultures, added calcium, without sugar and low-calorie drinks and juices. The main determinants affecting the purchasing decisions of Generation X are price, previous experience, discount and health claims. On the other hand, Generation Y is mainly influenced by previous experience, food compound and price. In both cases, the least important determinants affecting the food purchases are mainly the design of the packaging, the labels "vegan" and "lactose-free", and the material of the packaging.

Keywords: consumer behaviour, food marketing, functional foods, health trend, purchasing behaviour

Introduction

Expanding consumer interest in health and well-being has an impact on eating patterns and food preferences (Panhal et al., 2018). That being said, food preferences in developed societies have sig-

nificantly changed due to the growing understanding of the strong link between diet and human health. Menrad (2003) reported that demographic and socio-economic trends suggest a need for foods with added health advantages. Functional foods have swiftly entered the market and increased their market share as value-added products in industrialised countries (Niva, 2006). It is reported that the functional foods and beverages market is projected to increase by USD 132.84 billion from 2022 to 2027. In 2022 the global functional food market size was valued at USD 207.7 billion (Technavio. n.d.).

Functional foods impact particular bodily processes and hence provide advantages for health, happiness, and performance in addition to their usual nutritional worth (Villaño et al., 2022). Scrinis (2008) states that functional foods include novel food products (e.g. cholesterol-lowering margarine), nutrient-fortified highly processed foods (e.g. energy drinks), whole foods that have been nutritionally modified (e.g. reduced-fat milk) and unmodified whole foods (e.g. nuts and fruit).

There is some evidence that functional foods are growing in popularity and that the market for them will continue to expand. It makes sense to assume that the industry should truly understand what motivates consumers to choose functional foods, how they defend their decision, and to what extent their interest is connected to their values and fundamental culture in order to maximise the potential in this expanding market (Urala & Lähteenmäki, 2003). As reported by Siro et al. (2008), consumer acceptance is crucial for the development and success of functional foods. Thus, many studies have conducted empirical studies based on primary data gathering to examine consumer attitudes regarding functional foods. It is crucial to consider consumers' knowledge about food and beverages that support health and their steps to change their diets. For functional food market development, it is essential to comprehend customer acceptance. Recognising its determinants is a significant part of market orientation, consumer-led product development, and successfully negotiating market opportunities (Verbeke, 2005). According to several studies, several factors, including product features (such as the ratio of carriers to ingredients, price, taste, brand, and health information), can affect consumers' acceptance of functional foods. Based on the results of a few studies, price plays a significant role in accepting functional food by consumers. Price may therefore have a dual impact on customer acceptance: either a higher price may reduce consumers' buy intentions, or a higher price may enhance consumers' purchase intentions because it may increase the perceived quality of the products (Huang et al., 2019). Price has a considerable adverse impact on consumers' consumption of functional yoghurts, according to Ares et al. (2010a). Similar findings were made by Narayana et al. (2020), who discovered that many Sri Lankan consumers were more focused on the price of functional food than on the health advantages of the product. Besides the price and taste, Miroso & Mangan-Walker (2018) discovered that consumers are more inclined to accept functional foods

if they are familiar with the brand of the product. Moreover, one key factor impacting consumer adoption of functional foods is how health claims are presented on the labels. As a result, some health-related information on food labels may enhance consumers' perceptions of health benefits and favourably affect their acceptance (Marrete et al., 2010). According to González-Díaz et al. (2020), particular that health information (e.g. added functional substances and promotion of human health) may result in greater purchase intentions. To conclude, the success and expansion of the functional food sector depend on customer acceptance of the products. Consumer acceptance levels directly impact demand, sales, and market penetration of functional food products.

Many research studies have been conducted to assess consumer behaviour about functional foods. Few studies have been involved in investigating the attitudes of young and older consumers towards functional foods (Markovina et al., 2011, Rezai et al., 2012, Marina et al., 2014, Annunziata et al., 2015). However, there has been very scant research specifically for understanding consumer behaviour regarding functional foods of different generations. In order to fill the scientific gap, we focus on identifying the products that consumers of Generation X and Y consume the most, the factors influencing the purchase and the motivators of the consumption of functional products. The study's main objective is to identify the consumer behaviour of Generation X and Y towards functional foods in Slovakia. According to Pallister (2023), Generation X is a crucial demographic group for market analysts due to its impact on consumer behaviour and technology adoption trends. Researchers may be able to develop more effective marketing techniques that target this key generation by better understanding current patterns in consumer behaviour. Moreover, LaFleur (2023) states that Generation Y represents the largest generation in history. By analysing the preferences and behaviours of Generation X and Generation Y, businesses can adapt, innovate, and stay competitive in a rapidly changing consumer market. For the purpose of the study we proceed according to the definitions from of Brodahl & Carpenter (2011) and Borges et al. (2006). Generation X represents people born between 1965 and 1980, and Generation Y, people born between 1981 and 1996. When establishing the hypotheses, the research is based on the study by Nguyen et al. (2020), who focused on factors affecting consumers' intentions to purchase functional foods in Vietnam. Moreover, our study analyses the consumer and purchasing behaviour in the functional food market. In summary, investigating purchasing behaviour is essential for businesses, policymakers, and researchers to make informed decisions, improve products and services, and contribute to the overall well-being of individuals and society. The following hypotheses were suggested:

Null hypothesis H_0 : There are no differences in determinants affecting the purchasing decisions across Generation X and Generation Y.

Alternative hypothesis H_1 : There exist differences in determinants affecting the purchasing decisions across Generation X and Generation Y.

The study is structured as follows. The first part is focused on the purpose of the study and relevant literature regarding functional foods. The second section discusses the hypothesis and research framework. The subsequent part presents the findings and analysis, while the paper's final section concludes the study.

Material and methods

The present work is focused on evaluating the consumer behaviour of Generation X and Y regarding functional foods. The partial aim of this study is to find out the main motivations that would encourage consumption and to find out what are barriers to non-consumption. In addition, the study deals with the factors that influence the purchase decision process. Moreover, the study focuses on what types of functional food products consumers of individual generations consume the most. In order to achieve the research objectives, a quantitative survey was conducted.

Study sample

The survey represents a pivotal study and was carried out online using a Google Form platform. The paper is based on the results of a questionnaire research study conducted with the CAWI method. A questionnaire survey was distributed among respondents living in Slovakia. Moreover, the research is focused on Slovak consumers of functional food aged 18 and older. Data were collected from April to May 2023. In total, 327 answers were obtained. However, by applying selective criteria (consumer of functional foods, consumer from Generation X or Y, residence in Slovakia), the final research sample contained 292 participants. In Table 1, the socio-demographic characteristics of the research sample are summarised. Based on the data, the size of both research groups was approximate the same (Generation X – 54% and Generation Y – 46%). The majority of the research sample represented female consumers living in rural areas with secondary education who had monthly income between 1001€ to 1200 €.

Tab. 1. Socio-demographic characteristics of respondents

Variable	Category	n=292	%
Gender	Women	170	58.22%
	Men	122	41.78
Generation	Generation X	159	54.45
	Generation Y	133	45.55
Education	Primary	2	0.68
	Secondary	169	57.88
	University degree	121	41.44
Residence	Urban	136	46.58
	Rural	156	53.42
Monthly income	Up to 800 €	27	9.25
	801 – 1000 €	78	26.71
	1001 – 1200 €	122	41.78
	1201 – 1400€	42	14.38
	More than 1400 €	23	7.88

Source: own processing based on a questionnaire survey.

Statistical analysis

The Friedman test is used to test the hypothesis that the medians of more than two sets are in agreement. A test designed to compare dependent choices. In the calculation, we first sort the row observations in ascending order of size, and then assign the order to R_{ij} . For the purposes of the paper, we apply the Friedman test when determining the existence of statistically significant differences in the evaluation of determinants affecting the purchases in terms of importance. In the end, Nemenyi's method was applied, the purpose of which was to find out which statements have statistically significant differences. It is calculated as follows:

$$F = \left(\frac{12}{n \cdot k \cdot (k+1)} \sum_{j=1}^k R_j^2 \right) - 3 \cdot n \cdot (k + 1)$$

Results and discussion

The first part of the questionnaire contained general questions about functional foods. In the beginning, respondents had to answer a selective question about whether they had encountered the term functional foods. The next section of the questionnaire contained the definition of functional foods together with an open question about what foods they imagined under this term. Respondents who

stated that they did not know functional foods were redirected to the end of the questionnaire survey. In the next part of a questionnaire survey, respondents were asked to answer selective questions about the consumption of functional foods. According to the results displayed in Fig. 1, more than 88% of respondents declared the consumption. Only 12% of individuals declared that they do not consume functional foods.

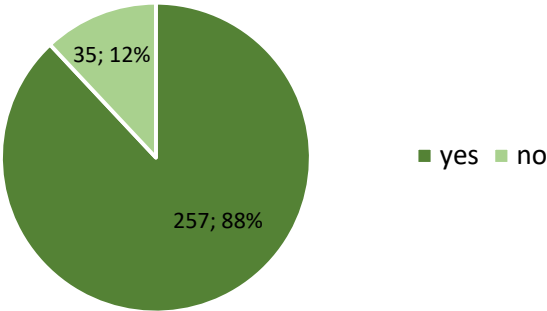


Fig. 1. Consumption of functional foods
Source: own processing based on a questionnaire survey.

Another question (Fig. 2) of the questionnaire survey was focused on the barriers that prevent consumers from not consuming functional foods. The main barriers of not consuming functional foods were primarily due to the taste (40%) and price (37%). Respondents also claimed they did not have enough information about functional foods (23%). Similar results were obtained by Dias et al. (2023), who found that the main barriers of Sri Lankan respondents were the bit awareness about functional foods, taste and price of the food. On the other hand, results by Moutinho et al. (2022) showed that the essential barriers for respondents in Portugal were price and availability of the product, followed by sensory attributes, lack of knowledge of how to prepare the food, and how much to consume. Research done by Chammas et al. (2019) and Vecchio et al. (2016), consumers were willing to pay more for functional products that make health claims. However, our results show that respondents of this study believed that taste and price were one of the main barriers preventing them from consuming functional foods.

Table 2 represents the results of the next question, which dealt with the factors that would encourage respondents to consume functional foods. This question was answered by the respondents who stated in the selective question that they do not consume functional foods. The total number of responses was 35. Using Friedman's test, it is evident that the main determinants that would encourage consumers to consume functional foods were mostly the doctor's recommendation, improving

health and preventing health risks. It was found that statements that functional foods as a new trend and functional foods as alternatives to conventional foods would not motivate respondents to consume functional foods. Our results are in line with Saher et al. (2004), who discovered that health is one of the most commonly given motives for consuming functional foods. Similarly, Kolbina et al. (2020) found that preventing and treating some diseases were essential motivators for purchasing functional foods in Kemerovo. According to Tahergorabi et al. (2015), sensory features (for instance, taste, flavour and texture) and the convenience of use remain very important for consumers. Jain et al. (2014) discovered that factors encouraging people to buy nutritionally enriched food were because of the health for children and to avoid medical treatment.

Tab. 2. The main factors that would encourage respondents to consume functional foods

Sample	Sum of ranks	Mean of ranks	Groups			
New trend	58.500	1.671	A			
Alternative offer to conventional food	107.500	3.071	A	B		
Availability of functional foods in retail stores	141.000	4.029		B	C	
Enrichment of the diet with valuable ingredients	162.000	4.629		B	C	
Improvement of condition	164.000	4.686		B	C	
Preventive nature against health risks	197.000	5.629			C	D
Improving health	197.500	5.643			C	D
Recommendation from a doctor	232.500	6.643				D

n = 35

Source: own processing based on a questionnaire survey.

When discovering which type of functional foods these two generations consume the most in the terms of frequency, the results are as follows: participants from Generation X tend to consume mainly dairy products with lower fat and sugar content and dairy products enriched with vitamins and minerals. Besides dairy products, protein bars, energy bars, low-calorie drinks enriched with vitamins and minerals, juices, and fruit/vegetable juices enriched with vitamins and minerals were also evaluated as the most frequently consumed functional food of Generation X. On the contrary, respondents of Generation Y declared the most frequent consumption of dairy products with probiotic cultures, dairy products with added calcium and dairy products without sugar. Moreover, more than half of the respondents claimed consumption of low-calorie drinks enriched with vitamins and minerals, juices, fruit/vegetable juices enriched with vitamins, minerals and protein chocolates, and creams. A study done in Turkey showed that dairy products were the most frequently consumed foods. In Turkey, dairy products are one of the most significant traditional foods. They are eaten for

breakfast, lunch, and dinner and are frequently the main dish ingredient (Gok & Ulu, 2018). Our results show that consumers from both generations tend to drink juices and drinks enriched with vitamins and minerals. Consumption of fruit juices was examined in many researches. Nearly 50% of Swedish respondents reported drinking juice with additional vitamins or minerals (Landström et al., 2007). Probiotic fruit drinks were another popular functional food among Swedish respondents. Juices with added vitamins were not widely drunk in Italy; only 7% of respondents declared the consumption (Annunziata & Vecchio, 2005). In contrast, nearly 42% of respondents in Spain reported drinking juices and other enriched beverages (Núñez-González et al., 2011). Concerning dairy products, probiotic milk products were widely consumed (60%) in Sweden. However, yoghurt with muesli was only consumed by 7% of respondents (Landström et al., 2007). According to De Jong et al. (2003), 32% of Dutch respondents drank yoghurt enriched with lactic acid bacteria. Poland consumers mostly consumed probiotic yoghurt drinks (Wadolowska et al., 2009).

Tab. 3. Frequency of consumption of functional foods across generations

Consumption frequency	Generation X		Generation Y	
	Frequency	Frequency in %	Frequency	Frequency in %
Daily	37	23.27	71	53.38
Several times a week	73	45.91	40	30.08
Several times a month	38	23.89	17	12.78
Occasionally	11	6.92	5	3.75

Source: own processing based on a questionnaire survey.

The results of the Table 3 illustrate the differences in the consumption of functional foods between the generations. According to the data, consumers of Generation Y tend to consume functional foods daily (53%) and several times a week (30%). On the other hand, respondents of Generation X mainly consume functional foods several times a week (46%) and several times a month (24%). Many studies deal with consumer behaviour in the functional food market, but none have investigated the behaviour of individual generations. With respect to purchasing frequency, it is evident that 34% of participants of Generation X tend to purchase functional foods once a week, and 25% of participants tend to purchase functional foods once a month. Generation Y purchased functional foods several times a week (35%) and once weekly (24%). Results of Annunziata & Vecchio (2011) discovered that consumers in Italy consume functional foods occasionally (28%), and 15% of respondents were daily consumers. Moreover, 21% of respondents never consumed functional foods. Annunziata et al. (2015) investigated the consumer behaviour of older consumers (60+) in Italy. Based on the results, 42% of respondents were occasional consumers. Daily consumption declared that 18% of respond-

ents and 18% of consumers have never consumed functional foods. Brečić et al. (2014) discovered that 17% of Croatian respondents consume functional foods once a week, and a similar percentage fall into the once-per-day category. However, 16% of consumers declared they had never consumed functional foods. In a study by Hassan et al. (2020), 51% of Malaysian participants consumed functional food more than seven times; 26% of participants consumed functional food between six and seven times per week, and only 4% of individuals consumed functional food either just once a week or not at all.

Using a 5-point Likert scale with a range of 1 to 5, where 1 indicated the least important element and 5 the most significant factor, this study analysed specific factors that selected generations taken into account while purchasing functional foods. The hypothesis, H₁, presupposed statistically significant variations in the assessments of the chosen components. We determined Friedman's test (Tables 4, 5, 6) and then applied Nemenyi's method. Our findings show that statistically significant differences occurred between selected factors (p-value = <0.001), and those across Generation X and Y differ.

Tab. 4. Results of Friedman's test

Parameters	Generation X	Generation Y
Q (Observed value)	737.979	458.031
Q (Critical value)	27.587	27.587
DF	17	17
p-value (one-tailed)	<0.0001	
Alpha	0.05	

Source: own processing based on a questionnaire survey.

According to the results, the most critical factor influencing the purchasing decisions of Generation X was price, followed by previous experience and discount (Tab. 5). The packaging design, together with the labels Vegan, Lactose-free, Organic, Gluten Free and the packaging material, were the least important determinants that affect the purchasing decisions of Generation X. These results can be explained by Ordun (2015), who declared that Generation X is most likely to look for the lowest cost item or discount rather than thinking of the investment value of purchases. Moreover, one of the reasons why Generation X mainly consider price is the high inflation in Slovakia. For 2022, it was one of the highest in the EU. When comparing, slight differences occurred between the generations. Considering Generation Y, the results showed that the most significant determinant affecting purchases is the previous experience (Tab. 6). Moreover, other crucial determinants influencing consumers from Generation Y were the food composition, and price, followed by health claim and country of origin. Contrarily, the least significant factors were the label Lactose-Free, the packaging design,

the label Vegan and the packaging material. These results are in line with Ordun (2015). Consumers of Generation Y are described as buyers who select and consume products that help them to define who they are, what is important to them and what they value in life also express some aspect of their own personality or image. They use their considerable knowledge of the latest trends, images, and products. Harrington et al. (2012) state that Generation Y has a strong relationship to experiences.

Tab. 5. Factors affecting the purchasing decision of Generation X

Sample	Sum of ranks	Mean of ranks	Groups																	
Design of the package	1007.500	6.336	A																	
Label "Vegan"	1020.500	6.418	A																	
Label "Lactose-Free"	1072.000	6.742	A	B																
Label "Organic"	1115.500	7.016	A	B	C															
Label "Gluten free"	1196.500	7.525	A	B	C	D														
Material of the packaging	1229.500	7.733	A	B	C	D														
Place of the purchase	1294.500	8.142	A	B	C	D														
Appearance	1403.000	8.824		B	C	D	E													
Promotion	1409.500	8.865			C	D	E													
Recyclable packaging	1432.500	9.009			C	D	E	F												
Country of origin	1527.500	9.607				D	E	F												
Brand	1531.000	9.629				D	E	F												
Size/weight	1636.000	10.289					E	F	G											
Compound	1761.000	11.075						F	G	H										
Health claim	1952.500	12.280							G	H	I									
Discount	2038.500	12.821								H	I	J								
Previous experience	2250.500	14.154										I	J							
Price	2311.000	14.535																		J

n = 159

Source: own processing based on a questionnaire survey.

According to our results displayed in Tables 5 and 6, one of the important determinants is the price in both generations when purchasing functional foods. The results of Dolgoplova et al. (2015) show that the higher price may decrease consumers' purchase intention; it can be a source of distrust, lowering consumers' acceptance in Germany. However, consumers are prepared to pay more for food when they believe it has certain qualities, such as safety and healthfulness, confirmed in research by Ares et al. (2010b), Moro et al. (2015) and Pappalardo & Lusk (2016). However, when consumers perceive the food values such as safety and healthiness, they are willing to pay an extra price

(Moro et al., 2015; Pappalardo & Lusk, 2016). The results of Dias et al. (2023) show that consumers in Sri Lanka mainly consider the nutrition composition of functional food, taste, price and health aspects while purchasing. However, the convenience of preparing the food was evaluated as the least essential determinant. Jain et al. (2014) discovered that taste and nutrition were the most significant determinants of purchasing functional foods. On the other hand, convenience and price were not significant at all while purchasing. For young consumers in Croatia, the most important functional food attributes were evaluated as taste and price (Markovina et al., 2011). According to Williams & Gosh (2008), health claims significantly guide purchasing decisions for functional foods.

Tab. 6. Factors affecting the purchasing decision of Generation Y

Sample	Sum of ranks	Mean of ranks	Groups											
Label "Lactose-Free"	863.000	6.489	A											
Design of the package	880.500	6.620	A	B										
Label "Vegan"	920.000	6.917	A	B										
Material of package	983.000	7.391	A	B	C									
Label "Gluten Free"	1059.500	7.966	A	B	C	D								
Promotion	1091.000	8.203	A	B	C	D	E							
Label "Organic"	1149.000	8.639	A	B	C	D	E	F						
Place of purchase	1155.000	8.684	A	B	C	D	E	F						
Appearance	1183.000	8.895		B	C	D	E	F						
Size/weight	1230.000	9,248			C	D	E	F	G					
Brand	1301.500	9.786				D	E	F	G	H				
Recyclable package	1373.500	10.327					E	F	G	H				
Discount	1385.000	10.414					E	F	G	H				
Country of the origin	1439.500	10.823						F	G	H	I			
Health claim	1513.000	11.376							G	H	I			
Price	1560.500	11.733								H	I			
Compound	1708.000	12.842										I	J	
Previous experience	1948.000	14.647												J

n = 133

Source: own processing based on a questionnaire survey.

A study by Szakály et al. (2019) examined consumers' willingness to pay for functional foods. According to the results, subjective consumer knowledge and consumer background (education and subject-

tive income level) strongly positively affect purchase patterns and beliefs about functional foods' attributes and nutrition and health. Moreover, a significant positive relationship was demonstrated between the consumer's health history and beliefs about the relationship between nutrition and health. The willingness to purchase is influenced by the lifestyle of the consumer (Goetzke & Spiller, 2014), the sensory (Kraus, 2015) and non-sensory features of the product, such as its price (Romano et al., 2015), the benefits of the product (Rezai et al., 2017).

Conclusions

Functional foods have gained significant attention in an era where consumers are increasingly conscious about their health and seeking proactive ways to enhance their well-being. The primary objective of this paper was to identify the consumer behaviour towards functional foods of Generation X and Y in Slovakia. The research was based on primary data obtained from Slovak respondents of Generation X (born between 1965–1980) and Generation Y (born between 1981–1996). When verifying the statistical hypothesis, we accepted hypothesis H1, which represented statistically significant differences that respondents of both generations consider when purchasing functional foods. Friedman's test was applied to find out what factors influence Generation X and Generation Y when buying functional foods.

The taste and price of functional foods are the main reasons why respondents do not consume them. According to the findings, doctor's advice, improved health, and risk prevention are the three most important variables that motivate consumers to eat functional foods. Based on the data, dairy products with lower fat and sugar content, enhanced with vitamins and minerals, protein and energy bars, were the functional foods that Generation X respondents consume most frequently. The primary factors influencing Generation X's purchase choices were the price, previous experience, discounts, and health claims. On the other hand, Generation Y consumed mostly dairy products with probiotic cultures, added calcium, sugar-free varieties, and low-calorie drinks and juices. Moreover, Generation Y was heavily influenced by previous experience, food composition, and price. In both situations, the packaging's design, "vegan" and "lactose-free" labels, and material had the least impact on food purchases.

The outcomes presented can serve as a foundation for professionals and researchers in the public health domain, as well as for food businesses engaged in the production of value-added food products. The findings of the study have implications for producers of functional food. One of the barriers of non-consumption was the lack of information about the products. Awareness among consumers can be fostered and elevated through the utilization of diverse marketing strategies employed by manufacturers and distributors. Additionally, state and public institutions can implement various

information campaigns, and there can be an emphasis on consumer education starting from a young age for children. Moreover, these findings enrich the extant literature and improve the understanding of how different factors affect consumer attitudes and intentions towards purchasing functional products in Slovakia. Finally, the study has limitations. The sample chosen for the research is not representative and for future research, it would be appropriate to focus on all generations, not just Generations X and Y.

Acknowledgements

This paper was supported by the project VEGA, "Implementation of the New EU Food Strategy in the Food Chain in Slovakia." Project registration number VEGA No. 1/0245/21.

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THE INFLUENCE OF BRAND INFORMATION ON MILK CHOCOLATE ACCEPTANCE

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DOI: 10.56091/CTQS.Qual-23

Abstract

The aim of the study was to examine how brand information affects consumers' perceptions of milk chocolates, with a focus on both sensory and visual attributes. A two-stage sensory evaluation study was conducted with private label and leading market brand milk chocolates. The first stage aimed at understanding the desirable quality attributes of chocolates and assessing the acceptance levels concerning the optimally desired intensity of specific features. The second stage considered the effect of brand familiarity and packaging attractiveness on overall desirability. Overall, there were no significant differences between the blind liking scores (color, odor, taste and melt in the mouth) of private label and manufacturer brands of milk chocolates. However, brand information and familiarization with the samples packaging resulted in changes in the overall liking of chocolates.

Keywords: acceptance, brand, chocolate, packaging, private label, sensory quality

Introduction

Milk chocolate is defined as a solid chocolate product that includes, apart from cocoa and cocoa butter, milk or milk-derived ingredients and sugar. The creamy texture and sweet taste of milk chocolate are due to the addition of these milk and sugar components. Depending on the region, regulations might stipulate the minimum content of cocoa solids in milk chocolate. For instance, in the European Union, the minimum cocoa solid content in milk chocolate is defined as 25% (Directive 2000/36/EC, Husain et al., 2018).

Branding, as a term, encompasses much more than just a name or logo associated with a product. It represents a collection of perceptions, emotions, and experiences in the consumer's mind that differentiates a product or service from its competitors (Kotler & Keller, 2019). These perceptions, both cognitive and emotional, drive consumer behavior, influencing their buying decisions and fostering loyalty towards certain brands (López-Menchero & de Miguel, 2015; Puška et al., 2018). The strategic importance of branding lies in its capacity to convey value, quality, and trust to consumers. It can effectively communicate a product's unique attributes, making it stand out in a crowded mar-

ket space. It is through this process that brands can establish a meaningful and durable relationship with their consumers (Keller, 2011; Kotler & Keller, 2019). Brand information, therefore, becomes a critical factor shaping consumer perception and influencing purchasing decisions. This information can be conveyed in various ways – through the product itself, its packaging, promotional activities, or even word-of-mouth (López-Menchero & de Miguel, 2015). In the context of the chocolate market, brand information becomes particularly relevant. Given the wide array of choices – spanning different flavors, cocoa content, fillings, and price points – consumers rely heavily on brand information to guide their decisions. Factors such as brand heritage, perceived quality, ethical sourcing, and price play a vital role in shaping consumer acceptability and preference for chocolate brands (Jaeger, 2006; Poelmans & Rousseau, 2016).

Private labels (PLs), also referred to as store brands, own brands, or retailer brands, are products that are manufactured or provided by one company but are sold under another company's brand, usually under the brand of a retailer (Keller, 2011; Kotler & Keller, 2019). PLs, as an evolving force in the retail market, have changed the way consumers perceive and interact with brands. Traditionally, private labels were associated with lower prices and perceived as lower quality alternatives to manufacturer brands. However, this perception has changed significantly over the years. Today, many PLs not only offer competitive prices but also maintain a level of quality that competes with manufacturer brands, leading to increased consumer acceptance and preference for these brands (López-Menchero & de Miguel, 2015; Kiss 2022, Taranko, 2016; Tores-Moreno et al., 2012). In contrast, manufacturer brands in the chocolate industry leverage their longstanding heritage, reputation, and perceived quality to connect with consumers. They build on the trust established over years, offering a sense of reliability and familiarity to consumers (López-Menchero & de Miguel, 2015).

Poland, in particular, has seen a growing interest in milk chocolate, which is reflected in the increasing market share held by both manufacturer and private label brands. Polish consumers exhibit a range of attitudes towards these brands, with an apparent division between those who prefer manufacturer brands for their perceived superior quality, and those who choose private label for their attractive price-quality ratio (Taranko, 2016).

Consumer acceptability of a product, particularly in the food sector, is a multidimensional concept that extends beyond the basic functional utility of the product. In the case of chocolate, sensory attributes such as taste, texture, odor, and appearance play a critical role in shaping consumer acceptability. They not only influence the immediate sensory experience but also interact with brand and product information to affect consumer perceptions, preferences, and ultimately their purchase de-

cisions (Ahmed et al. 2012; da Rosa et al. 2019; Del Prete & Samoggia, 2020; De Pelsmaecker & Gellynck, 2015; Kim et al., 2013; López-Menchero & de Miguel, 2015; Torres-Moreno et al., 2012).

Packaging also plays a meaningful role as it not only provides necessary product protection and information but also acts as a communication tool that influences consumer perception and purchase decisions (da Rosa et al. 2019; del Prete et al., 2020; Kim, 2013, Kovač et al. 2019). Attractive and well-designed packaging can enhance product visibility, stimulate consumer interest, and induce positive emotional responses (López-Menchero & de Miguel, 2015; Spinelli et al., 2015). Packaging design can significantly influence consumer preference and taste associations (da Rosa et al., 2019), further highlighting its role in chocolate acceptability. Thus, it can ultimately influence consumers' brand choices and purchase decisions (del Prete & Samoggia, 2020; López-Menchero & de Miguel, 2015).

The aim of the study was to examine how consumers perceive the private label brands of milk chocolate from selected retail chains compared to the leading brand in the market. The study was conducted in two stages. In the first stage, sensory evaluations were conducted to assess liking and acceptance of quality attributes in relation to their optimally desired intensity. The second stage of the research investigated the influence of brand familiarity and packaging on desirability. The findings from this stage provide a nuanced understanding of the role of extrinsic cues in shaping consumer perceptions, and the differential impact it can have on private label versus leading brands.

Materials and methods

The subject of the research were private label milk chocolates: Biedronka's store brand (SB_1), Dino's store brand (SB_2), and market leading brand: manufacturer's brand (MB). The evaluation of chocolates' samples was performed within the declared shelf-life period. Testing was carried out in a sensory laboratory at the Poznan University of Economics and Business, in accordance with ISO 8589:2007 requirements. A total of 66 untrained consumers took part in this study (73% female) with the participants' age ranging from 16 to 25 years old. All participants self-reported consumption of milk chocolate at least once a month.

The study was conducted in two stages. The first stage aimed to:

- assess the liking of the quality attributes of chocolates (9-point hedonic scale anchored on 1 = dislike extremely to 9 = like extremely for the liking attributes),
- evaluate the level of acceptance of quality attributes in relation to the optimally desired intensity of a given attributes (5-point Just About Right (JAR) scale anchored on 1 = too light, 3= just

right, 5 = too dark (color); 1 = not enough, 3 = just about right, and 5 = too much (other sensory attributes).

Chocolate samples, labeled with a three-digit code, were presented to the consumers on paper plates. Brand logos have been removed from chocolate samples. In this way, potential bias was minimized, allowing a more authentic assessment of the intrinsic qualities of the products. Consumers were provided with spring water.

In the second stage of the research, consumers were introduced to the brand names of the chocolates under study and were given photos of the packaging. The scope of the research included:

- assessing the legibility, graphic appeal, standing out on the shelf and encouraging purchase of chocolate packaging (5-point Likert scale ranging from 1 = definitely not to 5 = definitely yes,
- determining the influence of brand and packaging recognition on the overall liking (9-point hedonic scale anchored on 1 = dislike extremely to 9 = like extremely) (Samotyja, Sielicka-Różyńska, & Klimczak, 2020).

In addition, consumers were asked questions about their willingness to purchase the chocolates under evaluation (5-point scale ranging from 1 = I would definitely not buy this product to 5 = I would definitely buy this product).

Statistical analysis

Statistical analyses were performed using XLSTAT 2023.1.6. program. An analysis of variance (ANOVA) was employed to compare mean values. To verify the significance of differences between mean values, the Tukey HSD post-hoc test or Student's t-test was used. The critical significance level was $\alpha = 0.05$.

Results and discussion

Consumer liking

In the first stage, sensory evaluations of chocolates were carried out in terms of liking and acceptance of quality attributes in relation to their optimally desired intensity. The consumer sensory evaluation included such characteristic qualities of chocolates as color, odor, taste and melt in the mouth. The results are shown in Figures 1–2.

The desirability of the color for the examined chocolates was similar, with a score of around 6.0 (Figure 1). Over 50% of the consumers felt that the color intensity of the MB and SB_1 chocolates

was appropriate. Conversely, 49% of the consumers felt that the color of the SB_2 chocolate was too light (Figure 2A).

In evaluating the odor, no statistically significant differences were observed between the samples (Figure 1). Participants preferred the odor of the MB chocolate, with 49% characterizing its scent as "just right". An equivalent percentage of consumers felt that the SB_2 odor lacked sufficient intensity (Figure 2B).

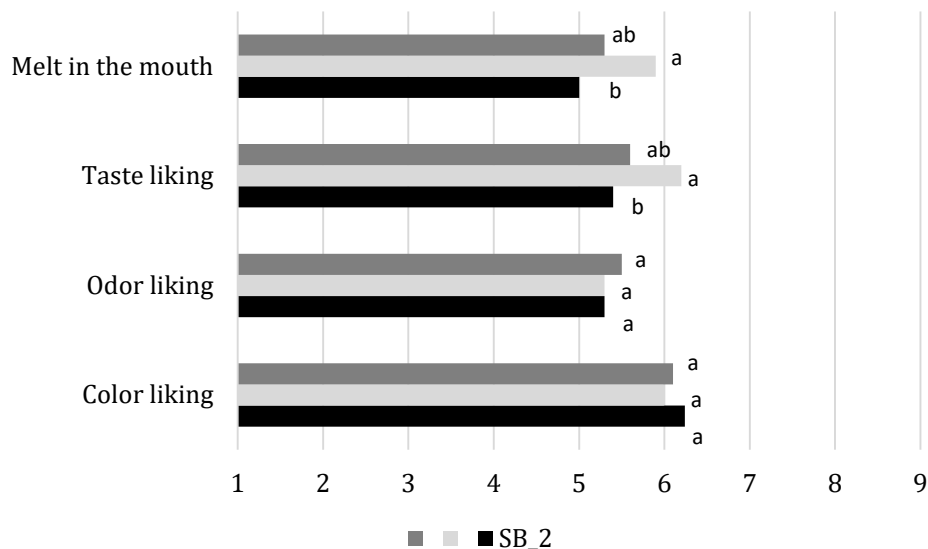


Fig. 1. Consumer liking of milk chocolates

Explanation: MB – milk chocolate of manufacture brand, SB_1 – milk chocolate of store brand (Biedronka), SB_2 – milk chocolate of store brand (Dino)

a–b – mean values, within a sensory attribute, marked with different letters, differ statistically significantly at the significance level of $\alpha=0.05$ (Tukey test, $p<0.05$).

Source: own study.

Of the three chocolates evaluated, the private label chocolate (SB_1) received the highest taste appreciation, scored 6.2 (Figure 1). 52% of consumers indicated that the sweetness intensity of this chocolate was optimal. However, for the SB_2 sample, 43% of the consumers felt its taste was insufficiently sweet (Figure 2C). Taste is the crucial factor when buying and consuming chocolate behaviour (Ahmed et al., 2012; Chawla & Sondhi, 2016; Del Prete & Samoggia, 2020; Miśniakiewicz, 2018). According to Miśniakiewicz (2018) 76% of Polish adults considered the taste of confectionery products and their sensory attractiveness a very important factor when choosing chocolate. Merlino et al. (2021) found that women are more inclined to sweeter chocolates than men whose preference is related to strong and bitter taste.

Regarding the melt in the mouth characteristic, the SB_1 sample obtained the highest score of 5.9 (Figure 1). Both the SB_2 and MB chocolates were perceived as lacking in their melt in the mouth quality (Figure 2D)

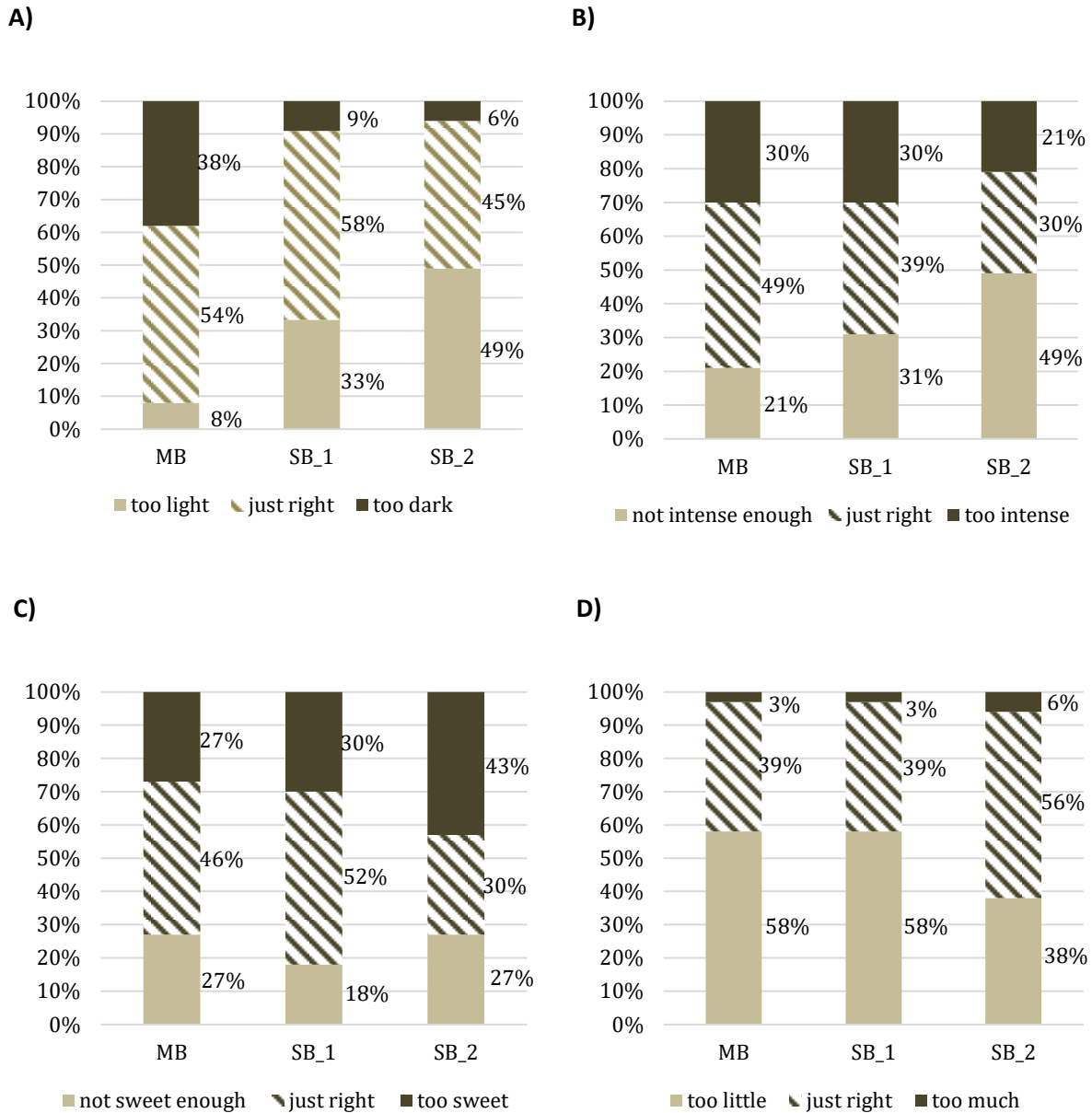


Fig. 2. Intensity of selected sensory characteristics of milk chocolates

Explanation: MB, SB_1, SB_2 – as under Figure 1; A) color, B) odor, c) taste, D) melt in the mouth

Source: own study.

Overall, there were no significant differences between the blind liking of store brands and manufacturer brands of milk chocolates.

Consumers' overall liking under blind and informed conditions

The overall liking assessment of the examined milk chocolates was carried out in two stages. Initially, evaluations were based on coded samples (blind test), followed by providing information about the brand name and presenting images of the chocolate packaging (informed test). In blind conditions, consumers are unaware of the brand, allowing for a purely sensory-based evaluation. This approach eliminates brand biases and offers a clearer picture of the product's intrinsic qualities (López-Menchero & de Miguel, 2015).

The results from both tests are compiled in Figure 3. A 2-way ANOVA was conducted to determine the effect of brand and sample treatment (test blind – test informed) on overall acceptance of milk chocolates. Among the evaluated coded chocolate samples (blind test), the SB_1 chocolate slightly stood out with a higher liking, scoring 6.2 ($p>0.05$). Brand information and familiarization with the samples packaging resulted in changes in the overall liking of chocolates, but the differences were not statistically significant in every case. For private label chocolates, SB_1 and SB_2, participants assigned them lower scores compared to the coded samples (differences were significant only for the SB_2). In the case of manufacturer's brand chocolate, degree of acceptance sample was slightly higher, but differences were not statistically significant. The significant interaction between treatment (blind-informed) and brand or packaging was observed for the overall liking of chocolates ($p<0.05$).

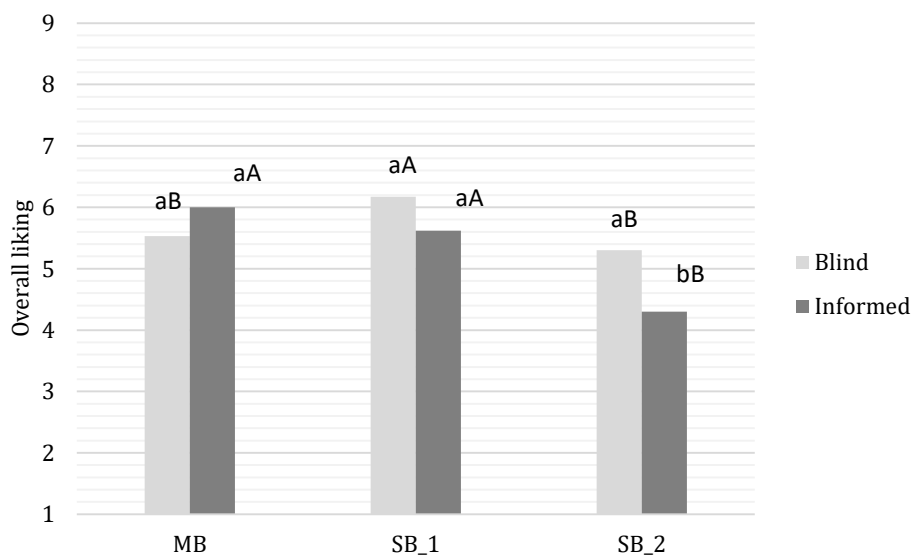


Fig. 3. Overall liking of milk chocolates under blind and informed test conditions

Explanation: MB, SB_1, SB_2 – as under Figure 1;

a–b – mean values, within the chocolate brand, marked with different letters, differ statistically significantly at the significance level of $\alpha=0.05$ (t-Student test, $p<0.05$)

A–B – mean values, within the test (blind-informed), marked with different letters, differ statistically significantly at the significance level of $\alpha=0.05$.

Source: own study.

When consumers are unfamiliar with a product, their purchasing decisions often hinge on external factors such as packaging, brand name, and price (Ozretic-Dosen et al., 2007; Puška et al., 2018, Torres-Moreno et al., 2018). Torres-Moreno et al. (2012) assessed consumer preferences for six dark chocolates under three conditions (blind, expected, and informed). Their study found that brand expectations majorly influenced liking scores. Store brand chocolates created lower expectations, but when they were tasted they were as acceptable as premium chocolates.

Several studies have analyzed the impact of sensory attributes on brand perception and acceptability of product (De Pelsmaeker & Gellynck, 2015; López-Menchero & de Miguel, 2015; Varela et al., 2010). De Pelsmaeker and Gellynck (2015) conducted that highlighted the role of sensory analysis in product development, using the case study of European filled chocolates. Their findings suggest that combining brand information with sensory quality greatly affects consumer preferences. Interesting results were revealed by Kim's research (2013) on the impact of information about the brand and packaging on consumer acceptance of milk chocolates. Brand and declared fat content influenced overall liking however brand name was less important for purchase intent of chocolate milk than fat or sugar content.

Evaluation of packaging

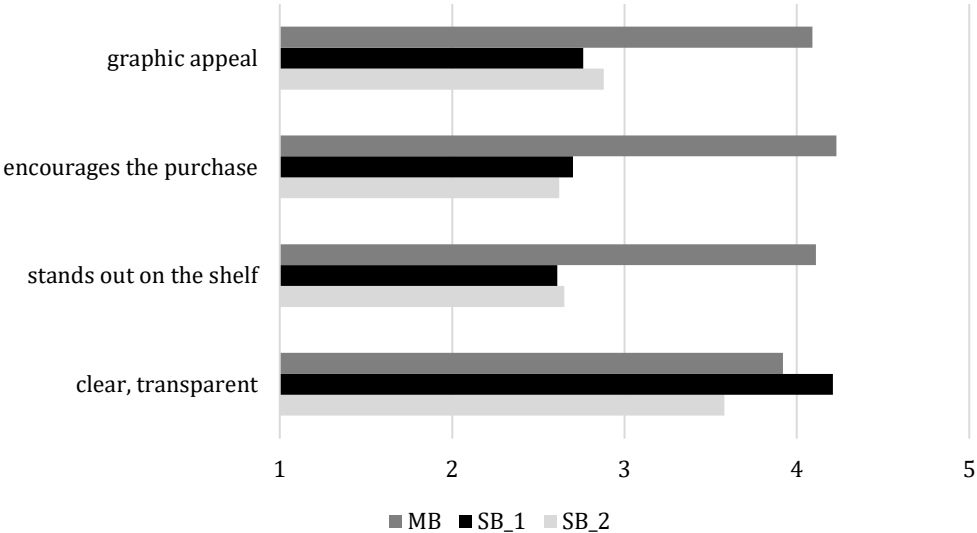


Fig. 4. Evaluation of milk chocolate packaging

Explanation: MB, SB_1, SB_2 – as under Figure 1

Source: own study.

Subsequently, consumers were asked questions regarding the packaging of the studied milk chocolates. These questions focused on their legibility, graphic appeal, standing out on the shelf and en-

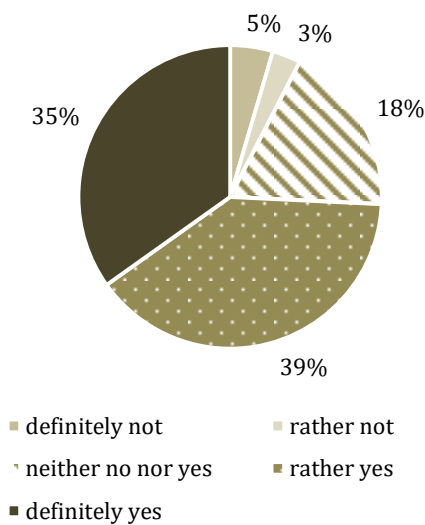
couraging purchase. The packaging of the SB_1 (Biedronka’s store brand) chocolate proved to be the most legible, scoring 4.2 on a 5-point scale (Figure 4). The manufacturer's brand chocolate (MB), compared to the private label chocolates, received higher ratings in terms of graphic appeal, standing out on the shelf and encouraging purchase.

Purchase intention

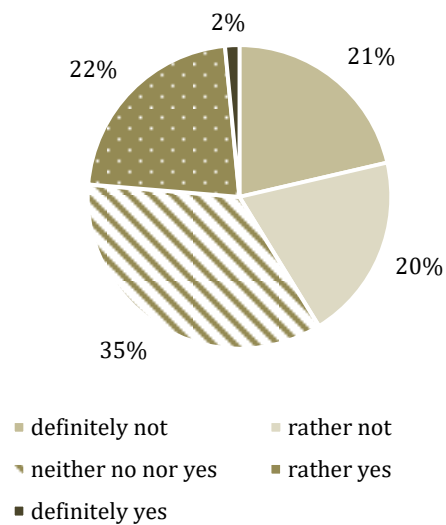
In the subsequent part of the study, consumers expressed their purchase intent for specific chocolates. 74% of consumers declared an intent to purchase the manufacturer's brand chocolate (MB, Figure 5A). For the Biedronka's store brand chocolate (SB_1), only 24% of consumers expressed a desire to buy it, while 41% showed no interest in purchasing (Figure 5B). Similar results were observed for the Dino’s store brand chocolate (Figure 5C).

Consumers' product purchasing behavior is a complex psychological, sensory and market process. Sensory perception of products has been recognized as a major determinant of consumer choice and purchase decisions (del Prete & Samoggia, 2020; Rosas-Nexticapa & O’Mahony, 2005). Several studies highlight the importance of branding in chocolate purchases (Ahmed et al., 2012; Chawla & Sondhi, 2016; Miśniakiewicz, 2018; Ozretic-Dosen et al., 2007). However, few have compared private label chocolates to those of manufacturer brands (De Pelsmaeker et al., 2015; Kiss 2022; Lybeck et al., 2006; Taranko, 2016). Findings from De Pelsmaeker et al. (2015), Kiss (2022) and Taranko (2016) suggest consumers generally prefer manufacturer-branded chocolates, seeing them as higher quality.

A)



B)



c)

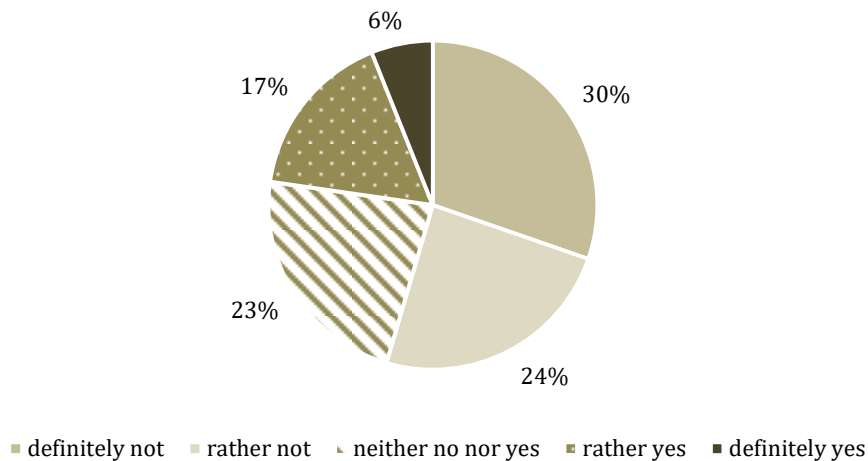


Fig. 5. Purchase intention of milk chocolates

Explanation: A) MB, B) SB_1, C) SB_2 – as under Figure 1

Source: own study.

Conclusions

This study investigated the effects of brand and packaging on consumer choices of milk chocolates. A limited number of samples were evaluated, which is a limitation of this study. For full conclusions, the study should be carried out on a larger number of samples. The results provide understanding of the role of extrinsic cues in shaping consumer perceptions, and the differential impact it can have on private label versus leading brands.

Overall, there were no significant differences between the blind liking scores (color, odor, taste and melt in the mouth) of private label and manufacturer brands of milk chocolates. Brand information and familiarization with the samples packaging resulted in changes in the overall liking of chocolates.

Private label chocolates scored lower when evaluated in non-blind test, compare to blind test.

It was found that the problem with private label chocolates is not the quality of the product itself, its color, taste or odor. The essence of the problem appears to be the packaging of these products, which, with its appearance, does not attract attention, hence consumers often choose leading brand products following the principle the more known the brand, the higher the quality.

So, studying how brand information affects chocolate choices it seems to be an interesting area for more research.

Acknowledgments

The author would like to thank Millano Group for free product support and Mr Michał Bialik for his help with the research.

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ORGANICALLY FARMED YERBA MATE PERCEIVED BY A SELECTED GROUP OF CONSUMERS

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DOI: 10.56091/CTQS.Qual-24

Abstract

A growing consumer interest in organic food is currently being observed, which is defined in Regulation (EU) 2018/848 of the European Parliament and of the Council of 30 May 2018 as a product developed without the use of artificial fertilisers and pesticides. The provisions of this regulation apply to food produced within the European Union as well as products imported into the EU market from third countries, such as Yerba Mate. Consumers' interest in organic products is offset by an increase in their awareness of sustainability principles as well as the perception that organic products are safer for their health.

Over the last years, a significant increase in Yerba Mate imports and increasing availability of organic Yerba Mate has been noticeable. The aim of this study is to assess consumers' perceptions of organically grown Yerba Mate and to identify the factors, both positive and negative, conditioning the purchase of this product group. A questionnaire survey is selected as the research method. The choice of the research group is purposefully non-random and the group of respondents consisted of Yerba Mate consumers associated in a group of lovers of this product on the social network platform Facebook.

The article presents the requirements for the production of organic Yerba Mate and the benefits, according to consumers, associated with the consumption of organic Yerba Mate. The results obtained show that the main determinants of choosing organic Yerba Mate are the absence of chemical contaminants and higher taste qualities compared to conventional products.

Keywords: consumer attitudes, organic products, organic Yerba Mate

Introduction

In the activities of modern companies, the concept of sustainable development which is intended to enable society's needs to be met while respecting the environment is becoming increasingly important. There is also a noticeable growth in interest among consumers in organically grown products which is part of the general trend towards the greening of consumption (Głodowska & Gałązka, 2017; Mazurek-Łopacińska et al., 2022). Organic food is perceived by consumers as being

safer, as well as having a high nutritional value (Trokhymchuk, Klima, & Stańczyk, 2022). Consumers play an important role in protecting the environment through their choices when buying products (Dinu, Schileru, & Atanase, 2012). In response to the needs of today's consumers, which are determined by growing environmental awareness and the desire for healthy eating, a vast number of entrepreneurs, growers and farmers are opting for organic production. Organic production is a type of production that is carried out in accordance with the legislation on organic farming. The purpose of these regulations is to promote environmental protection, to maintain European biodiversity and to embed consumer confidence in organic products. Organic products available on the European market must be labelled accordingly so that conscious consumers can easily identify such products when shopping (Komisja Europejska, 2023; Kłobukowski et al., 2016).

European consumers have progressively easier access to domestic organic products but are also interested in organic commodities from developing countries, such as: Brazil, Argentina or Paraguay. Among the products from developing countries that are of increasing interest to European, including Polish, consumers is, undoubtedly, Yerba Mate (OEC, 2023). The growing popularity of this product from South America has resulted in a noticeable increase in its range over the last few years, both so-called pure Yerba Mate without additives *despalada* (without sticks) and *elaborada* (with sticks), as well as a wide range of products with added herbs or fruit. In addition, new products, such as: Yerba Mate *chimarraro* (dried powdered), *terere* (cold-prepared) and *tostado* (roasted) are appearing on the Polish market. The Yerba Mate range available on the Polish market also includes a rising number of organic products.

Given the growing interest in organic products and the noticeable expansion in the consumption of Yerba Mate in Poland, the aim of this study is to assess the attitudes of Polish consumers towards organically grown Yerba Mate and to identify factors, both positive and negative, conditioning the purchase of this group of products.

Requirements for organic Yerba Mate products available on the European market

The fundamental document outlining the requirements for organic products is Regulation (EU) 2018/848 of the European Parliament and of the Council of 30 May 2018 on organic production and labelling of organic products and repealing Council Regulation (EC) No 834/2007. These provisions apply to the products listed in Appendix 1 of the Regulation, including Yerba Mate, listed as Paraguayan tea.

Yerba Mate – *Ilex paraguariensis* A. St.-Hil. (Aquifoliaceae) is a plant native to South America. It is a medium-sized tree species native to a moderately large region covering eastern Paraguay,

north-eastern Argentina and southern Brazil (Burtnik, 2006, Heck & de Mejia, 2007). In Argentina, Yerba Mate is cultivated by small, medium and large farmers, family businesses, agricultural cooperatives or large-scale enterprises for both local consumption and export. The total area of *I. paraguariensis* plantations in the provinces of Misiones and Corrientes (Argentina) is 203,800 hectares with an annual production of 650 million kilograms of green leaves – the basic raw material – equivalent to 250 million kilograms of Yerba Mate (INYM, 2023). It is a plant that is part of the culture of its endemic countries, but has gained popularity all over the world, including Poland, due to its health properties. An infusion of Yerba Mate contains almost twice as many antioxidants as green tea, has health-promoting properties and also presents a stimulating effect, making it an attractive alternative to coffee (Bracesco et al., 2011; Heck & de Mejia, 2007; Kłopotek & Dmowski, 2022). *Ilex paraguariensis* can be grown using both conventional and organic methods. Commercial products from both forms of cultivation are available on the market. Organic Yerba Mate available on the Polish market most frequently possesses a name supplemented with the word "organic", which means that the terms 'organic' and 'ecological' are treated as synonyms. These products are manufactured from Paraguayan holly grown on organic plantations that comply with organic standards developed by authorised institutions. Yerba Mate of organic origin available on the European market should meet all the requirements for organic products contained in European Union (EU) legislation. Producers of organic Yerba Mate are required to cultivate with respect for natural systems and cycles and to maintain and improve the soil, water and air and ensure the integrity of the production and management of biological processes based on organic systems and using natural resources. In addition, organic plantations should minimise the use of non-renewable resources and external inputs and ensure the application of organic plant propagation material (Regulation 2018/848). Therefore, organic Yerba Mate cultivation depends on the natural vegetation cycle of the Paraguayan holly – this process without the use of pesticides lasts marginally longer, however the plants retain more nutrients and vitamins. Environmental protection is also aided by the manner the leaves of organic Yerba Mate are dried, which is completed with using hot air, without the use of smoke from the hearth (MateMundo, 2023). If the producer meets the requirements of the legislation, appropriate indications in the form of organic certificates and, above all, the EU organic production logo (Figure 1), which provides organic products within the EU a consistent visual identity, can be displayed on the packaging of organic products.



Fig. 1. EU organic production logo

Source: Regulation 2018/848.

This label enables effortless recognition of organic products and helps farmers market them in EU countries. In addition to the EU organic production logo, the number of the certification body should appear on the packaging, as well as the place of production of the agricultural raw materials included in the product. If the raw material has been produced outside the EU, the information 'non-EU agriculture' is indicated. The organic production logo can only be applied to products that have been certified as organic by an authorised certification body or control authority. It means that such products are obliged to comply with all the requirements concerning the production method, processing, transport and storage. The logo can only be used on products that contain a minimum of 95% organic ingredients and, moreover, meet other specified requirements for the remaining 5% of ingredients (Gadomska et al., 2014; Komisja Europejska, 2023; Pietraś, 2018; Your Europe, 2023). The packaging of organic Yerba Mate may also contain other labels specific to the endemic country of origin of the product, such as USDA ORGANIC or Organico Argentina. Certification bodies for organic products in Brazil or Argentina also expect certain conditions to be met. Growing organic Yerba Mate involves meeting the requirements for organic products such as not adopting pesticides and other agrochemicals, and applying the right planting density with respect for the environment (Eibl et al., 2017). In Argentina, some private companies that certify organic farming require buffer strips with native trees to prevent wind from carrying agrochemicals from nearby farms to organic Yerba Mate fields (Montagnini et al., 2011).

Due to the increasing interest of Yerba Mate producers in creating the conditions for organic production and the activities of the distributors of these products, who target to meet the needs of consumers for products of organic origin, it is advisable to undertake research into the identification of factors determining consumer behaviour in the market for this type of product.

Research method

The subjects of the study are Yerba Mate consumers and the selection of the research group is purposive, non-random. The research tool used to achieve the aim of the study is the author's survey questionnaire. The survey was conducted using the CAWI (Computer Assisted Web Interview) method, i.e. an electronic questionnaire for self-completion over the Internet, with the use of a Google form. The questionnaire consisted of 9 single and multiple-choice questions and a metric. The questionnaire was shared in three thematic groups on the social networking site Facebook, whose members are Yerba Mate consumers. The Yerba Mate themed groups selected were: "YMPL-Yerba Mate Polska", "Yerboholicy-Yerba Mate" and "Yerba mate Poland".

The questionnaire included questions on, among others, the frequency of consumption of organically grown Yerba Mate, the purchase origin, and consumer preferences regarding the size and price of the unit packs. Consumers were additionally asked about the barriers and factors motivating their purchase and consumption.

A total of 104 respondents participated in the survey. The structure of the respondents by gender and age is shown in Figure 2.

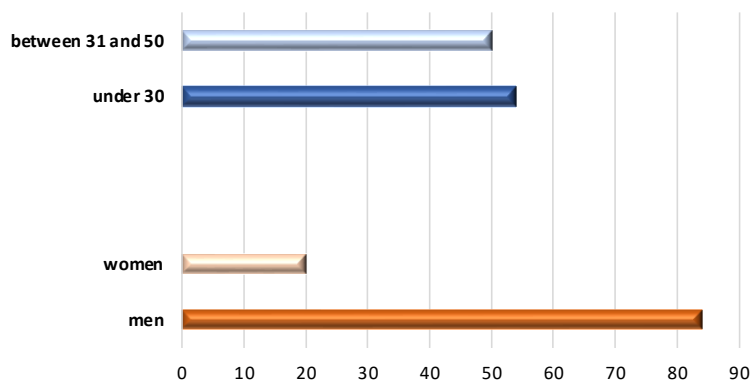


Fig. 2. Characteristics of respondents

Source: own elaboration.

The questionnaire was completed by 20 women (19%) and 84 men (81%), almost half of whom were under 30 years of age (10 women and 44 men). The rest of the respondents (50 respondents) were assigned to one group – between 31 and 50 years of age – in order to make the distribution of socio-demographic groups statistically comparable. Significantly fewer women than men took part in the survey, which is a recurrent phenomenon in consumer research on Yerba Mate, as men are the main consumers of this product. This may be mainly related to the sensory

characteristics of Yerba Mate infusions, i.e. a very intense bitter taste, which may be difficult for women to accept.

Results and discussion

The majority of the respondents (60%) stated they consume organically grown Yerba Mate. 31% do not drink this type of product, of which 21% had once tried it. The remaining group (10%) admitted that they do not know if the Yerba Mate they drink is organic. This may be due to the lack of consumer knowledge of organic product labelling, which is necessary to correctly identify this type of product.

The vast majority of respondents (81%) indicated that they shop at a specialist online Yerba Mate shop. Such shops offer a wider assortment and availability of Yerba Mate products than tea shops, which were indicated by 4% of respondents as their primary shopping destination, or organic food shops (2% of indications). It occurs that popular specialist online Yerba Mate shops also operate stationary shops, however, frequently due to the distances to such outlets and the convenience of shopping, consumers are more likely to choose to purchase online. This applies to both under and over 30-year-olds. The remaining group of respondents (13%) declared that they do not know where Yerba Mate is purchased because they do not shop independently.

The amount of organic Yerba Mate consumed among the total Yerba Mate consumed by respondents is depicted in Figure 3.

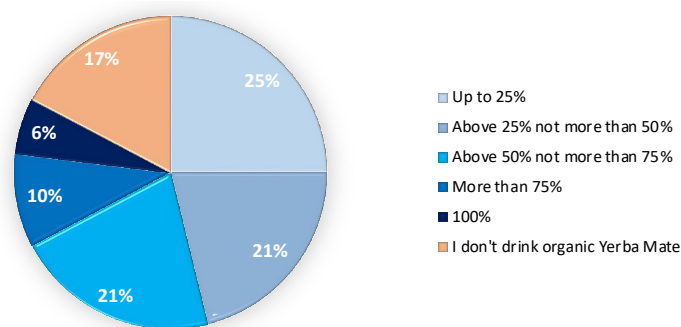


Fig. 3. Amount of organic Yerba Mate consumed among total Yerba Mate consumption

Source: own elaboration.

Although the majority of respondents indicated that they consume organically grown Yerba Mate, only 6% of them declared that they only consume organic Yerba Mate. The remaining consumers choose both organic and conventionally produced Yerba Mate to varying degrees. This may be related to the identified barriers towards the purchase and consumption of this type of product (Figure 4).

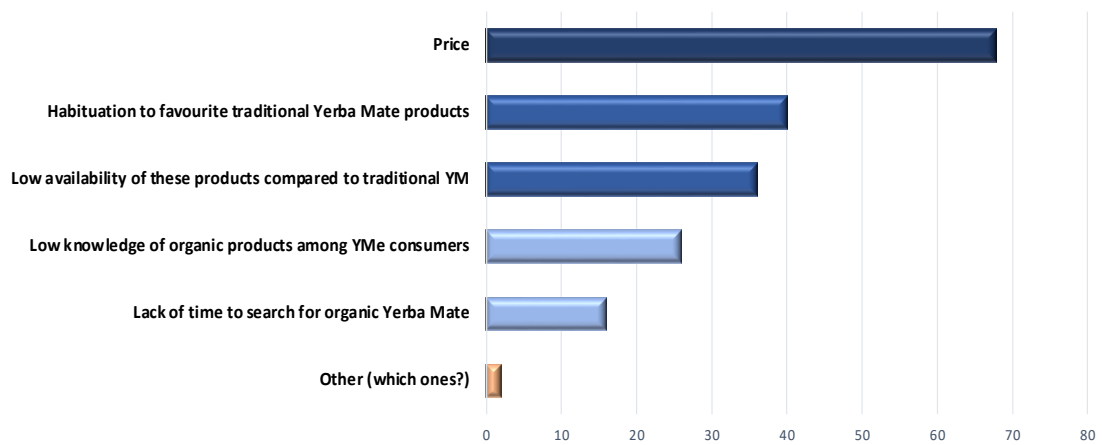


Fig. 4. Identifying barriers to purchasing organic Yerba Mate

Source: own elaboration.

The vast majority of respondents (65%) considered that the main barrier to the purchase of organic holly products by consumers could be their high price. Further constraints to the purchase of this group of products, according to respondents, are the habitual use of their favourite traditional products (38%) and the limited availability of these products compared to the range of traditional Yerba Mate (35%). A smaller group (25%) indicated Yerba Mate consumers' low knowledge of organic products and the lack of time to look for these products in the shop (15%). One Yerba Mate consumer indicated that there are no barriers against buying organic Yerba Mate, while another respondent stressed that the price of organic products is half that of traditional products, but it is worth paying more for a high quality product.

The study also aimed to discover the reasons that drive buyers to consume Yerba Mate from organic farming. The results obtained are presented in Figure 5.

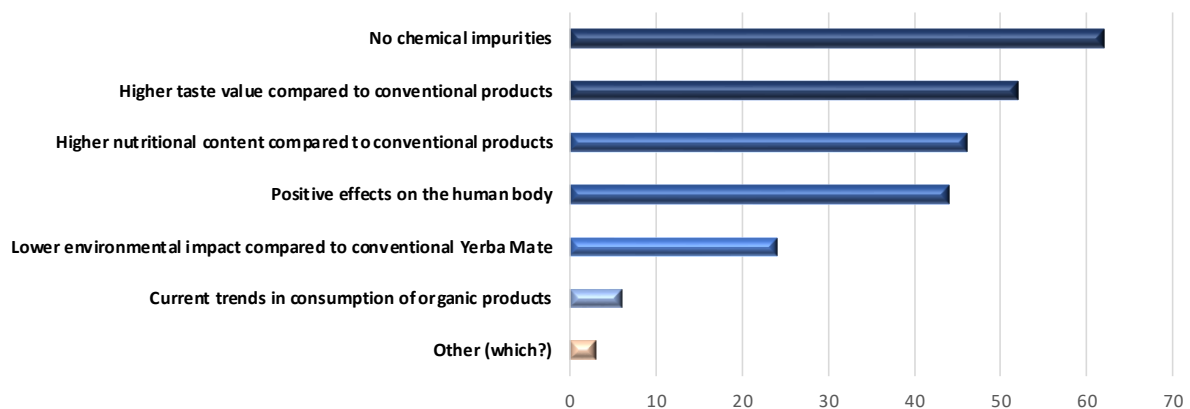


Fig. 5. Reasons for consumers to consume organic Yerba Mate

Source: own elaboration.

More than half of the consumers surveyed choose organic Yerba Mate primarily because, according to them, these products do not contain chemical contaminants (60% of indications), and also because of the higher taste qualities compared to conventional products (50%). Shoppers are also prompted to purchase organic Yerba Mate by its higher nutritional content compared to traditional Yerba Mate (44%), as well as its positive effects on the human body (42%). Furthermore, a large group indicated a lower environmental impact compared to traditional products (23%) as a reason for consuming organic Yerba Mate. These were largely people who also often or sometimes pay attention to organic certifications. Only about 6% of respondents are guided by current trends in the consumption of organic products when choosing these Yerba Mate. Among the additional reasons for consuming organic Yerba Mate, consumers indicated curiosity about the different taste, random choice, promotion of the organic product and choosing it if it is at the same price as the conventional product.

In order to find out respondents' opinions about organic Yerba Mate, they were asked to indicate to what extent they agree with the statements made about these products. The responses are highlighted in Figure 6.

The majority of respondents (65% of indications) believe that ecological Yerba Mate is an organic product. This is understandable, due to the fact that the wording of organic Yerba Mate is used interchangeably with the name: "organic product". On the packaging of organically grown Yerba Mate, the wording '*organic*' ('ang.') or '*organica*' ('es.') is repeatedly observed. However, 19% of respondents strongly disagree with this statement, which may be caused by a lesser knowledge of organic labelling. In contrast, 64% of respondents are of the opinion that the production of organic Yerba Mate involves less negative environmental impact than the production of conventional Yerba Mate. The requirements for organic production are stricter than for the production of traditional Yerba Mate, and, therefore, the harm to the environment must be significantly reduced.

According to the majority of respondents (71%), the price of organic Yerba Mate is higher compared to traditional Yerba Mate. The higher price of organic products is related to the need to meet additional legal requirements and a smaller production volume focused on product quality rather than quantity. In relation to other issues, opinions are more varied. Half of the respondents agree or strongly agree with the statement that the availability of organic Yerba Mate in Polish shops is lower than that of traditional Yerba Mate, which is undoubtedly related to the lower production of these products in endemic countries of origin compared to the wide range of traditional Yerba Mate, which affects the imported Yerba Mate assortment to Poland.

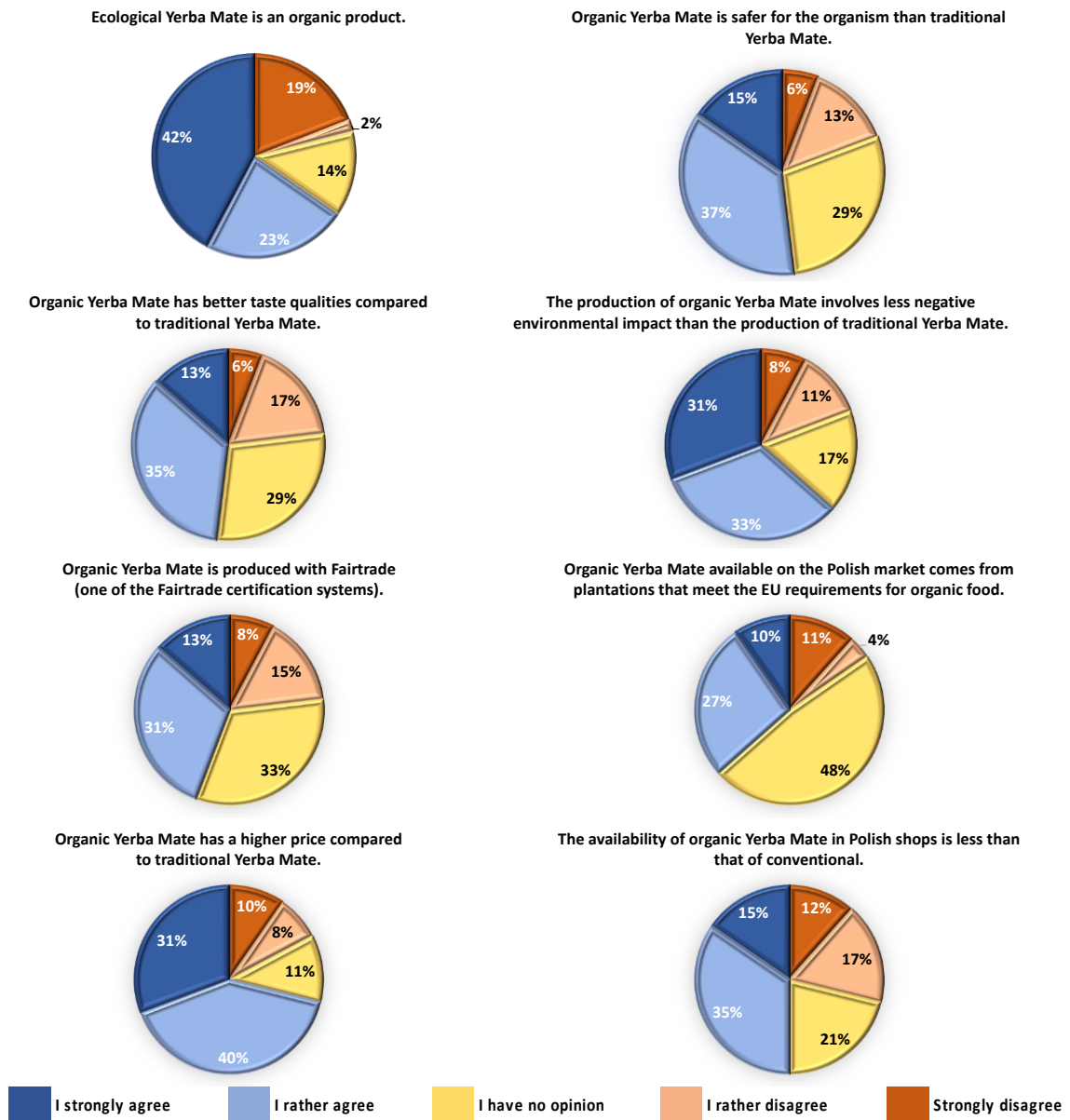


Fig. 6. Degree of consumer acceptance towards information on organic Yerba Mate
Source: own elaboration.

More than half of the group (52%) believes that organic Yerba Mate is safer for the body than the conventional one. This might be due to consumer awareness that this type of product has to be produced, for example, without the use of artificial fertilisers or chemical plant protection products, which undeniably reduces the amount of harmful substances in the product. However, 19% of respondents disagree with this statement, and a significant group of respondents (29%) admitted to having no knowledge of such a fact.

Almost half of the respondents (48%) indicated that, in their opinion, organic Yerba Mate has better taste qualities compared to traditional Yerba Mate. In contrast, 29% of respondents indicated that they had no opinion on the subject. These were mainly participants who do not consume organic Yerba Mate.

The greater safety of organic products and the higher content of health-promoting ingredients are related to the way they are grown. Organic Yerba Mate is grown without the use of agrochemicals, which simultaneously affects the chemical composition of the plant, including the content of phenolic compounds. When pesticides are not used, plants are more susceptible to phytopathogens and tend to produce more phenolic compounds as a self-defense mechanism. Organic products can contain up to 35% more total phenols (Carbonaro et al., 2002; da Costa Abrão et al., 2022; Dani et al., 2007).

Nearly half of respondents believe that organic Yerba Mate is produced with Fair Trade principles, which is, in fact, very often the case. Many varieties of Yerba Mate from organic plantations are also produced according to Fair Trade principles, which include creating opportunities for economically disadvantaged producers, providing decent working conditions or developing the capacity of small-scale producers (Barro et al., 2023; Dragusanu et al. 2014). However, there is also a significant proportion of respondents who do not have an opinion on this issue, which may be related to a lack of knowledge of Fair-Trade principles and unfamiliarity with the Fair Trade certificate labelling on product packaging. Furthermore, only 37% of respondents agree with the statement that organic Yerba Mate available on the Polish market comes from plantations that meet the European Union's requirements for organic food. Nonetheless, nearly half of the respondents (48%) have no opinion on the subject. This may also be related to the unfamiliarity with the correct labelling and organic certificates that are placed on such products when they are released for sale on the European market. The observed phenomenon is confirmed by the respondents' answers, as almost 40% of the respondents do not pay attention to the certificates present on Yerba Mate packaging, considering them irrelevant (27%) or not knowing these certificates (11%) (Figure 7).

The majority of respondents (46%) sometimes pay attention to the presence of organic certificates on the packaging of organically grown Yerba Mate and, additionally, about 15% of respondents often or always pay attention to the presence of such certificates when shopping. This may be connected with the fact that a very small group of respondents participating in the survey consume exclusively or mostly organic Yerba Mate. For the vast majority of the group, organic products represent a small part among all Yerba Mate consumed.

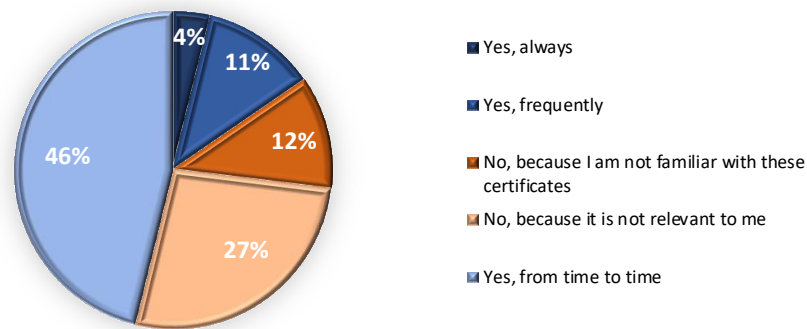


Fig. 7. Yerba Mate consumers' attitudes towards organic certification

Source: own elaboration.

According to the data available on the INYM website, 500g unit packs are the most commonly produced packs of these products and the most available on the market. This is confirmed by respondents' answers, as the majority of them (71%) indicated that they buy 500g packs. Organic and specially selected products are also frequently produced in smaller packs (250g–400g), chosen by 17.3% of respondents.

The vast majority (75%) purchase at the most standard price of a unit pack of Yerba Mate, i.e. between PLN 20 and PLN 40. Yerba Mate unit packs priced above PLN 40 are purchased by 23.1% of Yerba Mate consumers. These are people who frequently consume organic Yerba Mate and pay attention to the organic certificates present on the packaging, and, therefore, may be consumers who consciously choose organic Yerba Mate.

Conclusions

All products available on the European Union market distributed as organic must comply with the uniform requirements described in Regulation (EU) 2018/848 of the European Parliament and of the Council. Organic Yerba Mate additionally also complies with the requirements of countries of endemic origin for organic products and labelling with the appropriate certificates.

Based on the survey, it was found that organically grown Yerba Mate is chosen by consumers less frequently than conventional Yerba Mate, and that the organic label on Yerba Mate packaging is not an important criterion for choosing this product. Consumers are increasingly choosing organically grown Yerba Mate primarily because of the absence of chemical contaminants and higher sensory qualities compared to conventional products. In addition, the higher nutrient content compared to conventional products and the positive effects on the body are important to consumers. Despite the observed trends and fashions for consuming organic products, consumers declare that they do

not consider this as a motivation for consuming organic Yerba Mate. On the other hand, the main barriers that may influence the restriction of purchase and consumption of organically grown Yerba Mate by Yerba Mate consumers are mainly the high price, the habituation to conventional counterparts and the lower availability of organic products compared to conventional ones.

The noticeable increase in the number of organic Yerba Mate producers and the access to an increasing range of these products on the European and Polish market, as well as the increasing awareness of consumers about the benefits of consuming this group of products, may cause an increase in the consumption of these products in the future. Perhaps the benefits of consuming organic Yerba Mate will be a more important factor for them than the identified barriers to the purchase and consumption of these products.

The results of the study can be an important source of knowledge for Yerba Mate retailers and manufacturers, on consumer attitudes and factors influencing their choice of organic products. Based on the results of the research, vendors can build a marketing strategy, wishing to promote organic products.

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COMPARATIVE STUDY OF THE CHANGES IN THE USE OF MICROWAVE OVENS DURING THE COVID-19 PANDEMIC AND POSTPANDEMIC BY HOUSEHOLDS OF POZNAŃ UNIVERSITY OF ECONOMICS AND BUSINESS STUDENTS

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DOI: 10.56091/CTQS.Qual-25

Abstract

The restrictions introduced due to the COVID-19 pandemic in 2020 forced many people to remain in temporary isolation and start working or studying remotely – online. They have also changed the rules of operation of trade and services, including gastronomy, significantly limiting or temporarily eliminating access to canteens, bars, restaurants, etc. The situation has forced it to have an impact on the culinary behavior of consumers. These changes have probably affected the frequency and manner of using microwave ovens, which are standard equipment in about 70% of Polish households (Statistics Poland, 2022). The study presented below attempts to identify changes in the use of microwave ovens by a selected group of consumers during the COVID-19 pandemic and in the first months after its end. It could show midterm influence of pandemic on consumer behavior in microwave oven use in their households. The inquiry was carried out among households of students of Commodity Science and Management and Production Engineering, Poznań University of Economics. The obtained results indicate a significant increase in the frequency of use of microwave ovens in the study period. They also point to some changes in the application profile of microwave ovens towards intensifying the heating of ready-made dinner sets and snacks while reducing the operation of defrosting food.

Keywords: microwave oven, COVID-19, household

Introduction

Microwave ovens are currently standard equipment in households both in the world and in Poland (Detz & van der Zwaan, 2020; Piskiewicz&Radziukiewicz, 2018; Polish Statistic, 2022). The widespread use of microwave ovens in households is influenced by several factors, among which the most important are: availability and affordability of appliances, a wide range of culinary operations that can be carried out using a microwave oven, ease of use, speed of the food heating process, convenience of use, small dimensions of the device and safety. The advantages of using microwave ovens fit well with the expectations of consumers resulting from the model of life in a constant rush, which characterizes modern civilization. By reducing the time needed to prepare a meal, the microwave has

become, on the one hand, a convenient kitchen appliance, and on the other hand, one of the stimulators of social changes in terms of lifestyle, social relations, culinary and nutritional behavior. For the food industry, the widespread use of microwaves has created a receptive market for various types of food products adapted to quickly achieve cooking readiness in the microwave oven. Products of this type have a significant share in the market of the so-called convenience food. This term is used to describe food products that require little time and effort to prepare for consumption (Safko & Brake, 2009, p. 12).

Despite the small number of studies on the use of microwave ovens in Polish households and the methods and intensity of their use (Korzeniowska-Ginter and Tkacz, 2018; Melski, 2018; Czarniecka-Skubina, 2016), based on the available data and knowledge of the social, cultural and nutritional conditions of Polish consumers, it is possible to determine the role of the microwave oven as an auxiliary device.

The COVID-19 pandemic has had a powerful impact on social life, both in the areas of work, leisure, interpersonal contacts and culinary behaviour (Di Renzo et al., 2020). A variety of restrictions related to the COVID-19 pandemic were in place from March 2020 to May 2022. The most severe ones, related to e.g. the closure of catering establishments and drastic restrictions on trade and services, fall on the period from October 2020 to April 2021. Later, as the number of infections and deaths decreased, the restrictions were gradually eased.

A long period of isolation at home, working and studying online, closure of restaurants, cinemas, theatres, etc., has drastically changed the model of social behaviour, including the culinary habits of society. It can be assumed that the change in culinary behaviour forced by the period of isolation has changed the scope and frequency of use of home microwave ovens.

Both people staying at home due to the isolation order and those working remotely online were forced to eat at home. Similarly, as people who have previously used restaurants, bars, canteens, etc. They could use and eat meals prepared at home or have their meal delivered to their home or workplace. In the case of both consumption models, there was a lot of space to use microwave ovens, from heating ready-made dinner sets delivered to the house, through reheating dishes previously prepared in one's own kitchen, to heating various types of snacks. A larger amount of available time also allowed for an increase in the culinary activity of citizens confined to their homes. In this case, microwave ovens could be used to defrost food, which was a very common practice due to restrictions on trade and movement in public spaces. Many consumers were also able to experiment or develop their experience with microwave cooking. In general, there were conditions for consumers to be more active in their own kitchens, most of which were equipped with microwave ovens.

However, it should be remembered that a greater amount of available time in the kitchen results in an intensification of classic methods of preparing meals, including heating, cooking, baking, etc. In this context, determining the degree of change in the way and frequency of use of microwave ovens can broaden the view of the use of this device in households during the pandemic.

The few publications on the analyzed phenomenon do not clearly indicate significant changes in the intensity of microwave oven use in other countries (Mustapha et al., 2021; Zhou et al., 2022). The significant variation in consumer behavior in such a special situation as a global pandemic is due to a number of factors. The most important are: the timing, intensity and duration of the pandemic in a given country, the length and restrictiveness of the lockdown, culinary habits, cultural conditions, the economic situation of the country and its citizens.

The research hypothesis was formulated as follows: in the studied group of households, the long-term effect of the COVID-19 pandemic is a significant increase in the number of cooking operations performed with microwave ovens.

Methods

The small number of publications on the study of consumer behaviour in the use of microwave ovens and the need to refer to the previously surveyed group of consumers determined the choice of the surveyed group of consumers. The study covered first-year students of two faculties at the Poznań University of Economics and Business: Commodity Science and Management and Production Engineering. The choice of the study group was also justified by many years of research on this group, which allowed not only to examine consumer behavior through questions directly related to the pandemic period, but also to observe long-term trends (Melski, 2021). The applied solution gives only a fragmentary picture of the phenomenon, but allows to embed the obtained results in a broader time perspective.

The study was carried out in the form of survey. A set of questionnaires has been prepared in order to collect information from microwave oven users. The questionnaire was composed of 20 items giving the inquired people a possibility to choose his answer among indicated items.

The questions were divided into several groups: the use of a microwave oven, the dishes used, the pros and cons of microwave heating, knowledge of microwaves, their impact on human health and changes of microwave food heating habits during pandemic. The results of the first and last groups of questions were used in the presented analysis.

The survey was conducted on 155¹ students of the first year of two The Poznan University of Economics and Business courses: Commodity Science and Management and Production Engineering. The study was conducted between November 2022 and January 2023 by completing a paper questionnaire. In the instructions for the questionnaire, respondents were asked to answer questions about their family home. In the case of first-year students, behaviour towards dormitories and other accommodations was considered too modest to be studied. The structure of the study group is presented in Table 1.

Tab. 1. Structure of the study group – students of 1st year courses: Commodity Science and Management and Production Engineering, The Poznan University of Economics and Business

Variable	Feature	Number of respondents	Percentage of respondents
Gender	Women	83	54
	Men	72	46
Place of leaving	Village	39	25
	Small city, under 10 ⁴ inhabitants	20	13
	Medium city from 10 ⁴ to 2 · 10 ⁵ inhabitants	51	33
	Big city, over 2 · 10 ⁵ inhabitants	45	29
Number of persons in the household	1	2	1
	2	11	7
	3	35	23
	4	58	38
	5 and above	38	25
	n.a.	11	6

Source: own study.

Results and analysis

125 among the households surveyed (80.4%) possess microwave oven, which is 17 percentage point above the national average – 63.8% in 2021 (Polish Statistic, 2022). It should be noted, however, that the study involved middle-aged people more open to modern solutions. Taking into account the purpose of the study, only the responses of respondents who declared having a microwave oven in their household were further analysed.

¹ It was over 90% of active students.

Respondents determined the average frequency of microwave use in their households (Figure 1). The distribution of responses clearly shows that for about half of households, a microwave oven is an everyday appliance. In others, it is more of a kind of equipment used occasionally or is more like a home gadget.

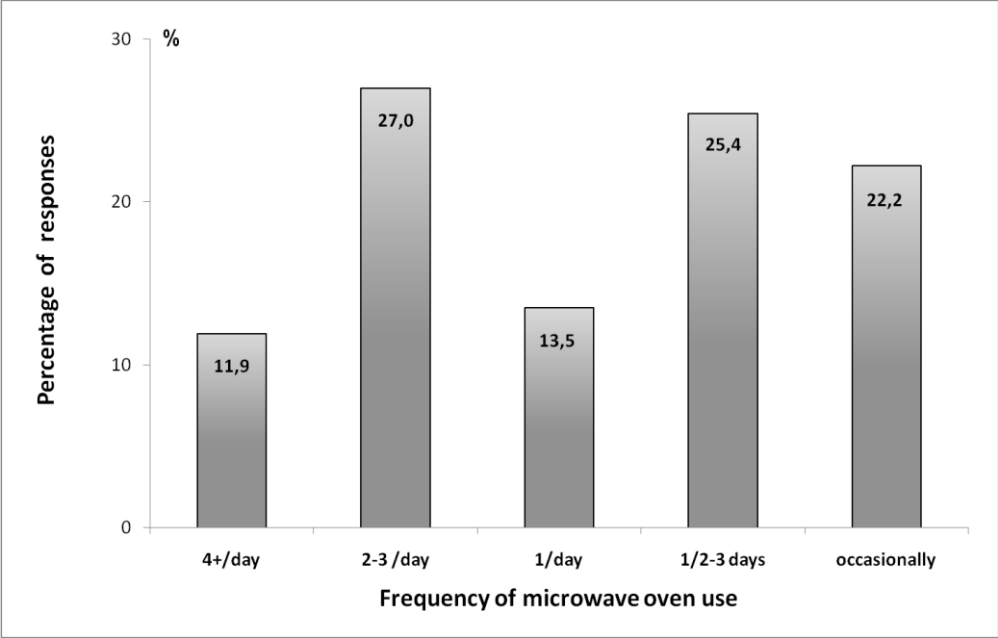


Fig. 1. Frequency of microwave oven use in households of questioned students
 Source: own study.

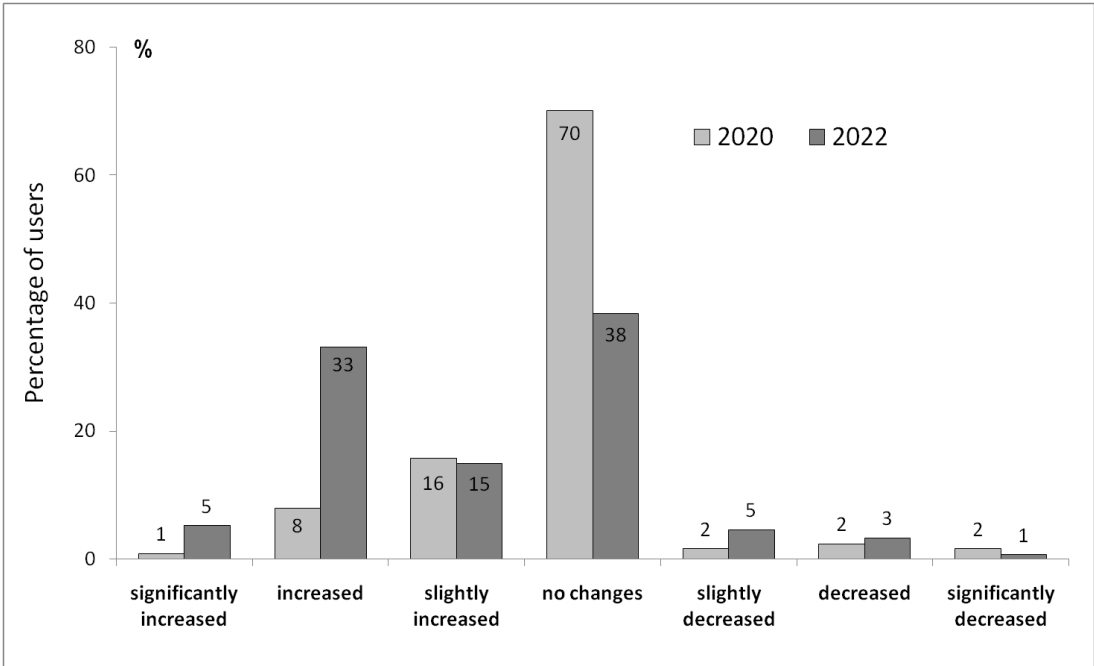


Fig. 2. Changes of microwave oven usage frequency during COVID-19 pandemic (rounded to full units)

Source: own study.

In the survey, the majority of respondents (53%) said they had increased their use of microwaves during the COVID-19 pandemic, while 8% of respondents said the opposite (Fig. 2). A change in the assessment of culinary habits in the kitchens of the surveyed group is clearly visible along with the time that has passed since the period of isolation at home during the pandemic. Compared to the results of the 2020 survey, there was a more than twofold increase in the percentage of people declaring an increase in the frequency of using a microwave oven, which took place at the expense of a group of respondents who did not see a difference in the frequency of use (70% in 2020 vs. 38% in 2022).

A longer time perspective allows to look at the events that arouse great emotions at a given moment in a more balanced way, free from the emotions of the moment. In addition, the presented study concerns a longer period of time, which includes not only the period of the pandemic, but also the time of easing pandemic restrictions to their almost complete lifting. This is the imposition of a disruption related to pandemic restrictions on long-term trends.

The changes that have taken place have probably resulted in new culinary habits of microwave oven owners, which overlap with the existing trends in the frequency of their use and the culinary functions performed. In the long run, they should dominate the previous trends. In order to determine the changes taking place, it is necessary to follow their course in the long term. This process should be preceded by standardization of data obtained from surveys conducted over 20 years.

In order to compare current results with the data obtained during the research from previous years, the results of the analyzed surveys were objectified by determining for each individual editions the coefficient of microwave oven use F_{μ} , expressed by the formula:

$$F_{\mu} = 0,01 \sum f_i k_i$$

where:

f_i – percentage of responses per category,

k_i – coefficient taking values according to Table 2.

F_{μ} takes values from -1 to 1, with negative values showing standardized indications at the level of less than once a day. Positive values, on the contrary, more than once a day. For $F_{\mu}=0$ the mean frequency of use microwave oven is estimated as once a day.

Tab. 2. Value of k_i coefficient for answer according to frequency of use microwave oven in household

Frequency of use microwave oven	k_i
4 and more times a day	1
2-3 times a day	0.5
once on a day	0
once per 2–3 days	-0.5
occasionally	-1

Source: own study.

As indicated in Figure 3, during 20 years of research, a clear tendency to reduce declared frequency of microwave ovens use, with a tendency to stabilize at $F_\mu = -0.25$ has observed before pandemic.

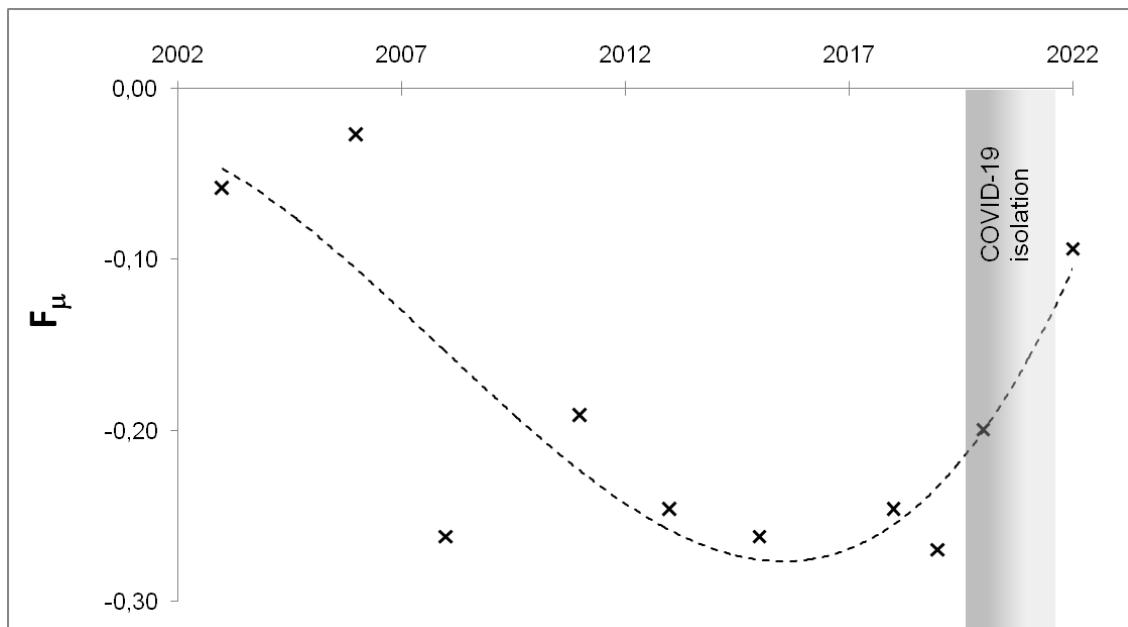


Fig. 3. The changes of declared frequency of using microwave in household express as the F_μ ratio

Source: own studies.

The results obtained in the years after the outbreak of the pandemic: 2020 and 2022 clearly indicate a significant increase in the frequency of use of microwave ovens (Figure 3). The $F_\mu = -0.09$ obtained for 2022 is a return to the average level of years 2004–2005. It should be noted, however, that at that time, 52.2% of respondents declared having a microwave oven in their household, while in 2022 it was 80.6%. The minimum value of the coefficient for 2019, immediately before the pandemic, indicates the decisive impact of pandemic-related isolation on the reversal of the trend. A further increase in the coefficient after the removal of pandemic restrictions seems to indicate a lasting im-

pact of the COVID-19 experience on the change in the culinary behavior of the studied group in terms of the frequency of microwave use.

The questionnaire lists five groups of cooking operations that are usually carried out using a microwave oven: cooking meals, heating pre-prepared meals, heating ready-made dinner sets, preparing snacks, and defrosting food. There is also the option to indicate another cooking operation carried out in the microwave. It was possible to indicate any number of answers.

Among the culinary processes carried out with the use of a microwave oven, the reheating of previously prepared products deserves to be called common (Figure4). Heating ready-made dinner sets is also popular, declared by 2/3 of respondents who have a microwave oven in their household. Cooking in a microwave oven is the least popular among the indicated processes, declared by less than one in six respondents.

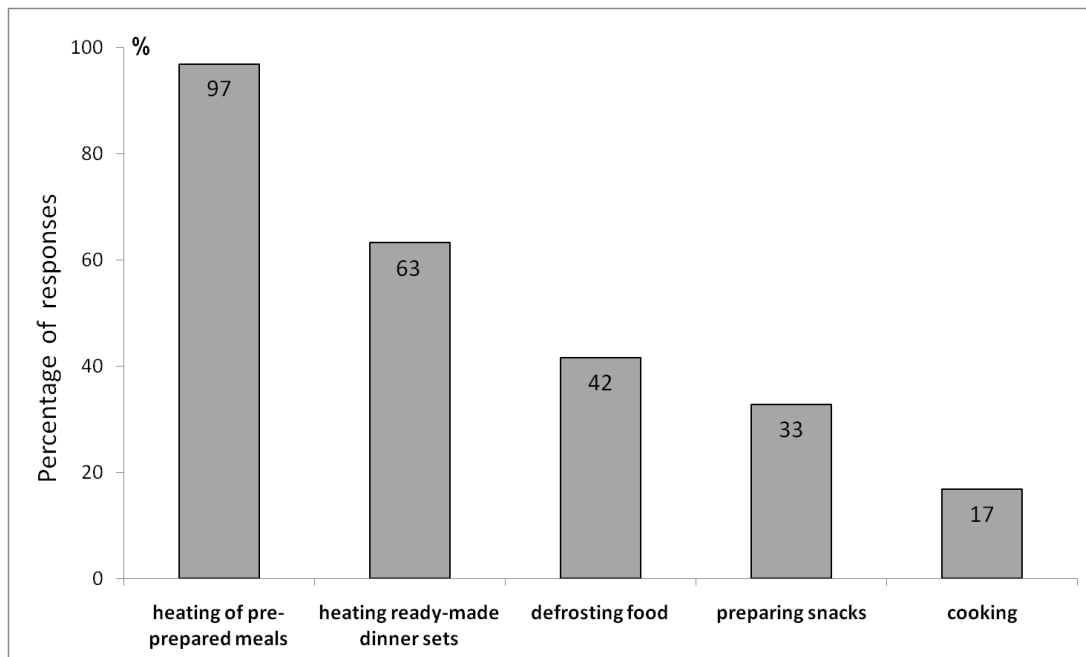


Fig. 4. Percentage of microwave oven users declaring using it to selected culinary processes
Source: own study.

The resulting distribution results from many determinants of consumer behavior. The traditional model of the Polish family with a clear division of roles, also in the kitchen, should be considered as basic. This assumption is supported by the dominance of heating previously prepared meals as the main, common direction of use of microwaves. The second, almost half as popular process is defrosting food.

A comparison of the declared distribution of heating processes carried out in microwave ovens in 2022 with the results of analogous research from 2003–2018 (nine cycles) is presented in Figure 5 (Melski 2018; 2021). In the case of three culinary operations: heating pre-prepared meals, defrosting food and cooking, the percentage of declarations of use perfectly coincides with the trend set by the results of research from the last two decades. Therefore, it can be assumed that the COVID-19 pandemic has not disrupted the long-term trend in the area of indicated culinary operations.

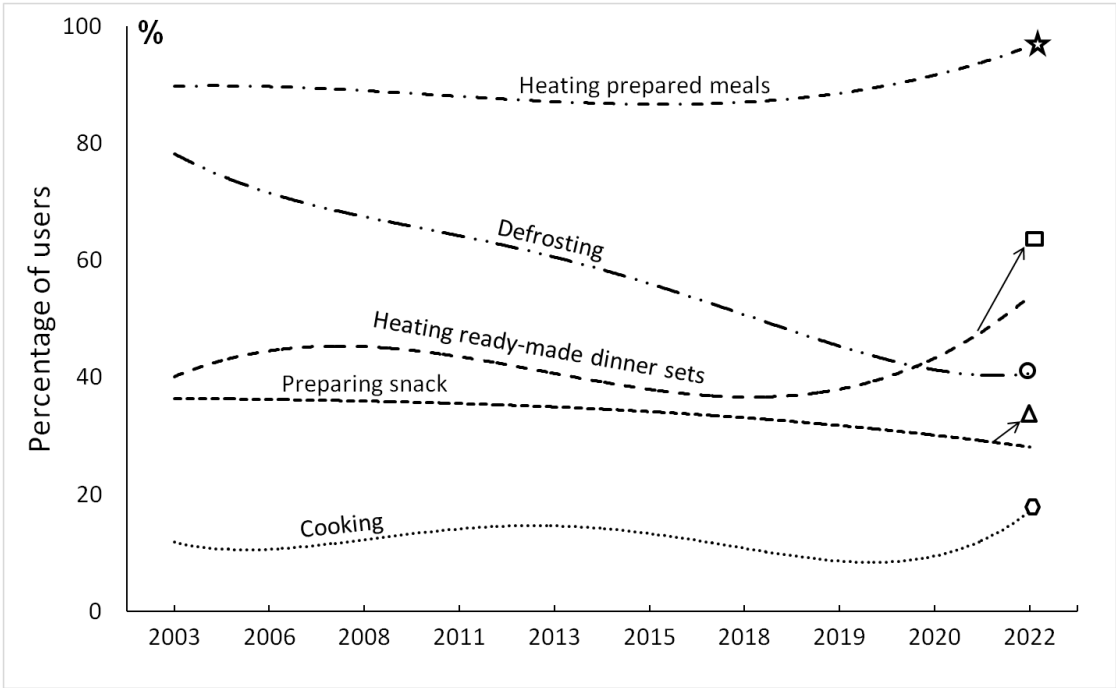


Fig. 5. Percentage of microwave oven users using it to selected culinary processes: lines – the results of earlier surveys, points – year 2022
 Source: own studies.

A significant deviation from long-term trends, towards an increase in the percentage of households conducting this type of operations, was observed in the case of heating ready-made dinner sets. A particularly large increase was recorded in the case of heating ready-made dinner sets. This result corresponds to the popular trend for ordering food, including dinner dishes to be delivered to the home. During the pandemic isolation and the closure of restaurants, bars, etc., this was the only option to consume a meal prepared in a catering establishment. This restriction has been compounded by the development of the takeaway market, which has been going on for several years, contributing to its dynamic development. For many consumers, this type of behavior has become a standard, which has also remained to varying degrees with the removal of pandemic restrictions.

This distribution corresponds to the most widely declared applications of microwave ovens in the last two decades. It clearly indicates an increase in the use of auxiliary functions of the microwave oven,

with a clear decrease of defrosting function. The percentage of users cooking with microwave oven is still low, taking into account character of the question considering it, it seems rather marginal.

The observed trends confirm that microwave ovens play an auxiliary role in Polish households. The basic culinary processes consist of preparing dishes for the whole family in a traditional way. Depending on the needs of individual family members, resulting mainly from the daily schedule, individual portions are reheated individually immediately before consumption. In recent years, especially since the outbreak of the pandemic, there has been an increase in the percentage of households where ready-made dinner sets are heated. This is probably due to the growing popularity of services related to the delivery of ready-made meals to homes. The second factor is the increase in the variety of microwave-ready meals offered by retailers.

Conclusions

The results of the study clearly indicate a significant increase in the frequency of microwave oven use in the surveyed households during and after the formal end of the COVID-19 pandemic. Significant changes were also observed in the distribution of processes in the microwave oven towards a simple function of heating meals. Compared to the previous study conducted during the pandemic, the current one, shows trends in a way that is free of behaviors and emotions directly related to isolation.

On the other hand, it should be borne in mind that the currently observed trends in the use of microwave ovens are the result of the processes taking place in this area since the introduction of microwave ovens to the market and the changes in consumer behavior caused by the pandemic and related changes.

In addition, factors not directly related to the pandemic, but rather to the current, post-pandemic state of development of Polish society and economy, are having an increasing impact.

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APPLICATIONS PREVENTING FOOD WASTE ACCORDING TO USERS' OPINION

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DOI: 10.56091/CTQS.Qual-26

Abstract

The food waste represents a problem of major social significance. In the context of the global food crisis and the growing problem of hunger spread worldwide, proper management and distribution of food products and the reduction of food loss and waste is essential to improve food security. In recent years, numerous increasingly popular initiatives, such as foodsharing, 4P and zero waste have been implemented. An innovative and promising tools designed to reduce food mismanagement are internet apps, intended to prevent food waste, promote recycling and contribute to reduction of the amount of food waste generated on daily basis. The aim of the research undertaken and presented in this publication was to learn about the awareness and knowledge of respondents regarding the issue of food waste and ways to reduce it, as well as to learn the opinions and shopping preferences of users of the Foodsii and Too Good To Go mobile applications, intended to improve the management of surplus food products. Research has shown that all respondents are familiar with applications that offer surplus food products or expired food products at discounted prices, and almost 70% of them make frequent purchases through them. Unfortunately, the quality of the offered products is assessed as good by less than 60% of respondents, and as bad by almost 20%. The analysis of the obtained results, allows to indicate the high development potential of applications preventing food waste, but this requires appropriate promotional and educational activities.

Keywords: food waste, food security, food management, SDGs, sustainable food system, consumer behavior, mobile application, Foodsii, Too Good To Go

Introduction

The Food and Agriculture Organization of the United Nations (FAO) reports that one third of the food produced in the world is wasted every year, which means over 1 billion 300 million tons of food products. At the same time, realizing that over 800 million people are starving in the world, food waste is not only an economic and ecological problem (environmental pollution, degradation of natural resources) that threatens food security, but above all an ethical one, which has been significantly deepened as a result of the negative effects of the COVID-19 pandemic (FAO et al., 2022). In low-income countries, food is lost mainly at the production stage. In developing countries, the main reasons for food waste are low consumer awareness and lack of knowledge about proper han-

dling during transport, storage and preparation of food, as well as lack or inadequate technical facilities. In industrialized countries, more than 40% of food losses and food waste occur at the retail and consumer level (Ankiel & Samotyja, 2020). It can be stated that food losses and food waste increase in direct proportion to the dynamic increase in consumption and this applies to each stage of the food chain, producers and processors, distributors and final consumers (Żukiewicz, 2018). Due to the high importance of the problem of food waste, numerous actions are taken to reduce it at virtually every stage of the food chain. In many countries, one of the seventeen Sustainable Development Goals, set in 2015 by the United Nations, has been adopted and implemented in legislation. Goal 12.3 of the General Assembly calls for halving per capita global food waste at the retail and consumer levels by 2030 and reducing food losses in the production and supply chain (United Nations, 2015). For example, the European Union implemented the goals of reducing food waste contained in Directive 2018/851/EU (reduction by at least 30% by 2025 and 50% by 2030), the US adopted a national goal of reducing food losses and waste by 50% by 2030, and China adopted a law on supporting food waste prevention (Martin-Rios et al., 2023).

In Poland, the issue of food waste is regulated by the Act of 19 July 2019 on counteracting food waste (The Republic of Poland, 2019, Sejm of the Republic of Poland, 2020). The Act defines the rules of food handling and the obligations of food sellers in order to counteract food waste and the negative social, environmental and economic effects resulting from food waste. In July 2021, the Chief Inspector of Environmental Protection (GIOŚ) assessed the effects of the Act's implementation and paid special attention to such desirable effects as: (i) food support was provided to people who needed it, and thus several thousand tons of products were saved, (ii) reduction of methane emissions to the atmosphere thanks to proper storage of waste and proper management of bio-waste (Sejm of the Republic of Poland, 2021). However, it should be emphasized that the Supreme Audit Office in its 2021 report points out that the legal regulations introduced in September 2019 apply only to trade, which did not allow for the creation of a comprehensive system that would fully effectively reduce the scale of the problem of food waste in Poland, while pointing to the need to conduct education and social campaigns addressed to consumers on rational shopping planning or the principles of proper food storage (Supreme Chamber of Control, 2021).

In the aspect of social activities in our country, initiatives such as Food Banks, foodsharing or applications that are gaining more and more users are particularly noticeable, which are intended to support, but at the same time educate consumers in the fight against food waste. Two anti-waste apps, Too Good to Go and Foodsy, are dynamically developing in our country. Too Good To Go is an app developed in 2015 by a startup founded in Denmark. And the application is available in 16 countries, in Poland it became possible to use it in July 2019. Currently, Too Good To Go offers its solution in all

Polish cities over 100 000 inhabitants, in 80% of cities with a population of between 50 000 and 100 000 inhabitants, as well as in many centers with less than 50 000 inhabitants. The Too Good To Go application, which cooperates with over 2400 establishments with surplus food, is already used by over 780 000 users in Poland. The partners of the platform are companies such as: Carrefour, Auchan, Starbucks, Costa Coffee, many well-known and popular restaurants, dietary catering, small cafes, bakeries and local vegetable stores. According to the available information, in 2020, thanks to the application users, over 1 million parcels were saved in Poland, which translates into 2 million 500 thousand kg CO₂ (Gronek, 2021; Mierwiński, 2023). Foodsi is a startup founded in 2019 by M. Kowalczyk and K. Fryszczyn with over 800000 users of the mobile application, of which over 500 000 were joined in 2022. The startup intends to help in the fight against food waste in the spirit of zero waste. The Foodsi platform allows restaurants and shops to sell surplus food. Customers can purchase products by paying up to 25% of the starting price. Since the beginning of Foodsi, consumers have saved over 850 000 food packages. Application users can choose from over 3 000 offers premises throughout Poland, primarily in Warsaw, Wrocław, Kraków, Poznań, Gdańsk, Gdynia and Sopot. Foodsi's partners include restaurants, bakeries, confectioneries, cafes and grocery stores (Szczepański, 2022). The applications operate as a managed marketplace (an online platform that services both buyers and sellers by connecting them together and building features that increase the ease of making transactions). Users can choose from the offers of available locations and reserve a package of their choice in a few seconds. This one will be available for collection on the same or next day before the closing of the premises. The content of the package is usually a surprise (only some points inform about the content of the package) and depends on the current menu or regular sale at a specific point. It should also be added that the applications use mechanisms characteristic of gamification - users wait impatiently for packages, and then try to overtake others when booking (MG, 2022; Szczepański, 2022). In the literature related to the subject of food waste, it is emphasized that in the current times of having a smartphone or tablet, mobile applications, thanks to the possibility of reaching a huge number of consumers, can be a cheap, scalable and effective approach to changing the behavior of buyers of material goods, in particular food products (Mummah et al., 2017; van der Haar & Zeinstra, 2019).

The aim of the research undertaken and presented in this publication was to learn about the awareness and knowledge of respondents regarding the issue of food waste and ways to reduce it, as well as to learn the opinions and shopping preferences of users of the Foodsi and Too Good To Go mobile applications, intended to improve the management of surplus food products. The results of the obtained pilot studies are intended to constitute the basis for conducting further, detailed research using random methods, identifying sensitive areas, which are necessary from the point of view of the

success of the mentioned applications, determining purchase satisfaction or their lack within strictly defined groups of consumers interested in this method. reducing food waste. How important this topic is today is shown by research undertaken in this field in research centers in Poland and abroad (Amaral & Orsato, 2023; Wiśniewska & Czernyszewicz, 2023).

Materials and methods

The research method was the CAWI (Computer Assisted Web Interview) technique. The survey questionnaire was created via Google Form. The survey was completely anonymous. It took about 10–15 minutes to complete the questionnaire. A total of 203 people participated in the study. The survey was also made available on Facebook. Because the survey was addressed to all respondents willing to take part, the sample was selected in a non-random manner using the volunteer sampling method, which is considered effective in conducting an online survey. The survey was conducted in the period from February to May 2023, all questionnaires were completed correctly. The survey questionnaire contained 20 questions about the essence of the problem of food waste, respondents' knowledge and attitude to mobile applications intended to prevent food waste (Foodsi and To Good To Go), as well as their shopping preferences using these applications. In addition, the questionnaire contained 6 questions describing demographic, economic and social characteristics (sex, age, education, place of resident, social status, average monthly household income) of the respondents. The detailed structure of the research sample was presented in Table 1.

Tab. 1. Structure of the research sample

Variable	Characteristic	Frequency (%)
Sex	man	32.5
	woman	67.5
Age	18–24	41.9
	25–30	29.1
	31–40	18.7
	41–50	8.4
	over 50	1.9
Education	basic	1.0
	middle school	3.9
	essential vocational	10.3
	secondary vocational	12.8
	general/post-secondary education	16.8
	undergraduate/engineering	32.5
master's degree	22.7	

Domicile	a city with over 500000 inhabitants	30.5
	city from 200 to 500 thousand inhabitants	25.1
	city from 100 to 200 thousand inhabitants	12.8
	city from 10 to 100 thousand inhabitants	11.3
	city with less than 10000 inhabitants	9.4
Social status	rural area	9.9
	unemployed	1.0
	pupil/student	2.9
	student	22.7
	seasonal worker	5.4
	contract/work contract employment	21.2
	fixed-term employment	27.6
Average monthly income per person in the household	employment for an indefinite period	18.2
	other	1.0
	over PLN 4000	17.7
	3001 – 4000 PLN	43.8
	2001 – PLN 3000	30.1
	PLN 1001 – 2000	5.4
	below PLN 1000	2.9

Source: own study.

Results and discussion

The survey questionnaire opened with a question (Figure 1) regarding the respondents' opinions on the importance of the problem of food waste. As expected, a significant proportion of respondents believe that the issue of poor food management is a significant problem (a total of 80.3% of responses). Almost 20% of the answers indicating the low significance of the problem of food waste (and also no opinion on this topic) indicate the continuous need to educate the society, especially the negative effects of this phenomenon.

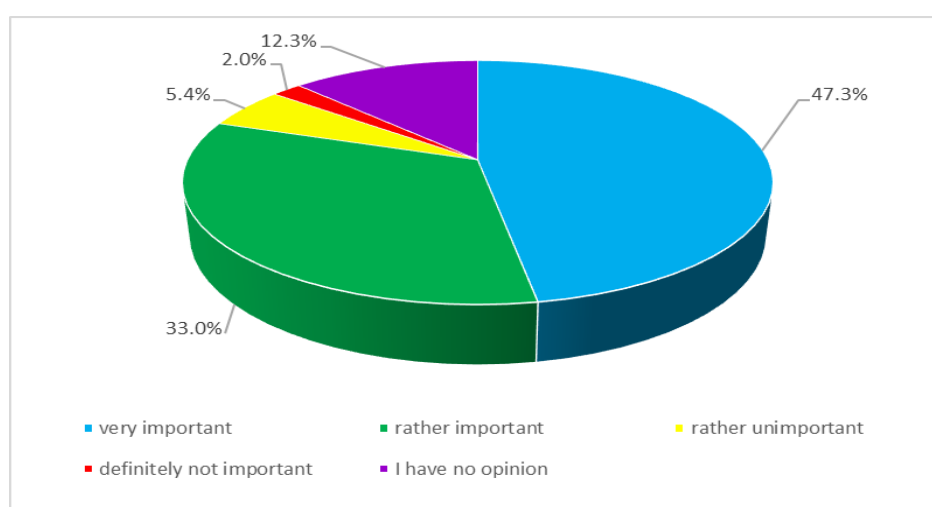


Fig. 1. In your opinion, is the problem of food waste significant?

Source: own study.

Respondents (over 40%) believe that most food is wasted in households, catering outlets and during agricultural production. In their opinion, losses occurring during transport and processing are also significant, while the smallest significant losses are incurred at the stage of trading and displaying food products on store shelves (Figure 2). Such opinions largely coincide with the data presented in the Information on the results of the audit "Counteracting food waste" (Supreme Chamber of Control, 2021).

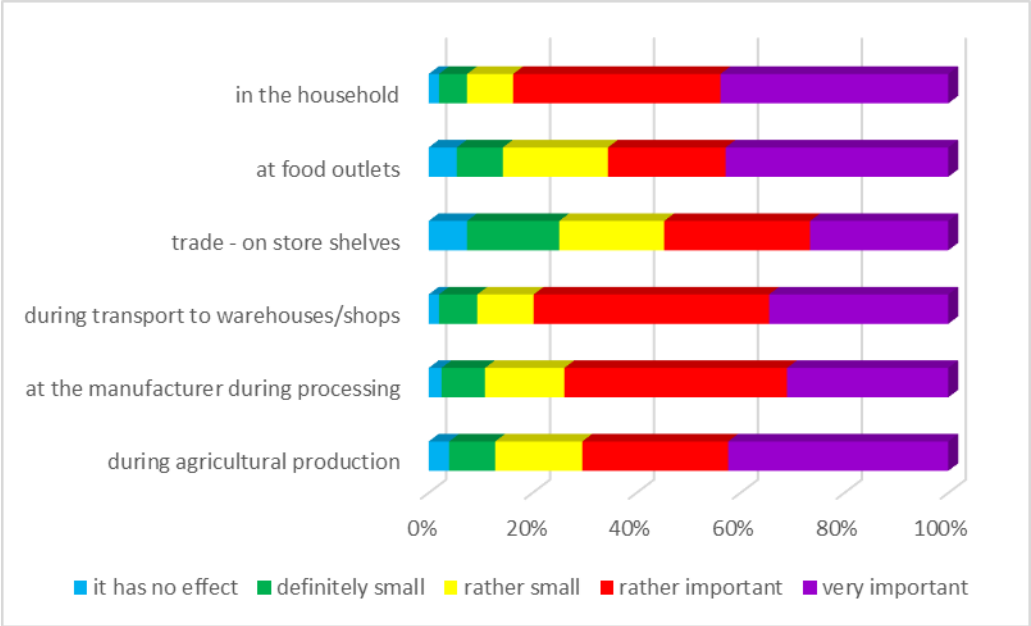


Fig. 2. Where do you think the greatest food waste occurs?

Source: own study.

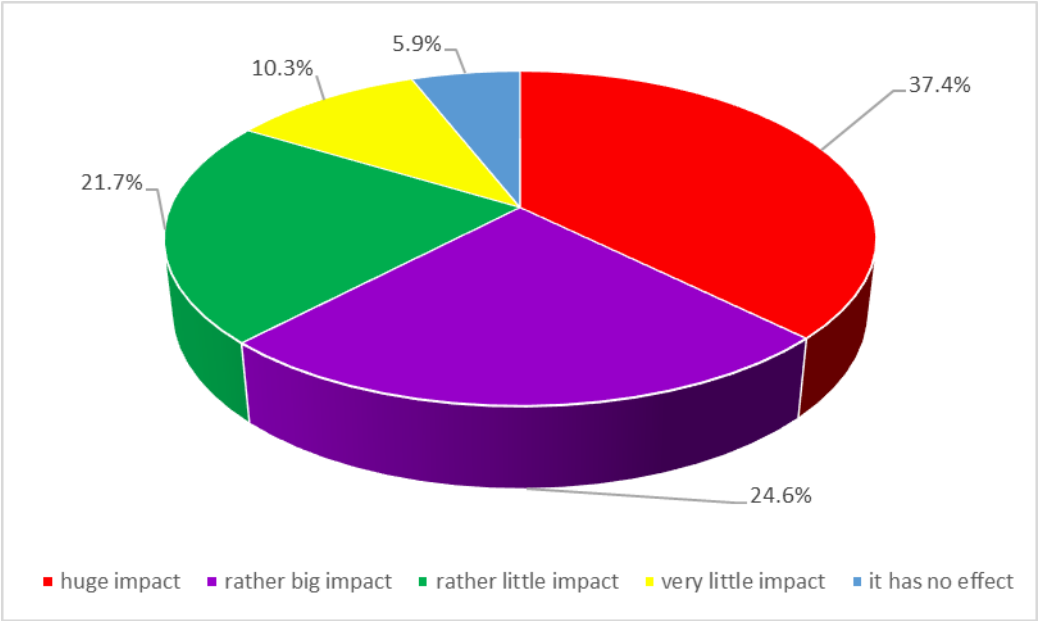


Fig. 3. How does your behavior affect food waste?

Source: own study.

When asked how their behavior affects food waste (Figure 3), respondents mostly admitted that their impact was high (37.4%) or rather high (24.6%). It should be noted that only 5.9% of the respondents believe that their behavior has no impact on the problem of poor food management.

Taking into account the potential causes of wasting food in the household (Figure 4), over 70% of the respondents indicated that one of them was primarily the purchase of too much food (often unconsidered). Respondents admit that very often food is thrown away due to improper storage, lack of ideas on how to use the products to prepare meals, as well as the purchase of too large food product packaging.

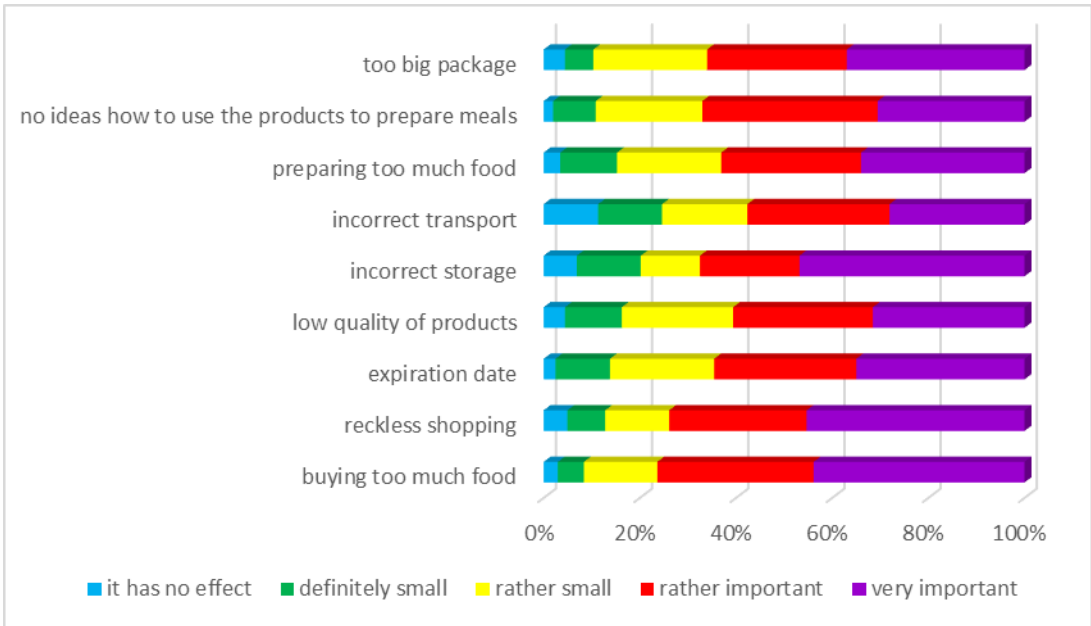


Fig. 4. Reasons for food waste in a household
 Source: own study.

Respondents were asked how they prevent food waste (Figure 5). The most frequently indicated answers were: sharing food, buying seasonal fruit and vegetables and preparing smaller portions of meals. Respondents also attach great importance to planning meals and preparing them from unconsumed food and paying attention to shelf life. The social sensitivity of the respondents expressed by their very high willingness to share undeveloped food and food products is very optimistic.

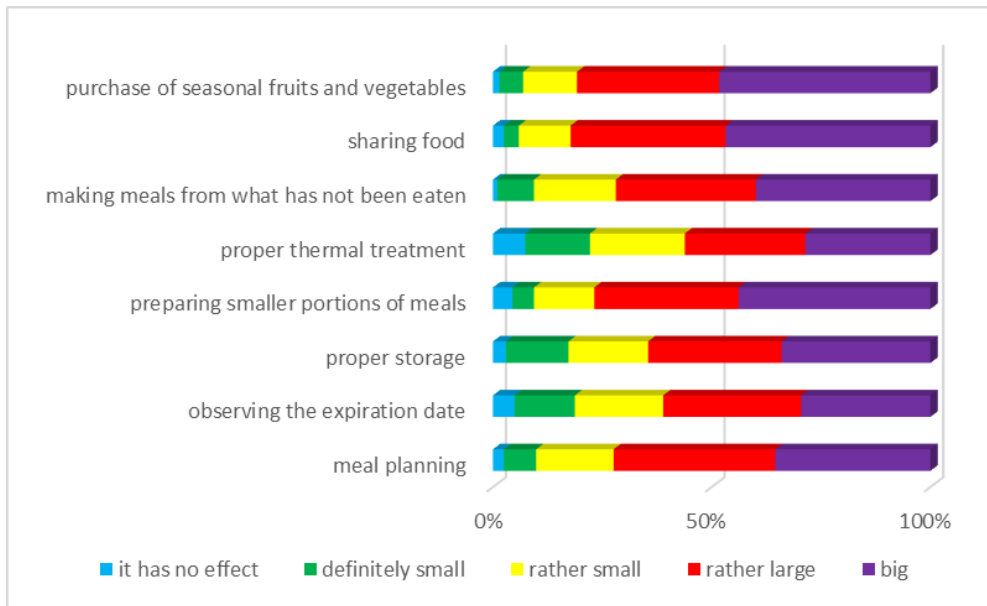


Fig. 5. How do you prevent food waste?

Source: own study.

The next question concerned paying attention by the respondents to the durability dates of food products when making purchases (Figure 6). It turns out that only less than 60% of respondents pay attention very often and rather often (32.5% and 25.1%, respectively) to the durability dates on the packaging. Unfortunately, over 30% of the respondents indicated that they rarely check the durability dates of the purchased products, and almost 7% of the respondents do not do it at all. Therefore, it is necessary to continuously and broadly educate consumers about checking and making them aware of the importance of shelf life dates presented on packaging.

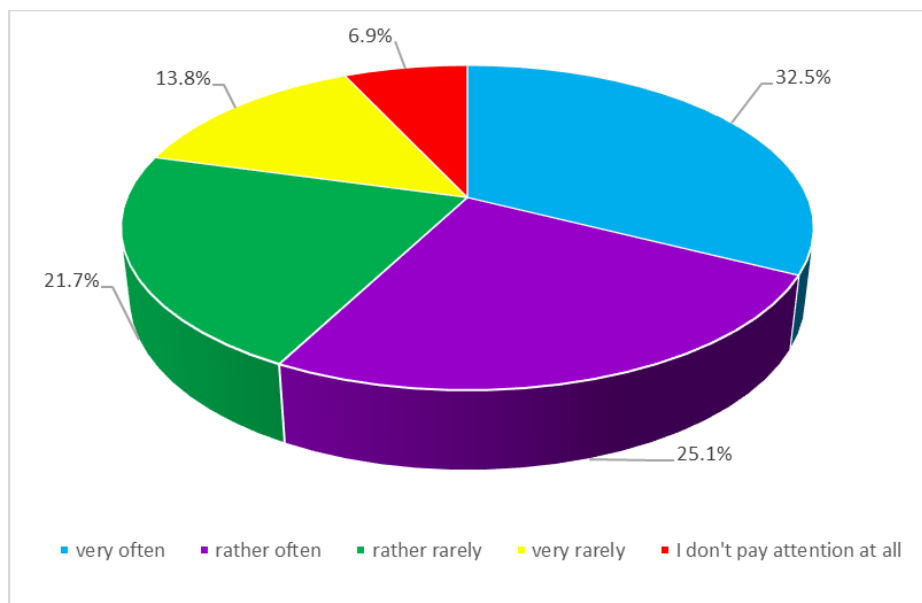


Fig. 6. How often do you pay attention to shelf life dates of food products when shopping?

Source: own study.

Respondents were also asked if they were aware of the effects of food waste. Unfortunately, only less than half of the respondents (47.2%) answered affirmatively. It can be concluded that despite the fact that the issue of waste is present in the social and media space, the information about the negative effects of this process is definitely insufficient or too little to provoke reflection. Therefore, it seems necessary to verify the means and methods of communicating the issue of food waste.

People who answered positively to the question about knowledge of the effects of food waste were asked to list examples of the consequences of the discussed problem. The most frequently indicated answers were (Figure 7): hunger, rising food prices, climate change and environmental degradation. In this case, the indicated effects of food waste largely coincide with those most often indicated both in the mass media and in scientific publications (Ankiel & Samotyja, 2020).

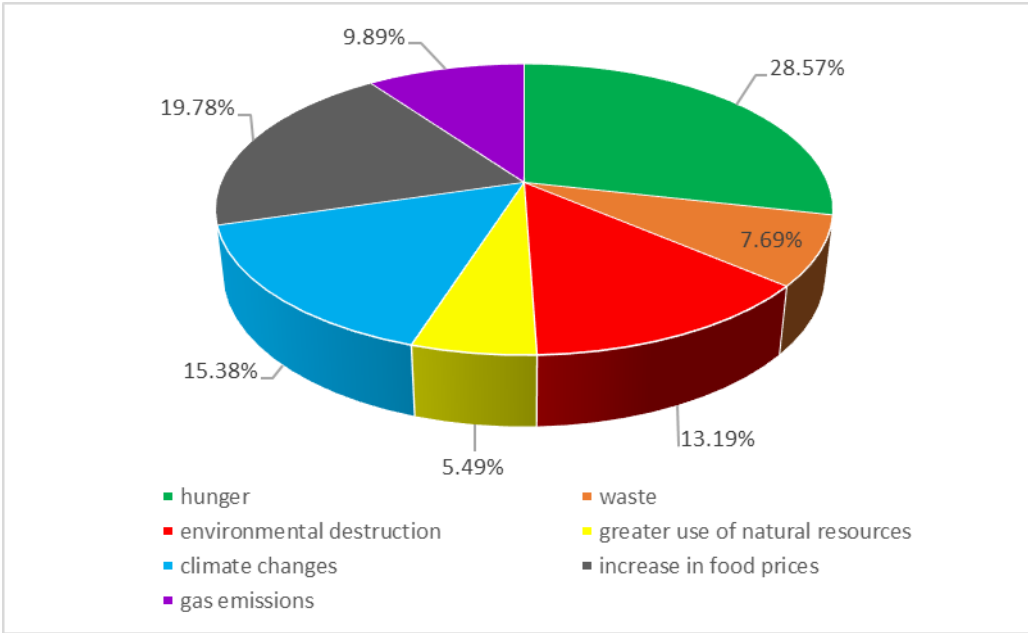


Fig. 7. Please list the effects of food waste known to you?
Source: own study.

Another group of questions was related to applications intended to support the global fight against food waste by activating direct consumers to take actions to increase the use of food products. The first question about familiarity with the Too Good To Go and Foodsy apps received 100% positive responses. Next, respondents were asked to answer the question about the frequency of using these applications (Figure 8). Almost 30% declared very frequent use of the application, and less than 40% rather frequent use of Foodsy or Too Good To Go. The remaining participants in the study indicated a rather rare or very rare frequency of using the indicated applications. Only 1.5% of respondents answered that they do not use them at all.

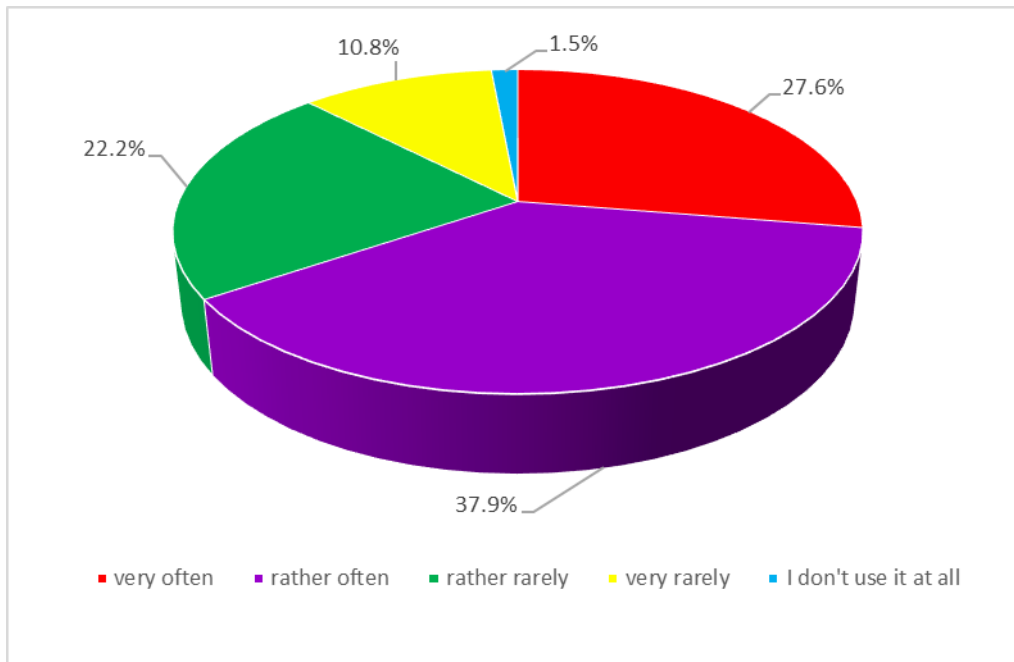


Fig. 8. Have you used applications preventing food waste?

Source: own study.

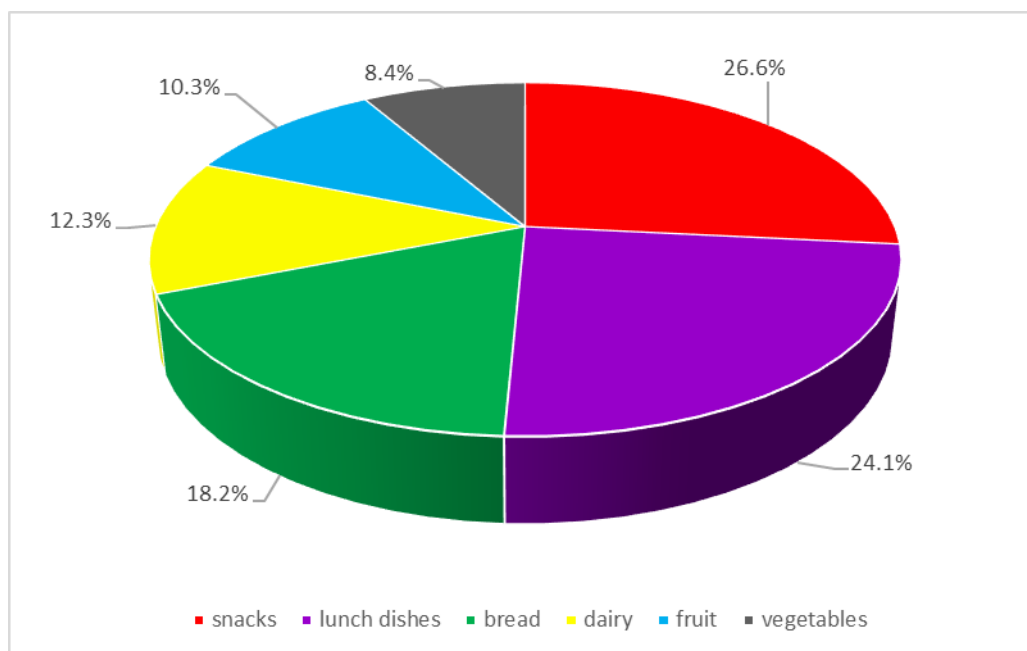


Fig. 9. What type of products do you most often purchase via the above-mentioned applications?

Source: own study.

The next question was about the type of products that consumers most often purchase via apps (Figure 9). The products most often indicated by the respondents were: bread, lunch dishes and snacks. To a lesser extent, respondents pointed to such food products as dairy products and fruits and vegetables. According to the Supreme Chamber of Control report (Counteracting Food Waste, 2021), bread is the most frequently wasted product, therefore, reducing the waste of this product

can be viewed with optimism through the prism of the significant propensity of mobile application users to purchase bread, despite the fact that this product is usually not offered fresh, but comes from baking one or more days ago. It should be remembered that bread from smaller, often artisanal bakeries is characterized by a very high price and is not available to consumers with limited financial resources. A much lower price in this case gives you the opportunity to purchase such a product and at the same time get to know the manufacturer's product offer, which may be an additional element that encourages you to use the Foodsie or Too Good To Go applications.

Interesting opinions were obtained by asking respondents how they assessed the quality of food products purchased through applications (Figure 10). Nearly one third of the respondents believed that the quality of food products purchased via the Foodsie or Too Good To Go applications is rather good, however, a slightly smaller group of respondents admitted that the quality of food was average. Only less than 20% of the respondents considered the degree of freshness of the purchased food to be very good, and as much as about 15% indicated that the quality of the food was bad or very bad. Browsing online forums dedicated to the aforementioned applications, unfortunately, you can find confirmation of the above-mentioned unflattering opinions. There are offers of products whose best-before date has not expired, but the product had visible effects of deterioration and was nevertheless offered to the users of the application (this concerned in particular perishable products marked with the "use by" expiry date).

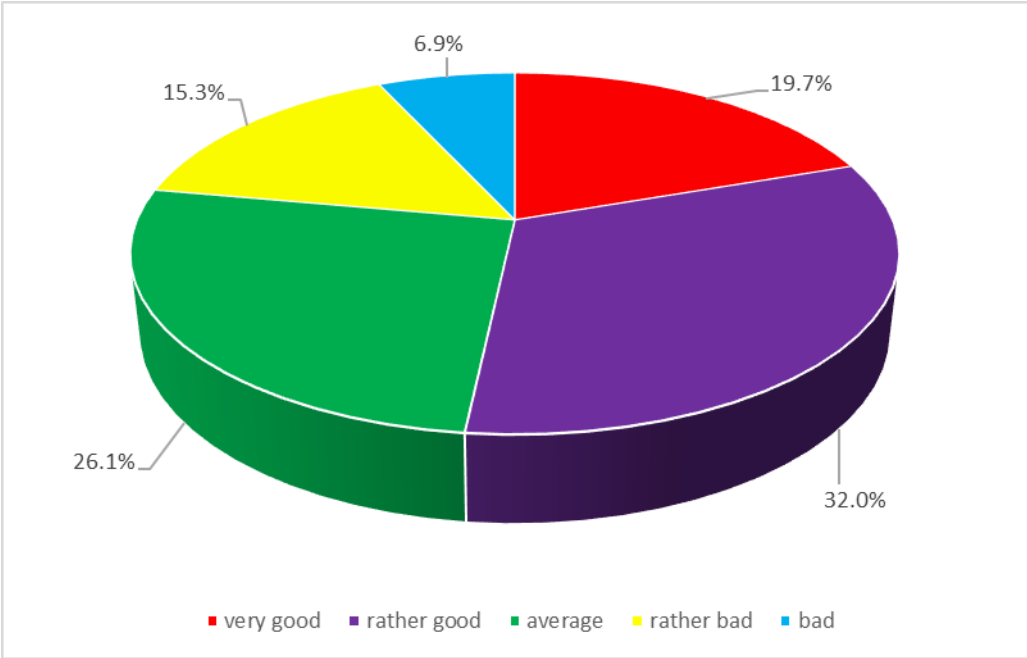


Fig. 10. How do you assess the quality of food products purchased via applications?
Source: own study.

The survey questionnaire also addressed the issue of price satisfaction for products offered through the application. The answers obtained allow us to conclude that practically 2/3 of the respondents were satisfied with the amount spent for the purchased products. However, the percentage of respondents dissatisfied with the costs incurred remains high. When browsing online forums, it can be concluded that the most dissatisfied with the costs incurred (price paid) are consumers purchasing products, in particular in restaurants and hotel cafes. The regular price of the products offered in them is very high, and after the reduction for the purpose of offering the product in the application, it remains at a high level, especially for people with low or medium income.

The next question concerned the indication of factors influencing decisions to purchase food products through applications preventing food waste. For the majority of respondents, an important factor in the purchase of products through applications were, above all, the opinions of other users, the conviction of the positive impact of purchases in applications on the environment, as well as the price and quality of the products offered (Figure 11).

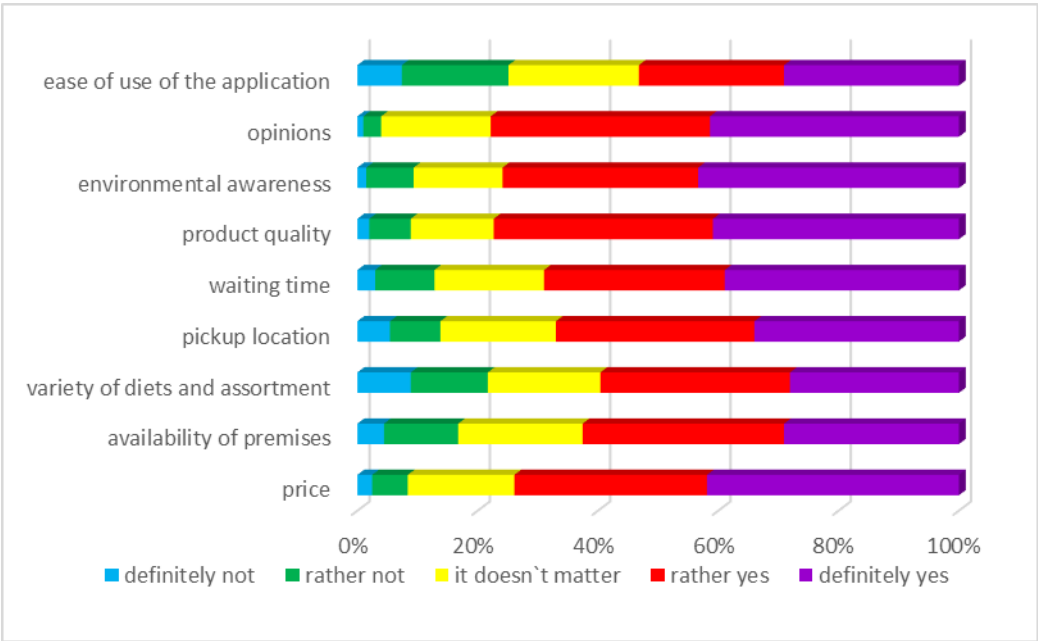


Fig. 11. Factors determining purchasing decisions via the Foods/Too Good To Go apps
Source: own study.

Figure 12 presents the factors that, according to the respondents, determined their satisfaction with shopping. Most of the respondents indicated that satisfaction with purchased products depends on their freshness and attractive price. Long shelf life also increases the positive perception of products. Other aspects, such as the variety of the assortment, short waiting time and friendly service had a lesser impact on the satisfaction of the respondents.

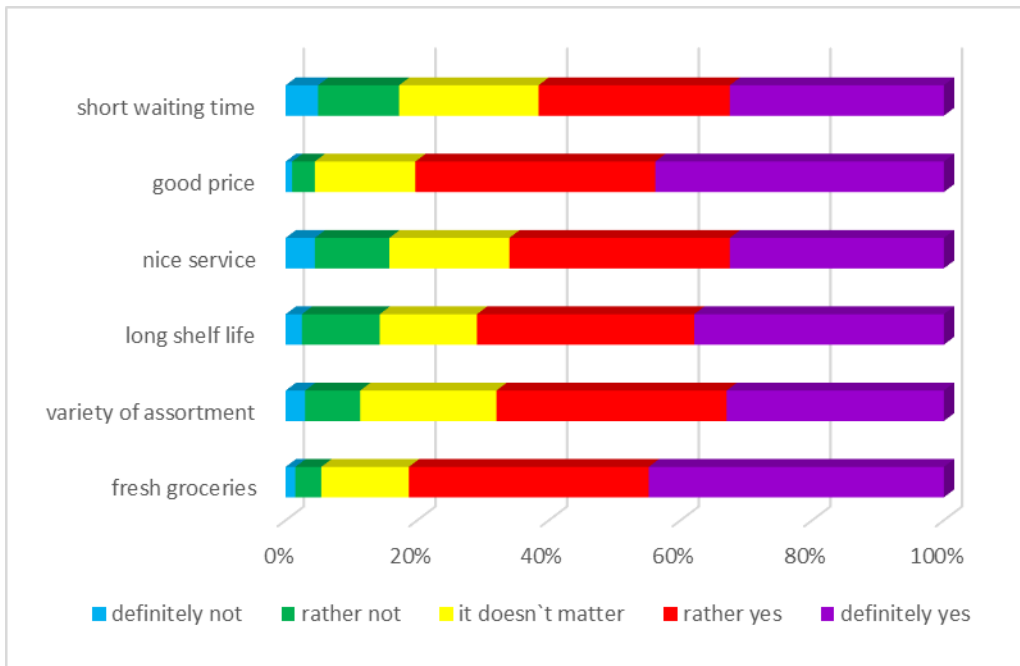


Fig. 12. What factors influence your satisfaction with the purchase?

Source: own study.

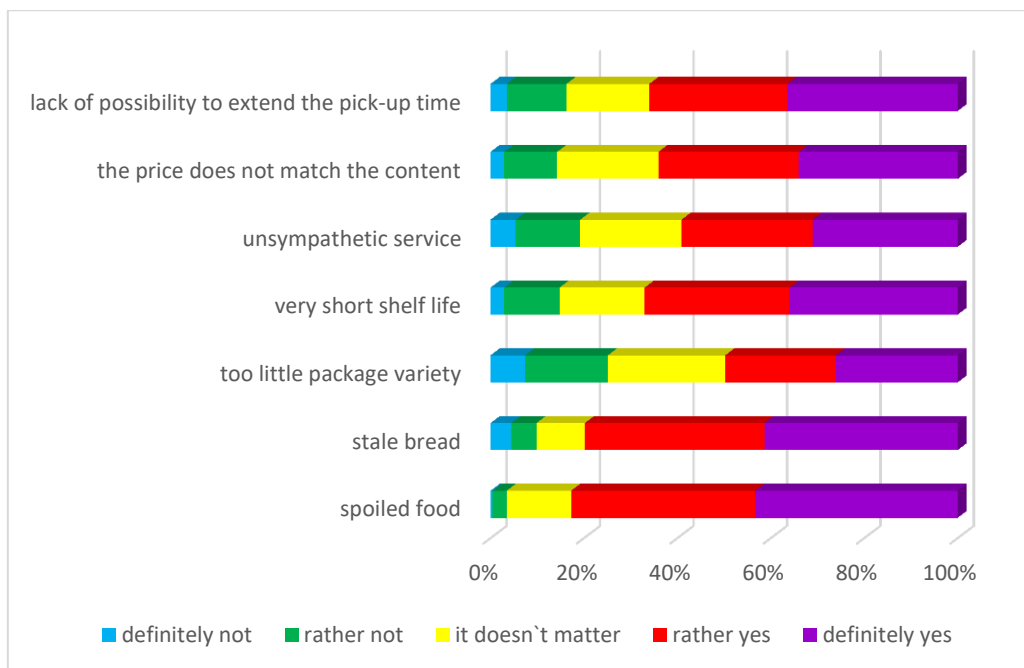


Fig. 13. What factors influence your dissatisfaction with the purchase?

Source: own study.

In contrast to the previous question, respondents were asked about the factors that influenced dissatisfaction with purchases through applications (Figure 13). For almost half of the respondents (43.4%), the most important factor negatively affecting purchase satisfaction was the purchase of food with signs of spoilage. Another key factor determining dissatisfaction with the purchase was the staleness of the bread. It should be mentioned here that the idea behind these applications is to of-

fer food that is not sold on a given day (surplus) or is about to expire. It is not uncommon for bread to be offered the next day after it is produced, therefore some of its organoleptic characteristics may differ from those of freshly baked bread, but it is still wholesome bread. The users of the application were less discouraged by the lack of the possibility to extend the collection time, the very short shelf life or the price inadequate to the content.

Respondents were also asked to determine the impact of selected factors inducing them to specific actions after a satisfactory purchase. Satisfaction with shopping prompts application users primarily to buy other groceries from the same supplier and to recommend the seller among friends. A slightly smaller (but high) percentage of respondents indicated that satisfaction after purchasing food via applications rather persuades them to repeat this purchase from the same supplier and to increase their purchases with the help of Foodsfi or Too Good To Go in general (Figure 14).

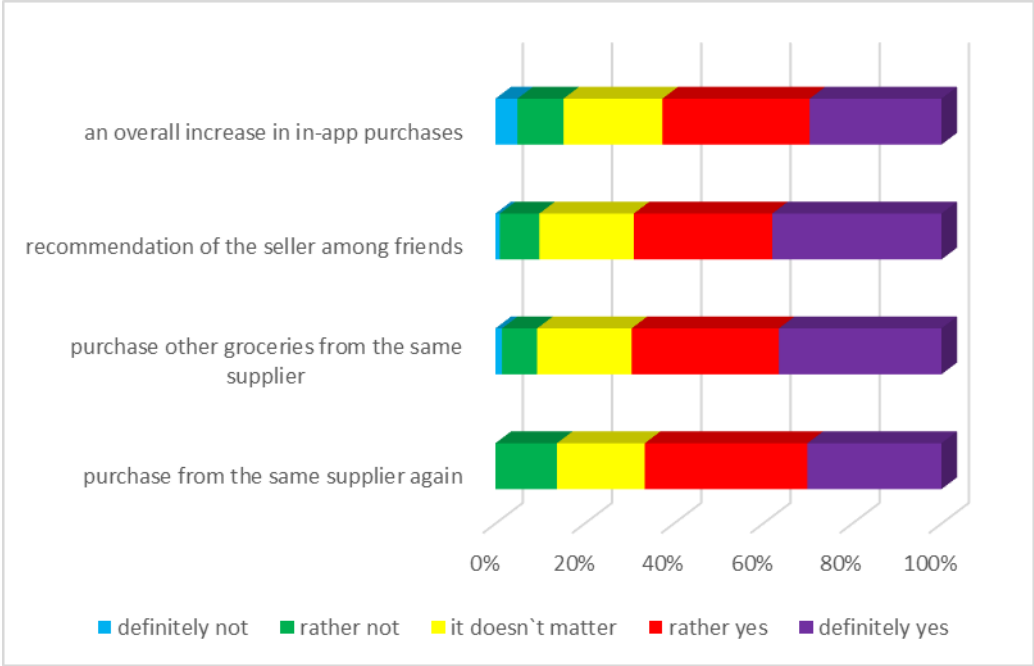


Fig. 14. Decisions driven by satisfaction with the purchase

Source: own study.

Finally, respondents were asked to define their reactions after an unsatisfactory purchase (Figure 15). In this case, almost 70% of survey participants are not willing to buy again from the same supplier, and 43.4% of them stated that they would definitely not make any purchases from a given seller. Unfortunately, dissatisfaction significantly discourages respondents from repurchasing food via applications. In addition, the majority of respondents advertise their negative experiences and opinions about a particular store or general purchase through apps to their friends.

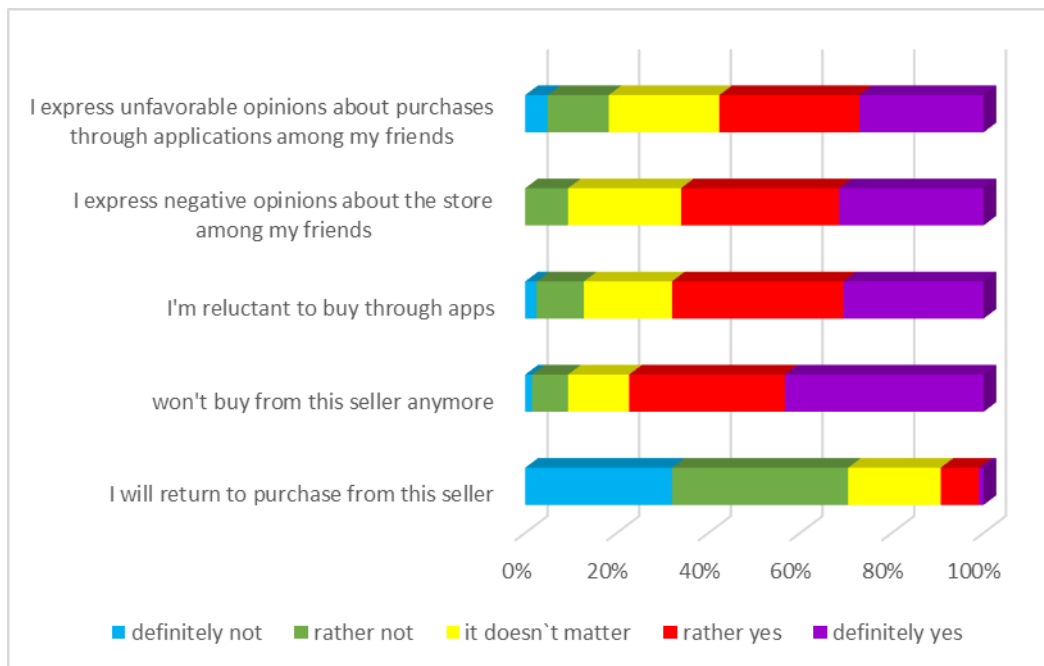


Fig. 15. Actions caused by an unsatisfactory purchase

Source: own study.

Conclusion

The conducted consumer research with the use of a survey questionnaire helped to learn about consumers' awareness of the problem of food waste, ways to counteract this phenomenon as well as the preferences and shopping experiences of users of the Foodsii and Too Good To Go applications. The problem of food waste is widely known, consumers' awareness of proper food management is increasing and they have knowledge about the causes and possible effects of food waste and understand the scale of the given problem. However, there is a high percentage of respondents who consider this issue to be of little or no importance, and the behavior of the respondents influencing food waste is not fully satisfactory. It is therefore necessary to constantly conduct social campaigns aimed at raising awareness of the issue of food waste (including poor management of food products by consumers), in particular its potential effects both on a macro (global) and micro (household) scale, in particular the promotion of behaviors affecting the proper management of food products.

The analysis of the survey questionnaires showed that the Too Good To Go and Foodsii applications are perceived by consumers as useful tools to prevent food waste. Participants of the study are aware of the idea of an application that allows you to purchase products that are unsold on a given day or with approaching end of shelf life at significantly lower prices to avoid unnecessary disposal of food products. Despite the positive attitude of respondents to these applications, not all respondents used them. Over 30% of the respondents rarely use the application, and almost 2% of the respondents did not purchase food products in this way at all. Consumers satisfied with the purchased

products, first of all, recommend applications to their friends, which is their huge development potential. Unfortunately, although the respondents generally claim that they understand the ideas of Foodsii and Too Good To Go, when analyzing the results of the survey or browsing discussion groups devoted to these applications, it can be stated that they do not fully understand that certain products may differ from freshly produced products in terms of some organoleptic features. Products unsold the previous day, for e.g. bread or confectionery, are wholesome products, although their certain organoleptic characteristics may differ compared to a freshly produced product. However, it is obvious, that buyers would often like to receive a product with characteristics identical to a freshly manufactured product. Equally often there are opinions that the purchased product has a shelf life of one or two days, and the buyer would expect a product with a longer shelf life. In these cases, consumers forget that they are "saving" wholesome food by deciding on a lower price, but practically intended for direct consumption. In this aspect, information and promotion campaigns of applications supporting the fight against food waste require significant improvement. It should be mentioned that, unfortunately, there are offers of products whose shelf life has not yet expired, but they have clear signs of deterioration (e.g. mold, rancid smell). Such cases affect, on the one hand, the disqualification of the seller by the consumer, but on the other hand, they also discourage the application itself, despite the fact that it is only a link between the seller and the consumer.

Analyzing the cross-section of respondents who took part in the presented survey, it can be concluded that the most interested in using mobile applications are young people (up to 40 years of age) living in cities with over 200 000 inhabitants (greater availability and diversity of the mobile shopping offer) and characterized by average income per person in the household (Table 1). The indicated respondents should undergo detailed consumer research using random methods. The obtained research results can indicate both the strengths and weaknesses of the current system, which serves to develop the applications themselves and the idea of preventing food waste promoted by them. Summing up the research results, it can be stated that the discussed applications face a huge development potential, however, requires constant monitoring of consumer attitudes and needs for continuous improvement. There is a need of raising awareness of both parties of the purchasing process regarding the idea of their functioning.

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ASSESSMENT OF SUSTAINABILITY AND FOOD SECURITY IN SELECTED EUROPEAN COUNTRIES AND REGIONS

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DOI: 10.56091/CTQS.Qual-27

Abstract

In the current period of unstable socio-economic situation and the impacts of climate change, even the countries of the European Union are increasingly exposed to the risk of food insecurity. When evaluating food security in the article, we base ourselves on two pillars – access and sustainability which include seven relevant indicators. Namely, share of food consumption expenditures, gross domestic product, prevalence of moderate or severe food insecurity, median equalised disposable income, government support to agricultural research and development, agricultural factor income per annual work unit and ammonia emissions from agriculture. Based on the mentioned pillars, the goal of the research is the assessment of food security through the construction of food security index for 26 countries of European Union in the time 2015-2020. The data are collected from the FAO, World Bank and Eurostat databases. From a methodological point of view, a multidimensional statistical method – principal component analysis – is used to determine the weights of the indicators within each pillar. The achieved results are compared with the published Global Food Security Index. Based on the constructed Food Security Index, we can see, that the EU's most food secure nations are Ireland and Denmark, while Bulgaria and Romania have the highest levels of food insecurity.

The Czech Republic is the most food secure country among Visegrad group. It is followed by Slovakia, Poland, and Hungary, which has the lowest level of food security index. Slovakia scores similarly to Poland in terms of the indicators that are tracked. We discovered anomalies in the agricultural factor income metric. Poland's indicator level reached 36% of Slovakia's (19,371 euro per annual work unit) in 2020. Poland's government expenditures on agricultural R&D that year (2.9 euro per capita) were, on the other hand, 24% higher than Slovakia's.

Keywords: access, correlation analysis, food security index, principal component analysis, sustainability

Introduction

The ability to access high-quality, nutritionally balanced food is critical for human health. Adequate food access can benefit economic growth and job creation, poverty reduction, health and healthcare, commercial prospects, and the expansion of global security and stability. Poverty is typically the root

cause of food insecurity, making it difficult for nations to expand their agricultural markets and economies. Agriculture expansion is at least twice as successful as other sectors in decreasing poverty (NIFA, 2022). Food security and sustainability share several characteristics. They are broad and insightful concepts, designed to frame and constitute common objectives for the international community, that are used by many scientific fields as well as lay groups like non-governmental organizations and governments, who frequently create their own definitions. There have been more formal attempts to connect the two concepts over time (Berry et al., 2015).

The study's goal is to capture both the concepts of food insecurity and sustainability in European countries using a complex index created from socioeconomic and agricultural determinants of food insecurity. Simultaneously, one of the main aims is to analyse changes between 2015 and 2020, as well as to compare the Slovak Republic to the best and worst countries in terms of food insecurity.

Connection between food security and sustainable development

Due to economic and ecological crises, the idea of sustainable development came to the attention of the world in the second half of the 20th century, and the term has since been given many different definitions (Buzko et al., 2019; Daly, 2013; Stoddart et al., 2011; Todaro et al., 2011). The Brundtland Report, published by the World Commission on Environment and Development, contains the most well-known and internationally recognized definition. According to the members of the commission, sustainable development is "development that enables the satisfaction of needs without jeopardizing the claims of future generations to satisfying their needs" (Brundtland, 1987).

Food security definitions have evolved over the last thirty years to reflect changes in political thinking. During the World Food Conference in the mid-1970s, the first concept of food security was developed. Food supply – ensuring the availability and price stability of basic foods at the international and national levels – has been defined as "availability of adequate global food supplies of basic foods at all times to maintain and continuously expand food consumption and to compensate for fluctuations in production and prices" (World Food Conference, 1974). The concept of food security should be more focused on the issue of access to food because the availability of food is not a guarantee of access to food and food rich in calories is not a guarantee of a balanced diet (Pinstrup-Andersen, 2009). The Food and Agriculture Organization's analysis of the issue of access to food in 1983 led to an update of the definition of food security based on the balance between the demand and supply sides of the equation, which included the individual and family levels in addition to the regional and national levels of aggregation. The essence of food security is to "ensure that all people have, at all times, physical and economic access to the basic food they need" (FAO, 1983). During the following years, the pillars of utilization (FAO, 1996) and stability were added to the definition of food security

and the phrase “four pillar of food security” was used for the first time (FAO, 2009). However, there are some problems with visualization of food security dimensions by equal pillars. Firstly, the dimensions are interrelated and interdependent and the pillars cannot catch the linkage between them. Secondly, there is also the weighting problem, because the pillars do not have equal importance and the weighting process should be context and country specific (Decancq & Lugo, 2013; Dobbie & Dail, 2013). When evaluating food security, sustainability should be considered as part of the long-term time dimension. From this viewpoint, the idea of sustainable diets can play a significant role as a goal and a means of preserving dietary balance and health while assuring the sustainability for long-term food security. Future food insecurity could be directly caused by current policies and initiatives if sustainability is not included as an official aspect of food security (Berry et al., 2015). The relationship between food security and sustainability is reciprocal. Long-term food security can be viewed as requiring sustainability. Food availability and biodiversity preservation are both dependent on the environment, particularly the climate and the availability of natural resources (Sperling & McGuire, 2012). Sustainable agriculture is becoming a concern in various countries as it incorporates migratory, technological, environmental, and economic aspects of food security. In sustainable agriculture, innovative organic farming practices are utilised, particularly in industrialized EU states where food security policy is largely focused on supporting healthy lifestyles and organic commodities (Grosso et al., 2020; Petrunenko et al., 2021).

Materials and methods

Data for the research were drawn from the FAOSTAT and EUROSTAT databases. The subjects of the analyses are 26 EU countries, excluding Luxembourg, in the years 2015 and 2020. Luxembourg was omitted from analysis, because it appeared to be an outlier, particularly when looking at GDP. For this reason, the given country was not considered in the analyses. The Covid-19 pandemic hit the European continent in the year 2020. Due to the comparison of the effects of COVID 19 from the perspective of the topic, the analyses are therefore performed during two time periods. Since the Covid 19 epidemic peaked in Europe in 2021, it would be more interesting to compare changes with this year. However, because there were no data for this year in the FAO database, we used 2020 for our analyses.

Food security was assessed based on the access pillar from FAO according to the following 4 relevant indicators: food consumption expenditures (share of consumer expenditures spent on food of total consumer expenditures in %), gross domestic product (per capita in purchasing power standard), prevalence of moderate or severe food insecurity (percentage of people in the population who live in households classified as moderately or severely food insecure) and median equivalised net income

(eur). Sustainability was assessed based on the following 3 relevant indicators: government support of agricultural research and development (eur per inhabitant), agricultural factor income per annual work unit (eur per annual work unit) and ammonia emissions from agriculture (kilograms per hectare). According to Eurostat (2017), the household income is "equivalized," or adjusted for household size and composition, to express the incomes of all households in comparable terms. Equivalised disposable income is a measure of the financial resources at a standardized household's disposal.

The min-max approach was used in the research to normalise the data to the same direction of development. The obtained values therefore fell within the range [0; 1], where the value 0 denotes the nation with the lowest indicator value and the value 1 denotes the country with the greatest indicator value among the analysed countries. Every year, the data is modified in this manner. The indicators were adjusted to the growing direction of development, meaning that the higher the indicator value, the better the country is perceived from the perspective of the indicator under analysis. Sustainability and food security were assessed based on our summary Food Security Index (FSI).

We considered the various weights of each indicator when developing the index. The methodology of factor analysis was used to establish the indicators' relative contribution to the influence on the summary index. We used Principal Component Analysis to estimate the factor analysis model. We used the eigenvector values after varimax rotation to calculate the weights. Rotation is a standard step in factor analysis, it changes factor loadings and thus interpretation. We used the approach based on Nicoletti et al. (2000) to calculate the weights. It is a method of aggregating individual indicators with the largest loading of factors into the indicator's intermediate composite. Individual temporary composites are then aggregated by assigning each one a weight equal to the data set's proportion of explained variance. The acquired values are then multiplied by the weight of the relevant factor. The individual indicator weights are modified so that the sum is equal to 1.

The aggregate Food Security Index was then calculated according to the relationship:

$$FSI = \sum_{k=1}^n w_j * i_{j,k}$$

where:

FSI – is the Food Security index,

w_j – is the weight of j-indicator, $j=1, \dots, 8$,

$i_{j,k}$ – is the value of the j-indicator of k-country after min-max normalization.

Results and discussion

In the presented research, the EU-26 countries are evaluated based on selected indicators of food security and sustainability. The composite food security index was developed, whose creation considered the various weights of the examined indicators. The weights were determined using Factor analysis based on Principal Component Analysis. Table 1 displays the eigenvalues of the Correlation Matrix for the years 2015 and 2020. According to the table, the first two factors appear to be sufficient for both periods, with the proportion of the original data variability explained by these factors being roughly the same (year 2015: 79.3% variability and year 2020: 80.1% variability). To create indicator weights, we only used Factor 1 and Factor 2.

Tab. 1. Eigenvalues of the Correlation Matrix

Eigenvalues of the Correlation Matrix: Total = 7 Average = 1						
	2015			2020		
	Eigenvalue	Proportion	Cumulative	Eigenvalue	Proportion	Cumulative
1	4.413	0.630	0.630	4.540	0.649	0.649
2	1.136	0.162	0.793	1.064	0.152	0.801
3	0.573	0.082	0.875	0.598	0.085	0.886
4	0.416	0.059	0.934	0.385	0.055	0.941
5	0.211	0.030	0.964	0.198	0.028	0.969
6	0.150	0.021	0.986	0.153	0.022	0.991
7	0.101	0.015	1.000	0.062	0.009	1.000

Source: own processing using SAS Enterprise Guide.

Table 2 shows the eigenvector values following Varimax rotation, as well as the explained variability values, which were used to recalculate the proportion of individual factors from the two factors under consideration. The weights were derived using the values of the eigenvectors, which were determined using Principal Component Analysis. The weights are built using the squared values of the eigenvectors, which represent the proportion of the total unit variance of the indicator explained by the factor. The values of the weights calculated in this manner can be found in the last two columns of Table 2.

Tab. 2. Eigenvectors after rotation Varimax

	Rotated Factor Pattern				Weights	
	Factor1 2015	Factor2 2015	Factor1 2020	Factor2 2020	2015	2020
Food Consumption Expenditures	0.781	-0.485	0.865	-0.347	0.141	0.146
Gross Domestic Product	0.879	-0.308	0.885	-0.148	0.179	0.153
Prevalence of Moderate or Severe Food Insecurity	0.512	-0.665	0.655	-0.563	0.061	0.084
Median Equivalised Net Income	0.878	-0.360	0.913	-0.290	0.179	0.163
Government Support of Agricultural R&D	0.883	0.209	0.877	0.163	0.181	0.150
Agricultural Factor Income	0.607	-0.539	0.747	-0.202	0.085	0.109
Ammonia Emissions from Agriculture	0.017	0.864	-0.059	0.932	0.173	0.196
Expl. Var.	3.565	1.983	4.125	1.478		
Var./Total	64.3%	35.7%	73.6%	26.4%		

Source: own processing using SAS Enterprise Guide.

The results in Table 2 show, that in 2015, the variables of Government Support of Agricultural R&D (0.181), Gross Domestic Product (0.179), and Median Equivalised Net Income (0.179) gained the highest weight in the development of the Food Security Index. In 2020, the indicator Ammonia Emissions from Agriculture (0.196) had the most significant impact, followed by the three variables that had a major impact in the creation of FSI in 2015 (Median Equivalised Net Income, Gross Domestic Product, and Government Support of Agricultural R&D). The aggregate Food Security Index was calculated for each nation based on the weights constructed in this manner. Table 3 shows its values as well as the ranks of the nations studied in both years. The change in ranks between 2020 and 2015 is shown in the last column.

In terms of evaluation of sustainability and food safety among European countries, Ireland and Denmark are at the top. These countries were ranked first and second in both years. Germany ranked third in 2020, up two spots from 2015. Bulgaria and Romania, on the other hand, finished last in both years. In the case of the Slovak Republic, the position deteriorated by two places; in 2015, Slovakia was ranked 15th; by 2020, it was ranked 17th. Slovakia was ranked second to the Czech Republic in the V4 group of countries (shown in yellow in the Table 3). In 2020, it was ranked 5 places lower than the Czech Republic, but only two places lower in 2015. In terms of sustainability and food security, the situation in Hungary and Poland is worse than that of Slovakia, according to the V4 countries.

Tab. 3. Values of Food Security Index in 2015 and 2020

Country	FSI 2015	FSI 2020	Ranking 2015	Ranking 2020	2020–2015
Ireland	0.84	0.86	1	1	0
Denmark	0.81	0.80	2	2	0
Germany	0.68	0.74	5	3	+2
Netherlands	0.64	0.71	7	4	+3
Finland	0.74	0.68	3	5	-2
Austria	0.65	0.66	6	6	0
Sweden	0.68	0.65	4	7	-3
Belgium	0.60	0.64	9	8	+1
France	0.63	0.62	8	9	-1
Spain	0.58	0.58	10	10	0
Italy	0.51	0.52	11	11	0
Czechia	0.44	0.49	13	12	+1
Cyprus	0.46	0.48	12	13	-1
Estonia	0.40	0.45	14	14	0
Slovenia	0.39	0.44	16	15	+1
Greece	0.37	0.40	17	16	+1
Slovakia	0.40	0.40	15	17	-2
Latvia	0.36	0.39	18	18	0
Hungary	0.34	0.38	20	19	+1
Poland	0.33	0.37	21	20	+1
Portugal	0.36	0.36	19	21	-2
Lithuania	0.29	0.35	24	22	+2
Croatia	0.33	0.32	22	23	-1
Malta	0.33	0.31	23	24	-1
Bulgaria	0.27	0.28	25	25	0
Romania	0.18	0.22	26	26	0

Source: own processing using SAS Enterprise Guide.

Table 3 also depicts the changes between 2015 and 2020. Countries with a positive change (improvement) in 2020 compared to 2015 are highlighted in green. Countries with the opposite tendencies (deterioration) are highlighted in red. Landscapes are unchanged without colour, which means their position was the same in both years studied. There were no significant differences between the

given years, according to the given column. The countries have improved, or they have dropped two positions. In nine countries, there was no change.

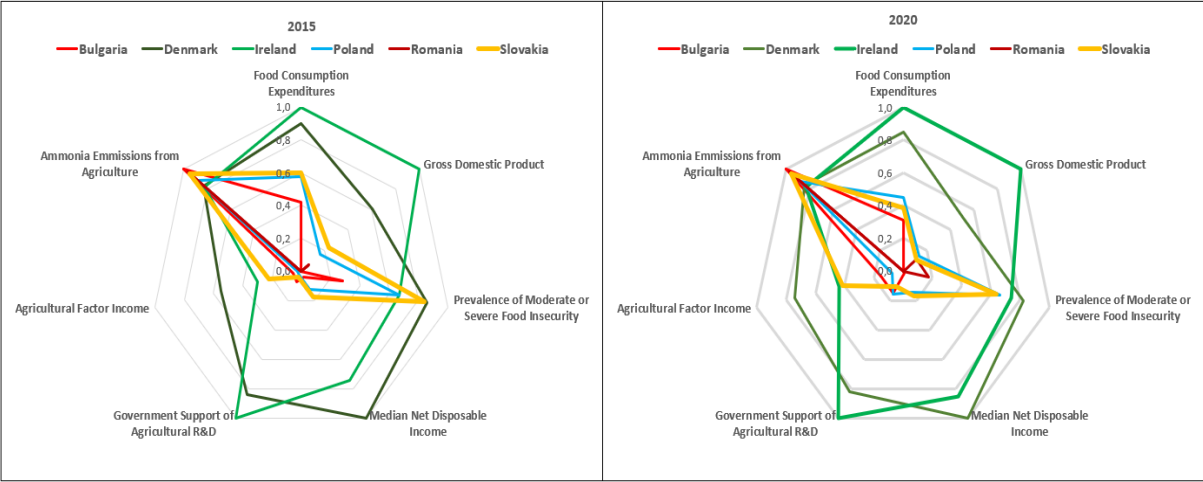


Fig. 1. Spider Plots of selected European countries based on analysed indicators in 2015 and 2020
Source: own processing.

In Figure 1, the spider plots were used to compare selected groups of countries in terms of individual indicators of sustainability and food security in 2015 and 2020. The plots show the best countries (Ireland, Denmark), the worst countries (Romania, Bulgaria), Slovakia, and Poland, that were chosen from the Visegrad group. We used min-max normalization to recalculate the original data, which adjusted them to the same direction of development. Adjusted indicators take on values from the range 0 to 1, with a higher value indicating a better position in the set of analysed countries. The distinction between the best and worst countries is obvious at first glance. Slovakia and Poland are in a similar situation. The countries Romania, Bulgaria, Slovakia, and Poland are better than Ireland and Denmark only in terms of one indicator, which is ammonia emissions from agriculture. However, this is not due to special attempts taken by these countries to reduce agricultural emissions, but rather due to lower level of animal production in those countries.

Conclusions

Food security is a subject that is receiving more attention, not just in relation to developing countries but also in Europe. Whether on a global, a national, a regional, or a home scale. Typically, a combination of indicators including the four aspects of food security—availability, access, utilization, and stability – is used to monitor countries and compare them. The Global Food Security Index is calculated using the values of the specified indicators. In addition to these pillars, food security is also closely linked to sustainability, and they influence each other. The study's goal was to assess sustainability

and food security in selected EU countries over two time periods (2015 and 2020) using a multidimensional perspective, and to create a composite Food Security Index based on Principal Component Analysis. For the research, we have chosen seven indicators from two dimensions – access and sustainability. The data were transformed via min-max normalisation. The findings show that there were no significant changes in individual countries' positions during the studied time periods. The most developed EU countries held the best positions. Eastern and Central European countries were at the bottom of the list. We can only evaluate positively the lower production of ammonia emissions from agriculture in this group of countries, which is primarily due to lower animal production in these countries. From an environmental point of view, it will be necessary to take measures in the future to ensure sufficient food production to feed the population with lower ammonia emissions from this sector of the national economy.

Acknowledgments

The research presented in the article was supported by the VEGA project: Challenges for ensuring food security in Europe in the 21st century – key factors, socio-economic and environmental contexts, No. VEGA 1/0755/21.

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A REVIEW OF SELECTED ETHICAL CONSIDERATIONS REGARDING RESEARCH IN NEUROMARKETING AND NEUROMANAGEMENT

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DOI: 10.56091/CTQS.Qual-28

Abstract

This paper aims to identify and analyze selected ethical issues related to neuroscientific research used in marketing and management. Though prevalent criticisms of neuromarketing include unethical research practices, unethical applications of technology, and manipulations of consumers, yet, despite these criticisms, the volume of academic research in neuromarketing and related areas has grown steadily as does number of research and consulting firms worldwide. With the growth of the field, criticisms and doubts of neuromarketing's purported power also have grown. Some of these fears and doubts are not distinctive only of neuromarketing, because they do not involve any new controversy beyond that attributable to traditional marketing. It turns out that, most of the new ethical threats that are attributed to neuromarketing are unrealistic because powers are attributed to neuromarketing that are not really there. In fact, only a few ethical issues arise that is both distinctive and realistic. The commonly perceived potential ethical issues are fear that neuromarketing may render consumers' choices completely predictable and fear among consumers that neuromarketing can be used to influence their choices and decisions, putting them out of control via the brain's unique "buy button".

The paper focuses mainly on ethical aspects associated with introducing neuroscience into marketing research that are still waiting to be solved, such as observance of methodological rigor, transparency, quality certification and maintaining the privacy (medical data) of the tested persons.

In conclusion, we emphasize that it is possible to improve the credibility and trust in neuroscience research applied to both neuromarketing and neuromanagement, although no ethical concerns regarding neuromanagement have been raised in the literature so far.

Keywords: consumer neuroscience, ethics, neuromanagement, neuromarketing

Introduction

The Hippocratic Oath is a code of ethics defining correct behaviour by physicians, which they are required to commit themselves to before being accepted into the profession. Essentially, it was the

first code of ethics for any profession. From the Ancient Greece time, it subsequently evolved, but the current code still embodies many of the core injunctions of the original code. The development of neuroscience and neurotechnology could be subsumed with little modification under formulation of the Hippocratic Oath, extending this precept to a commitment not to harm the health and well-being of people (Gare, 2022). Advances in neuroscience increasingly challenge long-held views of the self and the individual's relationship to society. The exponential increase in neuroscientific research, the commercialization of cognitive neuroscience, and the increased attention being paid to public understanding of science all illuminate the important role of ethics in neuroscience (Illes & Bird, 2006).

Advances in neuroscience research have changed the ways in which the relationship between brain and behavior are studied and conceptualized. As neuroscience is increasingly used to, or proposed as, a means of controlling behavior, through criminal and civil legal systems, researchers have an obligation to articulate the assumptions, limits, and ecological validity of their findings. Yet, neuroscience researchers rarely describe the assumptions underlying the studies, the generalizability of the findings and most importantly, the limits of its applicability to real world behavior (Freedman, 2023).

The vast arena of neuroscience and its applications, facilitated the transition, evolution from economics, management, leadership and marketing to neuroeconomics, neuromanagement, neuroleadership, neuromarketing neurofinance, neurocoaching, neurostrategy and neurocommunication, which allows the access to a broad spectrum of knowledge, that confirms or denies hypothesis and assumptions, with various possibilities of applications (Palincu et al., 2020). However, there is a need to differentiate between, on the one hand, general concerns regarding the application of neuroscience to social science from, on the other hand, specific critiques regarding the application of neuroscientific concepts, theories, and tools to consumer research and neuromarketing (Braeutigam & Kenning, 2022).

The application of neuroscience in the field of marketing is attracting companies with the prospect of an extensive understanding of consumer behavior. Neuromarketing can be considered as the use of neuroscience and physiological research techniques to gain new insights into consumers' behavior, preferences, and decision making, as well as other aspects of human cognition and behavior related to marketing (Stanton et al., 2017). Neuromarketing is frequently distinguished from consumer neuroscience by restricting the former to industry applications and the latter to academic research (Plassman et al., 2012). However, some authors seem to treat the terms neuromarketing and consumer neuroscience as synonyms. While consumer neuroscience explicitly refers to a scientific approach, neuromarketing is the "application of the findings from consumer neuroscience within the

scope of managerial practice". In line with this distinction, consumer neuroscience and neuromarketing rely on the same methods but are committed to different objectives: The former focuses on basic research, while the latter is concerned with practical applications (Hensel *et al.*, 2017).

Therefore, in this this paper we will use the term "neuromarketing" generally, and specify when we are referring to differences that are peculiar to academics or industry.

Academic (consumer neuroscience) and industry neuromarketing have very different goals. For academics, a primary goal is public dissemination of knowledge, as seen in the publishing of protocols and data in peer reviewed journals. For industry, a primary goal is to develop a comparative advantage of one's competition, which leads to private collection of data and development of proprietary analysis approaches. Academics and industry also have different approaches to interpreting and implementing results in guiding future practice. Academics tend to run experiments and evaluate their results using stringent thresholds that protect against the possibility that their findings occurred by chance and are not representative of a truly significant result (for example, a tolerance of less than a 5% chance that the experimental result is untrue is commonly used). In industry the key is forecasting (for example, a 75% likelihood of predicting an outcome can be a gamble worth taking when a managerial decision involves millions of dollars) (Stanton *et al.*, 2017).

Ethical issues were first classified into two groups: (1) protection of various parties who may be harmed or exploited by the research, marketing, and deployment of neuromarketing and (2) protection of consumer autonomy if neuromarketing reaches a critical level of effectiveness (Murphy *et al.*, 2008). Later, Hensel *et al.* (2017) gone further and identify five points within these two groups (1) protection of research subjects, (2) protection of vulnerable populations from marketing exploitation, (3) full disclosure of goals, risks, and benefits, (4) accurate media and marketing representation, and (5) internal and external scientific validity.

The main aim of the paper was to identify and analyze selected ethical issues related to conducting neuroscientific research used in marketing and management. In our paper we focus mainly on ethical aspects associated with introducing neuroscience into marketing that are still waiting to be fully solved, such as observance of methodological rigor, transparency, quality certification and maintaining the privacy (medical data) of the tested persons.

What are neuromarketing and neuromanagement?

Neuromarketing can be considered as the use of neuroscience and physiological research techniques to gain new insights into consumers' behavior, preferences, and decision making, as well as other aspects of human cognition and behavior related to marketing (Stanton *et al.*, 2017). Neuromarket-

ing seeks information and insights beyond that revealed by traditional techniques such as surveys, focus groups, experiments, and ethnography – with the goals of enhancing marketing theory and practice (Plassmann et al., 2015). Sometimes neuromarketing is attempting to improve the accuracy of predictions of consumer preferences and behavior when combined with traditional techniques (Boksem & Smidts, 2015).

Is the use of psychological and neuroscientific methods for neuromarketing research always aligned with the principles of ethical research practice? Some neuromarketing endeavours have passed from informing consumers about available options, to helping to market as many products to consumers as possible. Needs are being engineered, using knowledge about the human brain to increase consumption further, regardless of individual, societal and environmental needs and capacities. In principle, the ground ethical principle of any scientist is to further individual, societal and environmental health and well-being with their work. If their findings can be used for the opposite, this must be part of the scientist's considerations before engaging in such research and to make sure that the risks for misuse are minimized (Christense et al., 2022).

In view of many authors, it is critical to note that neuroscience methods and the data they yield should not receive privileged status as a research method in marketing or any other behavioral discipline (for example: Braeutigam & Kenning, 2022; Hensel et al., 2017; Stanton et al., 2017). In contrast to these views, neuromarketing companies commonly claim that neuroscience provides a golden key to the brain that can unlock hidden secrets about consumer preferences. They claim to have found what others fear as a “buy button” in the brain (Stanton et al., 2017). However, it seems that neuroscience techniques still are but one of the many methods that allow for refining and improving predictions of consumer behavior. In many applications, traditional marketing research techniques will still account for the greatest portion of the variance in consumer behavior.

The observed rapid changes that take place at the global level require organizations to develop new strategies to cope with the current challenges. Neuromanagement is a scientific approach of management, which explores the managerial, economic and behavioral processes, from the perspective of the brain's activity and of the mental processes (Palincu et al., 2020). Neuroeconomist Paul J. Zak used the term Neuromanagement to describe how neuroscience findings can be used to create organizational cultures that motivate employees, cultivate trust, positive experiences, and generate a high level of organizational performance (Zak, 2004). In the last years, progress in social neuroscience and neuromanagement, have generated new knowledge that can be used also by organizational leaders to better coordinate the teams they work with, to communicate more efficiently and so on (Palincu et al., 2020).

Neuromanagement can be also considered as a subdiscipline of neuroscience and aims to explore the activities of the human brain and mental processes in situations when people face management situations. For this aim neuromanagement is using cognitive neuroscience, in conjunction with other scientific disciplines and neuroimaging technologies. The present research in the field of neuromanagement concerns research areas such as decision-making neuroscience, which offers a new perspective and new insights into human decision-making and general social behaviors, and how they impact management and economic processes.

One of the researchers in neuroscience, with very important contribution in neuromanagement, related to self-control and fear control or how the brain works in certain situations is Harvard psychiatrist and executive coach Srinivasan Pillay (Pillay, 2011). In his book “Your Brain and Business: The Neuroscience of Great Leaders”, Pillay provided us details related to brain functioning in professional life and addresses issues such as: positive and negative thinking, social intelligence and effective relationships, innovation and intuitive, formation of action oriented ideas, area formation of actions oriented towards change, training certain regions of the brain and mental processes.

While neuromanagement can also be simply defined as a new interdisciplinary, developing field that uses neuroimaging techniques to identify the neural substrates associated with decisions about people, human resources and associated behaviors, in organizational activity (Zak, 2004), neuromarketing can be simply considered as an interdisciplinary, developing field that uses neuroimaging and physiological tools to record the neural correlates of consumers’ behavior (e.g., decision-making, emotion, attention, and memory) toward marketing stimuli such as brands and advertisements.

Physiological tools such as ET (*Eye tracking*), GSR (*Galvanic skin response*), EMG (*Electromyography*), and ECG (*Electrocardiography*) can provide beneficial insights about the physiological correlate of consumer behaviour, while neuroimaging tools such as fMRI (*functional Magnetic resonance imaging*), PET (*Positron emission tomography*), MEG (*Magnetic encephalography*) and EEG (*Electroencephalography*) enables to capture the neural correlates of emotion processes (e.g., pleasure, motivations, and arousal), cognitive processes (e.g., attention, recall, recognition, and memory) toward marketing stimuli such as advertisements and brands. It means that marketers and advertisers may be able to identify the attractiveness and aversion aspects in advertising campaigns before using them in the real world (Alshari et al., 2021).

Both neuromarketing and neuromanagement apply nearly the same neuroscientific tools and methodology. Therefore, ethical responsibility mainly refers to practitioners of both disciplines. Fortunately, literature studies show that so far neuromanagement is not burdened with moral and ethical doubts, unlike neuromarketing, at least when it comes to attempts to covertly manipulate people.

Selected ethical issues related to neuromarketing research conducted by neuromarketing companies

In the last few years, most of the discussions related to neuromarketing and ethics have focused on the commercial use of neuromarketing (Hensel et al., 2017). As could be expected, the main issues were consumer manipulation and the lack of transparency in this field (Fishe et al., 2010). Another important ethical issue refers to serious shortfalls in consumer autonomy (Murphy et al., 2008). Ramsøy (2014, p. 498) strongly stated “while academic researchers are, or should be well versed in ethics codes of conduct, this is often not the case for commercial uses of neuroscience”. Just because of the potentially commercial nature of the studies, some people fear that the data collected could be used unethically (Satel & Lilienfeld, 2013). The authors stated “in 2011, a consortium of consumer protection groups filed a complaint with the Federal Trade Commission against Frito-Lay for allegedly using neuromarketing” in order to determine emotional and subconscious reactions that would help “promote high-fat snack food to teens” (Satel & Lilienfeld, 2013, p. 43). This clearly demonstrates that conducting neuromarketing research poses several serious ethical issues that marketers (both academics and practitioners) need to take into account.

That is why for the development of a regulatory ethical guideline in neuromarketing was called (Ulman et al., 2015). Most of the already existing ethics codes seemed to be rather too general to clarify certain issues that are important for marketers. Accordingly, Hensel et al., (2016) created an extended guideline, the “EGNM” (Ethical Guideline in Neuromarketing) of a common piece, the NMSBA (Neuromarketing Science & Business Association) Code of Ethics, which provides guidelines on conducting neuromarketing studies ethically. The EGNM guideline from Hensel et al. (2016) was more refined and improved on the basis of consensus with the answers provided by interviewed neuromarketing practitioners (Hensel et al., 2017).

Apart of five main ethical issues mentioned in Introduction, Hensel et al. (2017) discuss the five additional codes evaluated in their interviews:

(1) sufficient sample size for neuromarketing studies (for example, the optimum sample size for EEG research is around 30–40 people),

(2) specific quality check of neuromarketing data (“garbage in, garbage out.” This true, as far as neuromarketing research is concerned – if the data used as input is not appropriately cleaned and processed, the results cannot be trusted. The quality of the recorded data derives from the fact that in neuromarketing research, one works with small samples),

(3) no agreement for neuromarketing studies for optimizing “unhealthy” products, e.g. tobacco or alcohol industry) (especially with respect to youth customers (the NMSBA Code of Ethics does not explicitly ban neuromarketing vendors from working for potentially controversial industries, like tobacco, alcohol, or politics),

(4) no use of non-scientific methods (when choosing between different tools or technologies, one needs to take into consideration publications and third-party assessments of those particular tools),

(5) studies with complex methods (e.g. fMRI, EEG) conducted by neuroscientists (neuromarketing companies initially developed around neuroscientists who were working in universities and had access to medical equipment and data analysis software. As decision-makers from the business world started to understand its commercial potential, more and more companies started to follow this trend. Nowadays, most neuromarketing firms employ neuroscientists either as full time staff or consultants. So, theoretically, the neuroscientists should to ensure that the claims and findings extrapolated from neuroimaging tools are grounded in science and that salespeople do not get carried away in promising clients that they are able to solve all manner of problems with neuromarketing research).

It seems that the most frequently raised concerns (claimed by the participants involved and the general public) – threats to consumer autonomy, privacy, and control – do not rise to meaningful ethical issues (have been overblown) given the current still limited capabilities and implementation of neuromarketing research. However, potentially serious ethical issues can arise from industry neuromarketing research practices themselves, which may be largely proprietary and opaque.

Adherence to methodological rigor

Scientific results are only valuable if the methods used to collect them are robust. Frequently, industry clients who hire neuromarketing companies to conduct research are not likely to have sufficient background knowledge to evaluate the methods used to collect and analyze neuroscience data. It should be taken into account that neuromarketing companies may be motivated to use poor research methods, collect insufficient sample sizes, employ insufficiently trained staff, etc., if they can convince the client that the data is useful to them. In addition, neuromarketing companies are highly motivated to exaggerate their capabilities and potential outcomes in order to attract customers. Neuromarketing companies lack peer review when reporting results to clients, and peer review can effectively guard against the risk of overstating of results. Thus, the established principles of the quality of work of neuromarketing companies and the results provided are not as clear as in the case of traditional methods of market research. Importantly, neuromarketing companies tend to maintain

proprietary control of the data they collect, and they also do not publish or share their data collection protocols. Obviously, this opacity means that the extent to which neuromarketing companies' data are valid, or in correspondence with their promotional claims, remains unclear. There would be an ideal situation, when neuromarketing companies that do not produce deliverables derived from rigorous methods would eventually be overtaken by firms that are using rigorous methods and producing valid and reliable data. Surely, such firms will better aid their clients in making accurate predictions in the marketplace through their higher quality research.

Unlike in industry, overstating results or capabilities is less likely in academia because the peer review publication system that is applied to published research is designed to ensure methodological rigor and accurate interpretation of results. However, academics are not free from performance incentives that can compromise data quality. There are numerous cases of academic researchers who published completely fraudulent data in a quest for tenure, promotion, and other incentives, which upon discovery has led to the retraction of many published journal articles (Stanton et al., 2017). So overstating is still possible in academia as it is in industry.

In an effort to address the greater risk regarding neuromarketing in industry, Stanton et al. (2017) propose that there would be benefits to clients and consumers if neuromarketing companies adopted policies of data and protocol transparency. Key aspects of such transparency would include information regarding the Institutional Review Board (IRB) that oversaw the study and the IRB full protocol associated with the study as well as procedures for gaining informed consent. It seems reasonable to assume that full transparency could lead to such a shift in the market that companies attract customers because of the rigorous and thoughtful way they use proven experimental procedures, not because they have developed new (and sometimes questionable) unique methods to measure brain function.

If neuromarketing companies wanted to switch to full transparency, a first step would be to adopt science advisory boards composed of scientists trained in the techniques that the company employs. It is likely that such an approach neuromarketing companies to maintain proprietary control of the data and method protocols. At the same time, it would allow for a higher level of methodological control. The obvious weakness of this approach is that members of scientific advisory boards (of any company, not just neuromarketing) are often motivated by money, which can lead to board hijacking. In reality the scientific review boards are not truly independent from the company. Independent scientific review boards would limit the possibility of bias arising from remuneration directly from the company.

Third-party quality certifications

The neuromarketing industry may also benefit from third-party quality certifications. Such certifications ensure ethical treatment, protection of subjects and methodological rigor are being strictly employed. For example, an external group could be organized to provide a certificate of quality that would enable consumers of neuromarketing research to make a more informed choice regarding the product that they are purchasing (Stanton et al., 2017). The need for this may be unique to neuromarketing research compared to traditional marketing research. This is because neuromarketing research methods are technically sophisticated and lack the feasibility of traditional marketing research methods (Hensel et al., 2017).

Private health information threats

Several aspects are taken into consideration when looking at the protection of test participants. One of them deals with the management of incidental results or clinical findings that are not directly related to the objective of the research. Other aspects look at the impact of high incentives on respondents, at the procedures in place to ensure informed consent and respondents' rights (e.g. the right to unilaterally terminate a study) (Murphy et al., 2008).

Neuromarketing data are of course close to private health information, which implies a different type of privacy threat. As an illustrative example we can consider the typical approach of collecting data about brain function using neuroimaging technology, say, fMRI, PET or EEG. It is known that fMRI data extracted from a neuromarketing experiment are highly contextualized and specific to the experiment at hand. In conjunction with fMRI is always measured the structural MRI, and participation in neuromarketing studies using fMRI would mean that companies have access to one's structural MRI data. Structural MRI, which creates a structural map of one's brain but does not measure brain activity (function), is more closely equated to other clinical private health information (such as HIV status, pregnancy, mental illness, cancer, and so on). Structural MRI can contain (private) information regarding pathology: neural system diseases, tumors, and structural malformations in the brain. Such information could potentially be used to discriminate in some form against individuals (non ethical behaviours). In the absence of informed consent, which is a significant problem in industry, the ways in which such data could be used or sold to other companies would be unclear and highly unethical to research participants and to some extent unregulated (Hensel et al., 2017; Stanton et al., 2017).

Described here private health information threats can not be generalized to traditional marketing research. Privacy breaches do not occur in traditional marketing research because participants can consciously and voluntarily disclose private health information in a survey. Therefore, we focused on

specific ethical issues that derive directly from the application of modern medical diagnostic techniques like neuroimaging that are currently used also in neuromarketing research.

Concluding remarks

In the industry neuromarketing research process several parties are involved: the beneficiary companies that commission the studies, the research agencies that carry them out and the participants (volunteers) involved. If research agencies fail to comply with ethical principles, both entities commissioning research and research participants may be potentially at risk. However, as a result of different sets of priorities and approaches, both academic and industry neuromarketing researchers face significant ethical challenges. Earlier raised threats related to autonomy, privacy, and control of consumer behavior do not seem so meaningful ethical issues given the current technical capabilities and implementations in neuromarketing research. When it comes to consumer welfare, ethical issues relate rather to the practical application of the findings from neuromarketing research within the scope of managerial practice and are a separate ethical issue.

Most of the new ethical dangers that are attributed to neuromarketing turn out to be unrealistic, because they assume that neuromarketing has powers that it cannot obtain in the near future. Only a few ethical issues arise that are both distinctive and realistic. The first commonly perceived, potential ethical issue is the fear that neuromarketing may render consumers' choices completely predictable. Similar criticisms regarding the prediction of consumer choice have been applied to traditional marketing research and practice, but are perhaps most accentuated in neuromarketing (Wilson et al., 2008). A second commonly perceived, potential ethical issue is the fear among consumers that neuromarketing can be used to go beyond prediction and influence consumer choice. It is argued that successful neuromarketing might rob consumers of control and make the marketed goods irresistible. Shaping consumers' choices is the goal of marketing generally, but does neuromarketing offer firms a unique and novel ability to find a "buy button" in the brain? No! (Stanton et al., 2017).

In this paper, due to size limitations, we have articulated only some identified common ethical concerns with neuromarketing research as currently practiced, focusing on the potential risks to test participants and the ethical decisions faced by companies and organizations. However, we pay attention on potentially serious ethical issues that may emerge from industry neuromarketing research practices, which are in high degree proprietary and opaque.

There are some possible steps that are able to mitigate associated ethical risks and to reduce the threats. The point which still unclear and needs the discussion on international level is the idea of determination of the neuromarketing as a separate science or as a subgroup of other. Neuromarket-

ing as all other research science should have clear and realistic regulation which will take into account all stakeholder of the research. With the appropriate legislative framework the situation of data leakages and overpowering the companies would be avoided. Plenty of countries and organizations started an implementation of research standards in order to solve problem of data privacy, misinformation of customers, legal consequences of using forbidden data and the human rights violation during neuromarketing research (Petropavlovskaya & Sydorov, 2021).

We believe that it will take much more time for neuromarketing research to be completely accepted, both academically and commercially. Regardless of its still pioneering role in the world of science, neuromarketing has already seen significant growth in the field of science through various forms of scientific research projects and articles, but also via direct applications in practice.

Neuromarketing is the new era, which as all other modern technologies has its own pros and cons. It has clear potential for positive influencing on society and consumers, what is rarely taken into account when discussing the ethical issues of neuromarketing. Referring to neuromanagement, we hope that in similar way will have positive impact on managed firms and organizations. Contrary to neuromarketing, with regard to neuromanagement, no ethical doubts have appeared in the scientific literature so far.

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FACTORS INFLUENCING THE QUALITY OF VOCATIONAL EDUCATION IN POST-SECONDARY SCHOOLS

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DOI: 10.56091/CTQS.Qual-29

Abstract

The objective of vocational education provided at post-secondary schools is to prepare students for work in specific professions. It is a series of didactic activities aimed at imparting a certain amount of theoretical and practical knowledge, and in particular specialised vocational skills, enabling graduates who have obtained a diploma confirming their qualifications to work in a given profession. Human capital, which is shaped by the education system and universal access to high quality education, is of key importance for progress and social and economic growth. This quality is conditioned by many factors, both economic and non-economic, and the task of the managers of the educational system units should be to strive to ensure high quality education.

The aim of this study was to identify the most important factors influencing the quality of vocational education in post-secondary schools in Poland.

The study used the technique of an individual, semi-structured interview conducted by telephone (CATI – Computer Assisted Telephone Interview). Non-probability sampling was used and 5 interviews were conducted with managers of public and non-public post-secondary schools in Poland.

All managers of institutions considered that a very important factor influencing the quality of vocational education is the level of teachers' salaries. They also pointed to the teaching resources of the school, above all the equipment of the vocational laboratories with modern technological solutions used in the given professions. Another important factor is the teaching staff consisting of teachers practising in the given professions, as well as systematic in-service training and self-education of teachers. None of the establishments has implemented a Quality Management System according to the requirements of the ISO 9001:2015 standard, and 3 of the 5 schools systematically carry out surveys of student satisfaction and expectations. The managers of the public institutions stated that an important element of the internal quality management system in their schools is the pedagogical supervision plan, in which they carry out inspections, support teachers and conduct observations of selected classes.

Keywords: education quality, vocational education, post-secondary school

Introduction

Vocational education is a set of activities and actions aimed at preparing students/scholars to work in specific professions. It involves providing them with a certain amount of theoretical and practical knowledge and specialised professional skills enabling them to obtain professional qualifications, perform work in a given profession and actively participate in the changing labour market (Dziewulak, 2020; Kupisiewicz & Kupisiewicz, 2009). Education in occupations of vocational education is realised in 4 types of post-primary schools, i.e.: industry school of the first degree, technical school, industry school of the second degree and post-secondary school. It may also be conducted at qualifying vocational courses and vocational skills courses (Journal of Laws 2019, item 316, as amended).

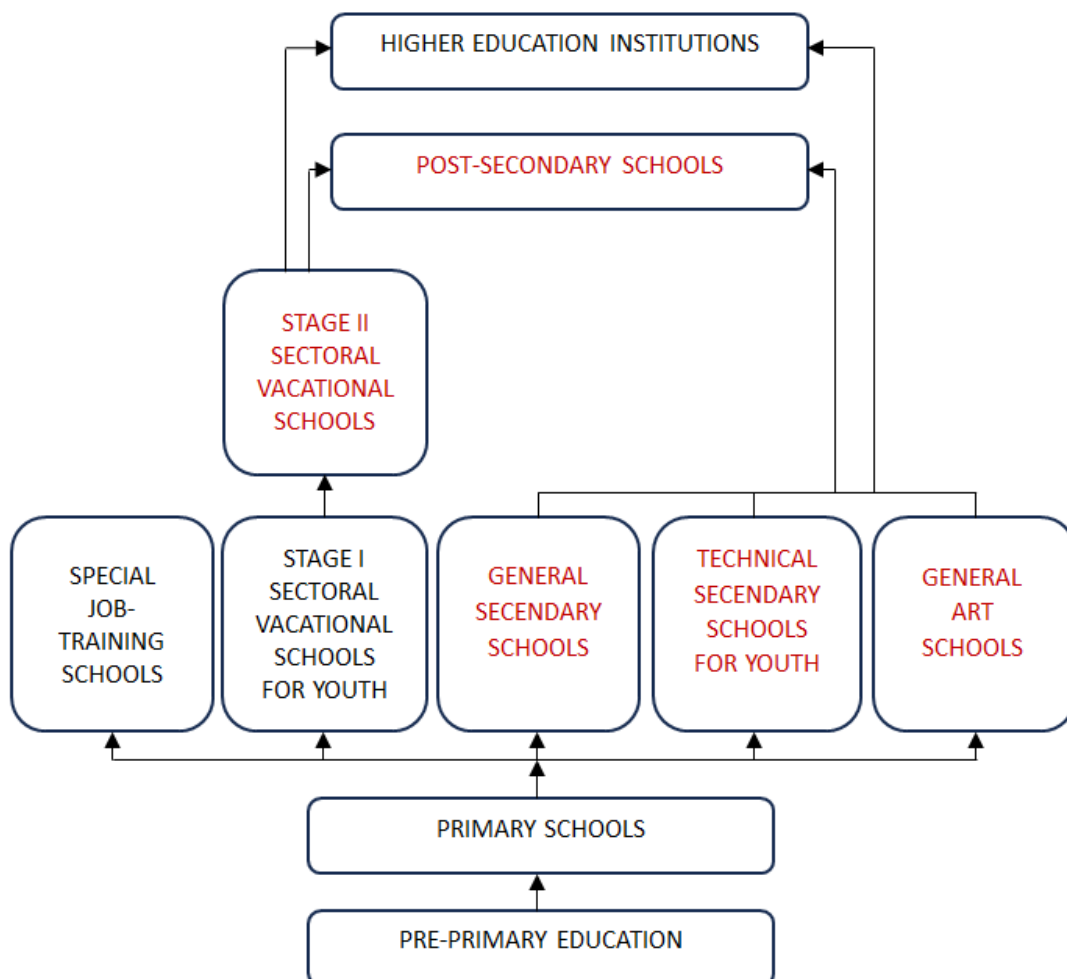


Fig. 1. Education in Poland by level of education

Source: own source based on Central Statistical Office.

Post-secondary school, according to Polish legislation, belongs to post-primary schools, educating individuals with secondary education or industry secondary education (Figure 1), and the teaching period in this type of school cannot be longer than 2.5 years. Education at a post-secondary school may be provided in the form of daytime, full-time or extramural education (Journal of Laws 2023, item 900).

Due to the fact that candidates for post-secondary schools may be persons with both general secondary education and secondary vocational education, these schools are perceived not only as a form of additional training and acquiring a profession, but also as a form of retraining, enabling people with secondary vocational education to change their profession. An additional advantage of post-secondary school is the possibility to realise the postulate of lifelong learning and the development of a knowledge-based society, for which continuous improvement is an important element (Garbalińska-Charchut, 2020).

Education in specific professions is conducted on the basis of the core curriculum for education in occupations of industrial education. Education is described in the form of expected learning outcomes, i.e.: knowledge, professional skills and personal and social competences (Journal of Laws 2019, item 991 as amended). Within individual professions of vocational education, specific qualifications have been created. This is a set of expected learning outcomes distinguished in the occupation, the achievement of which is confirmed by a certificate of professional qualification issued by a district examination board, after passing a professional examination for a given qualification. The occupations described in the classification of occupations of vocational education are single- or dual-qualification (Journal of Laws 2019, item 316 as amended). Completion of a post-secondary school makes it possible to obtain a professional diploma after passing professional examinations (Journal of Laws 2023, item 900). Students of the post-secondary school take the vocational examination voluntarily, after making a prior declaration. The exam is designed to test the knowledge and skills necessary to practise a given profession. The bodies supervising vocational examinations are the Central Examination Commission and the District Examination Commissions, which were established on 1 January 1998, under the Act of 25 July 1998 amending the Act on the educational system (Garbalińska-Charchut, 2020; Journal of Laws 1998 no. 117 item 759).

The Regulation of the Minister of National Education of 15 February 2019 on the general objectives and tasks of education in vocational education professions and the classification of vocational education professions presents 32 branches and the professions assigned to them, defines the general objectives and tasks of education in vocational education professions and presents the classification of vocational education professions. The regulation also takes into account professions previously

included in the classification of vocational education professions (Journal of Laws 2017, item 622, as amended), introduces changes in some professions and indicates new professions in the education system. The updated regulations are the result of consultations with representatives of individual specific industries, analysis of comments submitted by employers industry, specialists in a given industry and other labour market entities and institutions (Journal of Laws 2019, item 316 as amended; Journal of Laws 2017, item 622 as amended).

Educational regulations specify the types of post-primary schools in which education in a given profession can take place, allowing education of young people and adults in 237 professions. In post-secondary schools, it is possible to educate students in 10 industries, in 36 professions, where the training period lasts from 1 year to 2.5 years (Table 1).

Tab. 1. Industries and occupations in post-secondary schools

No.	Industry	Profession	Period of teaching (years)
1	Audiovisual industry (AUD)	Film and television production administrator	1
		Assistant film and television production	2
		Film animation technician	2
		Sound engineering technician	2
		Recording technician	2
2	Economy and administration industry (EKA)	Administration technician	2
		Archivist technician	2
		Postal and financial services technician	1
3	Hairdressing and cosmetics (FRK)	Podiatrist	2
		Cosmetic service technician	2
4	Precision engineering (MEP)	Optician technician	2
5	Security and safety of people and property (BPO)	Occupational health and safety technician	1.5
		Physical protection of people and property technician	2
		Fire-fighting technician	2
6	Horticulture (OGR)	Florist	1
7	Healthcare industry (MED)	Dental assistant	1
		Dental hygienist	2
		Medical assistant	1.5
		Orthoptist	2
		hearing care specialist	2

		Dental technician	2.5
		Electronics and medical informatics technician	2
		Electrocardiologist technician	2.5
		Pharmacy technician	2.5
		Massage therapist technician	2
		Orthopaedic technician	2
		Medical sterilisation technician	1
		Occupational therapist	2
8	Social welfare industry (SOP)	Disabled people assistant	1
		Elderly care assistant	2
		Assistant in a nursing home	2
		Childcare assistant	2
		Social work assistant	1
9	Information and communication technology (INF)	Technician in information technology for the blind and visually impaired	2
10	Water transport industry (TWO)	Marine engineer technician	2
		Maritime navigator technician	2

Source: own source based on Journal of Laws 2019, item 316 as amended.

A key element of vocational education is practical vocational training. In post-secondary schools it is organised in the form of practical classes and apprenticeships. These classes are aimed at the students' mastering of professional skills necessary to take up employment in a chosen profession, as well as at the deepening of knowledge acquired at school and the use of professional skills in real work conditions. An important aspect of practical vocational training is the opportunity to practise personal and social skills in contacts with real clients, especially in occupations related to service, caring, social assistance and other activities requiring teamwork or social contacts (Journal of Laws 2019, item 391).

Vocational training provided as part of compulsory education includes subjects in theoretical and practical education, with the proviso that the number of hours allocated for classes organised in the form of practical classes may not be lower than 50% of the hours allocated for vocational education (Journal of Laws 2019 item 639 as amended).

Tab. 2. Number of students and post-secondary schools in Poland

School year	Total number of post-secondary schools in Poland	Number of students in post-secondary schools in Poland
2021/2022	1357 (decrease)	232.7 thousand (increase)
2020/2021	1468 (decrease)	204.7 thousand (increase)
2019/2020	1638 (decrease)	188.4 thousand (decrease)
2018/2019	1981 (decrease)	222.3 thousand (decrease)
2017/2018	2167 (decrease)	239.3 thousand (decrease)
2016/2017	2331 (decrease)	251.8 thousand (decrease)
2015/2016	2355 (decrease)	258.8 thousand (decrease)
2014/2015	2382 (decrease)	264.5 thousand (decrease)
2013/2014	2467 (decrease)	278.8 thousand (decrease)
2012/2013	2735 (decrease)	334.2 thousand (decrease)

Source: own source based on Central Statistical Office.

Over the last 10 years, there has been a continuous decrease in the number of post-secondary schools in Poland, for many years a decrease in the number of students studying in this type of schools has also been observed, but in the last two years there has been an increase in the number of students studying in post-secondary schools. Analysing the data of the Central Statistical Office for the last 10 years, it can be observed that the majority of post-secondary schools (on average 80%) were managed by entities from the private sector (Central Statistical Office, 2021).

Human capital, which is shaped by the education system and universal access to quality education, is crucial for social and economic progress and growth. It is fundamental as it affects the rapid and sustainable development of countries and societies (Barro & Lee, 2013; Mankiw et al., 1992).

Quality is a complex and multidimensional term. Many definitions of quality have emerged over the years, with the origins of the term 'quality' dating back to ancient times. Attempts to define quality were made by Aristotle, Plato, Cicero, Descartes, Kant and others. The term has been considered from various angles, including philosophical, humanistic, sociological, technical, economic, marketing, from the consumer's point of view and from the producer's point of view, quality related to the product life cycle, quality in transformational terms (required quality, target quality, adjusted quality), quality according to D. Garvin (absolute, related to the product, related to production, quality from the user's perspective, quality formulated in relation to value), and quality taken narrowly – the so-called q (product quality), and broadly – Q (system quality). The concept of quality is also defined in ISO standards, according to PN-EN 9000:2001 'quality is the degree to which a set of inherent

properties meets the requirements'. Plato defines quality as a certain degree of perfection, while Crosby writes: "Quality is conformity to requirements". Taking into account these two simple definitions, we can see that quality is the high value of a product to meet customer expectations. Lao Tsu, a Chinese philosopher, described quality as continuous improvement (Wasilewski, 1998). In later years, the subject of quality was taken up and analysed by many researchers. They recognised the need to improve quality, to revise definitions and to determine methods of quality management. In the 20th century, W. E. Deming, J. M. Juran, P. B. Crosby, or G. Taguchi, regarded as authorities in the field of quality, presented their views and recommendations on raising quality level and its improvement (Gołębiowski, 2008).

There have also been many definitions of the term 'quality of education' over the years. Grudowski and Lewandowski (2012) are of the opinion that "educational quality can be defined as the degree to which the requirements for the educational process and its outcomes, formulated by stakeholders, are met, taking into account internal and external conditions." (Grudowski & Lewandowski, 2012).

The United Nations Educational, Scientific and Cultural Organization (UNESCO) provides a general definition of educational quality as: "a type of education that offers all young people, and other learners, competences that are adapted to the specific context in which they live and that enable them to participate actively in society; education that is adapted to life, to the aspirations and interests of learners, as well as their families and communities; and finally, education under the sign of social inclusion and respect for human rights." (UNESCO, 2006; Grudowski & Lewandowski, 2012).

The Education Law obliges schools and institutions to take the necessary measures to improve the quality of the work of the school or institution and its organisational development (Journal of Laws 2017, item 1611, as amended).

In the Polish educational system, ensuring the quality of education in primary and post-primary schools is included in the system of pedagogical supervision and is regulated by the Regulation of the Minister of National Education of 25 August 2017 on pedagogical supervision. The forms of pedagogical supervision are control and support. External assurance of the quality of education is performed by the Minister of Education and Science and other relevant ministers supervising specific types of schools, as well as school superintendents responsible for individual provinces, and the issues subject to control are defined in the directions for the implementation of the state's educational policy and in pedagogical supervision plans. Internal supervision of the quality of education is carried out by the headmaster in cooperation with the management staff. For this purpose, he or she draws up a pedagogical supervision plan for a given school year, in which he or she defines the scope and subject matter of inspections and teacher support activities. As part of internal quality assurance, the

headmaster also evaluates the work of teachers (Journal of Laws 2017 item 1658 as amended; Journal of Laws 2022 item 1822; European Commission, 2023).

The education system in Poland is subject to constant change, with new solutions being introduced to improve the quality of education and individualisation of teaching. Issues related to the quality of education in the era of intensive and diverse socio-economic and technological changes should be a priority of tasks undertaken by educational authorities, school authorities and managers of educational institutions. High quality vocational education is expected to provide a high class of specialists who can significantly contribute to the country's socio-economic development, meet the expectations of employers and the labour market (Hyżak, 2013; Bielawa, 2011).

There are many factors influencing the quality of vocational education in post-primary schools, all of which are important elements for improving the quality of school work. An important role in the educational process is played by the teacher/educator. His/her attitude, behaviour, involvement in the educational process, enthusiasm, creativity and passion for the profession greatly influence the quality of education at school (Tchórzewski, 1994). In order to achieve this, the teacher needs to ensure the students' sense of security, create an atmosphere of partnership, respect and dialogue that contributes to high quality work (Buchcic, 2014). The authors also point to appropriately selected and varied teaching methods to achieve the expected learning outcomes. Equally important in the pursuit of high quality education are students' talents and practical forms of classes, considered more effective in the assimilation of theoretical and practical knowledge (Hryniewicki, 2019; Polak, 2013).

The aim of this article is to identify important factors influencing the quality of vocational education in post-secondary schools as assessed by the managers of such institutions.

Materials and methods

The study was conducted by means of a diagnostic survey, using the CATI (Computer Assisted Telephone Interview) technique, individual semi-structured interviews conducted over the telephone. The planned interviews constituted a preliminary study. Before preparing the interview questionnaire, a literature search was conducted in order to establish the current state of knowledge on the issues under investigation. Bearing in mind the constant changes introduced in recent years in the area of educational law, the changing requirements of the labour market or the emergence of new technologies in the economy, the researchers decided to see if post-secondary school managers would identify new factors affecting the quality of vocational education not previously described in the literature. The research tool, in the form of an interview questionnaire, contained 6 main ques-

tions divided into 3 categories: opening questions (general, about the facility, aimed at relaxing the interviewee); questions on factors influencing the quality of vocational education in post-secondary schools; questions on the examination and control of the quality of education in the facilities. The selection of post-secondary schools for the study was random. The randomisation was based on the Ministry of Education's Register of Schools and Educational Institutions – RSPO. Individual interviews were conducted with 5 managers/directors of post-secondary schools; 3 directors of public schools and 2 directors of non-public schools participated in the survey. Respondents were informed that the data obtained would be used for research purposes only, and respondents would not be quoted by name or school name. The interviews were audio-recorded; respondents consented to the recording of the interviews on the condition that they would only be used for research purposes and would only be played back by the researcher. Due to the anonymity of the research and the impossibility of revealing data that could contribute to the identification of facilities, the names of the schools were coded (SP 1, SP 2, SP 3, SP 4, SP 5). In order to identify significant factors influencing the quality of vocational education in post-secondary schools, a qualitative study was carried out, where the basis for the analytical activities was qualitative material in the form of transcriptions of interviews – a form of written interview recording. The recorded interview was then subjected to analysis and interpretation. The first stage in analyzing the extracted material was coding, i.e. generating categories within the scope of the study area and attempting to combine them in order to look for connections between them. Initial coding began and, using a line-by-line coding strategy, the maximum number of categories covering the factual content of the empirical material was created. Categories were then selected for further analysis, limited to coding those variables that relate to the stated aim of the research (selective coding). The coding was completed when the selected categories were saturated, i.e. no new properties of these categories emerged (Glinska-Neweś & Escher, 2018).

Results and discussion

The first category of questions included in the interview questionnaire, enabled the interviewees to present the school's offer, fields of study, forms of education, present school activity and its organisation. These were introductory questions aimed at relaxing the interviewee. Table 3 shows the fields of study offered by particular schools assigned to the industries, in accordance with the Regulation of the Minister of National Education of 15 February 2019 on the general aims and objectives of education in vocational education professions and the classification of vocational education professions.

Tab. 3. Educational offers of the schools participating in the survey

No.	Post-secondary school	Number of fields of study offered	Number of industries
1	SP 1	14 professions	2
2	SP 2	12 professions	2
3	SP 3	12 professions	2
4	SP 4	15 professions	6
5	SP 5	25 professions	6

Source: own study.

All the schools in the survey offer daytime education both, full-time and extramural.

Later in the interviews, interviewees were asked about economic and non-economic factors influencing the quality of vocational education in post-secondary schools. Analysis of the interview transcripts showed consistency in the answers given in the area of several categories. All managers of educational facilities agreed that teacher salaries were a very important factor influencing the quality of vocational education. However, the follow-up questions showed that this opinion is formed only on the basis of information obtained from teachers employed in schools and general dissatisfaction with salaries in the education system, and not on the basis of research conducted in schools concerning, for example, the correlation between the salaries of teachers with particular levels of professional promotion and the quality of education in a given school.

Another factor common to all schools that influences the quality of vocational education is the school's teaching resources, also referred to by interviewees as the school's teaching base or teaching equipment. This is primarily the equipment of vocational laboratories with modern technological solutions used in the given professions and the continuous development and expansion of resources, enabling the acquisition of skills that meet the expectations of the changing labour market. The systematic expansion of the school's didactic base creates better and better learning conditions for students, gives them a ready and complete set of knowledge and skills and ensures that they are ready for work without the need for additional courses and training. Some of the interviewees pointed out that the lack of appropriate equipment in the vocational laboratories with the equipment and devices necessary for learning a particular profession limits the possibility of practical vocational training only to apprenticeships carried out at employers.

Among the discussed determinants of the quality of education, Grudowski and Lewandowski (2012) point to, among others, the equipment of the institutions implementing the education process, as well as the nature of the curriculum content or the system of motivation of students and employees.

School directors also pointed to the teaching staff consisting of teachers practising in the given professions, the so-called practitioners, as well as systematic in-service training and self-education of teachers, as important factors determining the quality of vocational education. It turns out that some teachers of vocational subjects employed in post-secondary schools do not have any professional experience in a given field of study, and they obtained the qualifications to teach vocational subjects in a given field of study as a result of completing post-graduate studies. The rapidly changing labour market, scientific and technological progress, automation of the production of goods as well as all transformations and changes in the labour markets force vocational teachers to a continuous update of their knowledge, skills improvement and self-education. In-service training and self-education should therefore be an indispensable part of their professional work, which was considered by the interviewees to be very important in achieving high quality vocational education.

An important factor influencing the quality of vocational education in post-secondary schools is also the use of modern and innovative teaching methods during classes, including activating methods and using information and communication technology – this opinion was expressed by three out of five interviewees. Nowadays, in the era of rapid technological development, 3D printers, space exploration, the use of artificial intelligence, streaming services, modern multifunctional smartphones, the teacher's work has become a real challenge. "How to make learning interesting and what methods to use in order to get the students interested in a given subject and motivate them to expand their knowledge", these are the questions teachers ask themselves, one headmaster points out. Another problem arises, as teachers, so-called professionals, often work simultaneously at school and at another workplace, where they practice their learned profession. On the one hand, this is a huge advantage, as they can give the students practical tips for their profession and inspire them to work in a particular industry. On the other hand, according to school principals, it happens that teachers treat their work at school as an additional place of employment and, due to their busy workload, they no longer find time to systematically update and modify their teaching methods. The quality of education, on the other hand, is inseparably linked to the continuous improvement of the procedures and activities performed by the teacher and the adaptation of working methods and forms and teaching materials to changing realities. The authors also draw attention to the need to train and improve teachers' scientific research skills. Nowadays, these skills are becoming increasingly necessary. Teachers repeatedly carry out didactic measurements, conduct pedagogical experiments pedagogical experiments, and they must also demonstrate their ability to identify educational problems using a variety of methods (Szempruch, 2022). Three out of five school directors also drew attention to the low level of interest of teachers in participating in trainings and courses to improve their work-

ing procedures, for example in didactics or methodology, even if these trainings are financed or subsidised from the school budget.

The directors of public schools highlighted an important factor determining the quality of vocational education, which is cooperation with employers, as part of which practical vocational training, i.e. apprenticeships and practical classes, is carried out. "Crucial for the quality of vocational education is apprenticeship at employers, in these places students get to know the realities of work in the chosen profession", "Apprenticeships carried out at employers show students what work in a given profession is like and allow them to gain their first experience, this is a very important element affecting the quality of vocational education", "The quality of vocational education and the level of professional skills of students is also influenced by practical vocational training". These were the opinions expressed by school directors on the cooperation of schools with employers in the implementation of practical vocational training. Improving the quality of education in vocational schools is not possible without the involvement of employers in the region. The key to this is dialogue with industry and entrepreneurs, as well as the willingness of companies to cooperate with schools (Lis & Miazga, 2016).

Three directors of public schools, when asked whether there was an internal system of education quality management in their institution, replied that the basis for ensuring the quality of education was the pedagogical supervision plan taking into account the conclusions of the supervision, the directions of the educational policy of the state and the issues considered important for the activities of the school.

All managers of the institutions interviewed also mentioned other non-economic factors which, in their opinion, have an impact on the quality of vocational education in post-secondary schools, including: the teaching climate - favourable learning conditions for students and teachers, the level of professional aspirations of students, the involvement of teachers in their work and the monitoring and supervision of the teaching process. However, each of these non-economic factors was indicated by a different interviewees, while the others did not consider them to be important.

None of the institutions have implemented a Quality Management System according to the requirements of the ISO 9001:2015 standard, and in 3 of the 5 schools, surveys of student satisfaction and expectations are systematically conducted to improve the quality of education.

As a result of a study conducted in one of the oldest vocational schools in the Podlaskie Voivodeship, several key factors influencing the quality of education were identified, i.e.: the use of appropriately selected teaching methods, the teacher's involvement in the educational process, the students' tal-

ents, the maintenance of discipline during classes and practical vocational training – classes conducted in a practical form (Hryniewicki, 2019). However, it is worth bearing in mind that the factors determining the quality of vocational education in post-secondary schools are conditioned by many variables, including: socio-economic phenomena, policies, as well as the actions and expectations of stakeholders (Śli et al., 2009).

Conclusions

There are many factors determining the quality of vocational education in post-secondary schools. A number of factors can be distinguished, both internal – related to the organisation of the school's work, and external – present in its environment. They may have a positive impact, improving the quality of education or a negative impact, decreasing it. All of these determinants should be taken into account when aiming to improve the quality of education and to ensure a high level of education. There is no doubt that an indispensable element of the educational system is the continuous pursuit of educational quality and, consequently, the systematic monitoring and improvement of the learning process. This has a huge impact on the quality of the educational offer and the level of preparation of graduates to provide services to employers. The authors presented the most important economic and non-economic factors determining the quality of vocational education from the post-secondary school managers point of view. The interviewees paid particular attention to: the level of teachers' salaries, the school's teaching resources, the teaching staff consisting of teachers practising in the professions concerned, the systematic in-service training and self-education of teachers, the use of modern educational methods, including activating methods, cooperation with employers and practical vocational training. The results of the research identified a new problem that school managers pointed out during the interviews. Teachers, the so-called professionals with the most experience in a given profession, work every day in their learned professions and are not interested in improving their teaching and methodological skills due to lack of time. They often treat school work as a side job and, despite having a lot of practical knowledge, they often find it difficult to pass it on to their students effectively. The survey carried out was preliminary. In the future, it would be advisable to expand the scope of the study and target interviews to more post-secondary school managers. The quality of education requires constant control and verification and adaptation of the school's organisation of work to the changing environment, economic changes, the needs of the labour market, as well as the expectations of employers. There was also the additional aspect of education in times of a (post)pandemic. It is therefore necessary to maintain a continuous dialogue, especially with post-secondary school managers, to examine the changes taking place and to identify the challenges, perspectives and needs of vocational education resulting from the changing reality.

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THE TWILIGHT OF COMMODITY SCIENCE. PART I. THE HISTORY OF THE COMMODITY SCIENCE

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DOI: 10.56091/CTQS.Qual-30

Dedicated to the memory of Professors Gerhard Vogel, Mitsuharu Mitsui, Josef Hölzl and Wolfgang Haupt*

**Josef Hölzl (1925–2022) – Professor at the Institute of Technology and Commodity Science, WU Wien (1972–1991)*

Gerhard Vogel (1944–2022) – Professor at the Institute of Technology and Commodity Science, WU Wien (1994–2012)

Mitsuharu Mitsui (1947–2022) – Professor at the Department of Business Administration at Kobe University (1976–2004)

Wolfgang Haupt (1954–2023) – Director of the commercial academy and commercial school as well as the advanced course "eco Telfs" (Tyrol, Austria) 2005–2019

Abstract

Commodity science is currently in a twilight period in many countries. On the one hand, many specialists have retired and many famous people have passed away. On the other hand, commodity science is being replaced by quality science and marketing.

This article reviews the history of the science of commodities.

Keywords: commodity science, commodity science history

Introduction

Originally, knowledge about tradable goods was at the heart of commercial and thus economic knowledge. Commodity science was therefore able to establish itself as a discipline within economics. This changed, however, after marketing and advertising, and later management disciplines, began to compete with commodity science. By placing their faith in unlimited growth and the inexhaustible carrying capacity of the planet, the economic sciences increasingly distanced themselves from their material basis. The result is well known. It is likely that a sound knowledge of commodities would not have led economists to such insights.

The basis of the research

In many countries, the study of commodities is currently under pressure of economic mainstream. At the same time, while the number and variety of commodities on the markets increases, the econ-

omists seem to have little interest in commodity science. Mainstream economics excludes also any conceptual link between commodities and money. Paul Samuelson has no index entry for "commodity" in his *Economics* (ninth edition, 1973). This is a paradox that seems to be due to economists' blind eye to the real world. We study the timeline of the development of commodity science, mainly by characterising changes in the literature on trade and commodities (Waginger, 2008). In doing so, we try to find out whether the neglect of commodity science in economics is justified (Waginger, 2010).

Evaluation of commodity science history

Early commodity Knowledge

The history of commodity science is closely linked to the requirements of trade, storage and transport. Early documents can be traced back to Iraq, such as the Code of Hammurabi (1793–1750 BC), which defines the requirements for good quality of products (textiles, agricultural products, works of art) and the penalties for non-compliance) to Egypt and India (Kautaliya Arthaśāstra wrote a book of commercial knowledge around 300 BC) (Brockhoff, 2017, pp. 99–106).

The commercial knowledge from the Orient came to Europe via Spain, invaded by the Moors and via Italy. Thus, the Italian merchants had knowledge of an Arabic trade treatise printed in Cairo in 1318 (Brockhoff, 2017). The author was Ali ad-Dimišqī (ad-Dimišqī [The Damascene]). The title of the work was: "Kitāb al-išāra ilā maḥāsin ar-tiġāra wa macrifat ġajjid al-acrāḍ wa radī'ha wa ġušūš al-mudallisīn fihā" meaning "The book of reference to the beauties of commerce". The scope of the work ranges from fragrances, to drugs, dyes, spices, paper, textile fibers, and foodstuffs. In his dissertation ("Ein arabisches Handbuch der Handelswissenschaft", Bonn 1917) (Ritter, 1917), the German orientalist Ritter Helmut (1882-1971) (Maas, 2018) gives a partial translation with a detailed introduction on the Greek and Arabic sources.

When the Florentine chancellor, Gian-Francesco Pagnini della Ventura of Volterra, undertook to write a book summarising the historical development of Florentine finance in the eighteenth century, he found the manuscript of Pegolotti's book in a Florentine library. Pagnini himself wrote two books and added Pegolotti's books and a fourth written by Giovanni da Uzzano in the fifteenth century; he printed the series of four books entitiled "La Pratica della Mercatura". Thus Pegolotti's work is only known through Pagnini's transcription since 1766 (Evans, 1936). Francesco Balducci Pegolotti (1315-40) himself, author of the original 'Pratica della mercatura' ('The Practice of Merchandising'), had been a commercial agent for the powerful Bardi family in Florence (Britannica.). He described the main trade routes and markets around the Mediterranean and Black Sea and in North-Western Europe, of which he had visited great parts. He described the most important commodities traded at

this time, such as spices (pepper, ginger, cinnamon....sugar from Damascus, resins, dye plants, dates, leather, fish, metals (copper from Poland for keys, buckets, vessels, silk) (Evans, 1936).

Early roots of commodity science can also be seen in the herbals and pharmacopoeias (ARANO, 1976) written by medieval monks in monasteries. They included advice on the health effects of everyday goods (e.g. on clothing materials). Modern codices, e.g. for food, could be interpreted as successors to these early commodity books (Waginger, 2008).

Based on a historical review of the commodity science literature, the authors distinguish several phases in the development of commodity science (Figure 1). This review shows how commodity science has always co-evolved with industrialisation until present. We are facing a new, fourth and even fifth wave of industrialisation that will completely change the production and consumption of commodities and that has already found its way into the new commodity science literature (Salerno-Kochan & Wojnarowska, 2023). A next generation will do a better job of classifying new developments. The distinction into periods is only a rough structure, there are many parallel developments and overlaps.

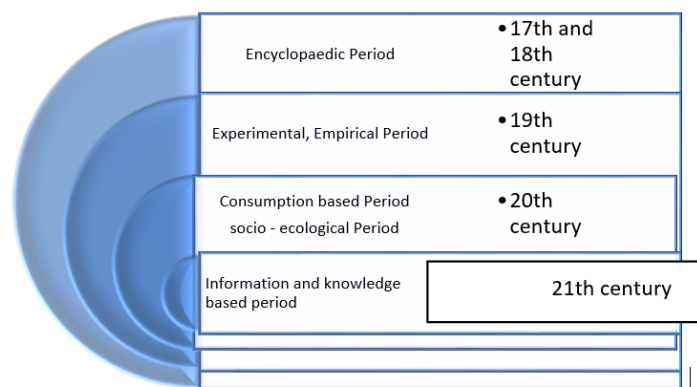


Fig. 1. Periods in the development of commodity science

Source: Waginger, 2008.

The Encyclopaedic Phase: the description of commodities

On the turn from the 17th to the 18th century industrialisation brought a transformation which was reflected by commercial literature. The work (“Le parfait Négociant ou instruction générale pour ce qui regarde de le commerce des marchandises de France, & des pays étrangers” Paris, 1675) of the Frenchman Jacques Savary (1622-1690) initiated this periode. For the first time in European business literature, he systematically structured his vast knowledge of commerce (Leitherer, 1936).

In Germany, Paul Jacob Marperger (1656–1730) called for the teaching of a subject on commodities to be introduced in all higher commercial schools. In 1756, Günther Ludowici (professor in Leipzig)

published the “Outline of a Complete Commercial System for the Instruction on Goods for the Basic Training of the Merchant” (“Grundriss eines vollständigen Kaufmannssystems zur Unterweisung über die Waren zur Grundausbildung des Kaufmanns”). The Göttingen professor and polymath Johann Beckmann (1739–1811) implemented Marperger's demands. He is also considered the founder of technology and commodity science (1772) (Grundke, 1954). He used the term "technology" instead of art history and defined it as the knowledge of the processing of materials and the knowledge of goods. He did not clearly distinguish between commodity knowledge and technology but wanted to melt the two disciplines. Subsequent authors as well described in detail the production processes for the commodities available at their time. Johann Carl Leuchs changed this practice: he wrote a General Encyclopaedia of Commodities "Allgemeines Warenlexikon" (Nuremberg 1825), in which he described the production of goods and their characteristics (labelling, components, properties, storage, etc.) as well as prices and trade routes (Günther & Beckmann, 1999).

Empirical Phase: experiments and the documentation and collection of commodities

With the development of industrialisation, especially the improvement of transport and communication a second globalisation of commerce took place (the first was after the discovery of America, when a number of new commodities came to Europe). At the same time, the end of the 18th century saw ground-breaking scientific discoveries and technical inventions that opened up unimagined possibilities for the production of new kinds of commodities (Nonn, 2022).

During the nineteenth and early twentieth centuries, commodity science was very prosperous – profiting from the progress of the natural sciences and commodity scientists applied natural scientific methods. Knowledge of raw materials was largely derived from experiments and material testing. Microscopes opened up a new world. Technology separated from economics and became the discipline of engineering. Scientific specialisation took place in specific fields such as raw materials and technology. Technology and raw materials science became integral parts of commercial school curricula. Knowledge exploded and books ceased to be encyclopaedic (Hölzl, 1982). This was still a world in which commodities belonged to the realms of minerals, plants and animals, without the hundreds of thousands of synthetic and xenobiotic substances.

Scientific collections (herbaria, drugs commodity collections) were set up. For example, the Austrian Oriental Museum, founded in 1874 (renamed "Handelsmuseum" in 1922), housed a commodity collection. In 1898 the objects were transferred to the Export Academy and in 1922 to the Institute of Technology of the Vienna University of Economics. Today, the Vienna Museum of Science and Technology ([tps://www.technischesmuseum.at/museum/online-sammlung](https://www.technischesmuseum.at/museum/online-sammlung)) houses around 23000 objects from this very large collection (Gruber, 2013).

Attempts were made to establish classification systems of commodities (e.g. influenced by Carl Linné 1707–1778; he had created a binomial nomenclature for plants and animals). In Austria, Professor Hölzl, who had studied natural sciences, revived the attempt of systematic classification under a resource perspective together with his colleague Engelbert Bancher from the Technical University of Vienna. Both published a book which classified natural organic resources according to their morphology and chemistry (Hölzl Josef, 1965).

Professor Grundke (Leipzig), whose books were particularly influential in the Eastern European countries, saw the inspection and classification of commodities as well as the care of goods including storage as the core areas of commodity science (Grundke, 1954).

From today's point of view, a systematic classification of goods in the old sense no longer seems necessary, since any characteristics and criteria can be assigned in databases by means of codes (e.g. EAN code) and allow a systematic order for any requirement. This opens up a new field for economists, where knowledge of commodity science would be useful (Waginger, 2022). In this way, an old dream of commodity researchers has been overcome by digitalisation, which helps to study commodities in many systematic ways. But before this was realised, another wave of commodity science emerged, which can be divided into an earlier consumer-oriented phase and a parallel developing social-environmental phase.

Consumer-Oriented Phase / Social-Environmental phase

In the course of the twentieth century, consumer satisfaction became important, raising issues of quality, standardisation, customer protection and, later, environmental pollution. Commodity science moved away from the natural sciences – commodities were interpreted as central economic phenomena and a manifestation of technology (Grünsteidl, 1970).

Between 1949 and 1966, the consumption of energy and resources and the demand for consumer goods increased exponentially (the "1950 syndrome", first described by the Swiss historian Christian Pfister) (Pfister, 1995).

Rapid industrial development took place, especially in the chemical and electronics industries, households have been mechanized. This development was inspired by military inventions (dual-use goods). Large-scale product tests carried out by consumer organisations were intended to inform consumers about quality and brought public attention and recognition to product science (Stiftung Warentest). Quality standards of private testing organisations such as the wool seal, but also the first eco-labels (Blue Angel, Nordic Swan) became generally known and enjoyed a high degree of credibility. This trust in labels has been increasingly undermined until today. One reason was the penetration

of marketing messages that often replaced facts with feelings. Influenced by the Anglo-American view of compliance, policy gradually shifted from command and control to voluntary approaches. The public interest in the control of products by state institutions faded out. Quality management systems, derived from the military, gradually penetrated all areas of civilian economic life and, as mentioned before, commodity science. This has led to the development of numerous standards and certification schemes, some of varying commitment and trustworthiness. In general, however, the quality of goods and services for consumers has improved significantly in terms of materials, usability, safety, and environmental and social issues. Over time, the improvements have been undermined by greenwashing and also by planned obsolescence. In countries under the USSR regime, such as Poland, the Baltic States, Romania, Bulgaria, Slovakia and Slovenia, this liberalisation of market control did not take place and product testing remained a task of state institutions and universities. As a result, some commodity science institutes at universities still have laboratories. It was even common practice for Western consumer testing institutions to outsource commodity testing to Eastern European institutions, where commodity science was less in conflict with economics and tests were cheap (Waginger, 2008).

Despite the political gap in Europe this period was a heyday of commodity science, which culminated in the foundation of the IGWT (International Society of Commodity Science and Technology). It was constituted in Salzburg, Austria in 1976 by the initiative of prof. Josef Hölzl and dr. Helge Gasthuber (both WU Wien, AT), dr. Lox (Gent, BE), prof. Paul Fink (ETH St. Gallen, CH) dr. Heinrich Schlick (Mannheim, GER), Otto Gekeler (Ulm, GER), prof. Otto Aalhaus (Heidelberg, GER) and prof. Traebert (Dermagen, NL) (Circolare, 1972). Very soon Poland, Hungary, Italy and Japan joined. Today, IGWT is showing signs of decay, having lost almost all its Western European members due to the academic liquidation in these countries, while Italian institutions are seeking cooperation with other disciplines.

Already in the early 1970s, consumerism faded into a socio-ecological wave. In retrospect, the events of this period were historically formative. The 1970s saw the first and second energy crises and the warnings of the Club of Rome. Scientists of sociology made out the "1970s syndrome": in the rich Western countries, social scientists observed a decoupling between domestic material consumption (what meant consumption goods and services) and GDP growth (Fischer-Kowalski, 2016).

The first signs of a paradigm shift towards sustainability changed some disciplines, including commodity science. The economic sciences, on the other hand, tried to avoid this shift until today and to open up new growth paths by "green growth". In some sciences, however, the historical interpretations changed fundamentally: The Industrial Revolution had shifted society's energy supply away

from renewable sources towards the exploitation of large but finite fossil fuel reserves. The resulting economic transformation over the last 200 years is usually interpreted as a story of continuous progress, growth and increasing general welfare. This mainstream narrative of economic history systematically ignores the associated social and environmental costs. New research on industrialisation as an ecological process has been developed by the disciplines of ecological economics (established in the late 1980s) and environmental history (for example calculating the “metabolism” of societies, calculation of footprints) (Stefania Barca, 2011).

Energy experts, engineers, and also commodity scientists contributed to the development of this transdisciplinary research. Energy and life cycle analyses as well as eco-design initiatives have been developed and standardised. In Austria, under prof. Hölzl, the researchers of his institute at WU Wien started with these studies in the 1970s. Inspired by Roegen's famous economic interpretation of entropy (Georgescu-Roegen, 1971), Hölzl's assistant and later successor professor Vogel focused on product entropy and increase in waste. He established, at least in Austria, separate waste collection and management: His habilitation thesis was a contribution of resource economics to minimising the entropy increase of irreversible economic processes in the open system Earth (Vogel, 1982).

In short, sustainability was and still is a major topic in commodity science, but for many decades our commitment to ecological issues was regarded suspiciously by a number of economic sub-disciplines. It was only in the first decade after 2000 that sustainability (after being transformed into the mainstream 'green' growth paradigm) became an important, even eponymous issue in a number of economic institutions. Yet many economists still seem to lack an understanding of the technical, social and environmental limits to growth. In the 1990s, the hypothesis of "decoupling through maturity", known as the Environmental Kuznets Curve, inspired a number of studies and supported the view that efficiency improvements alone could lead to green growth. However, this assumption has not been proven. This is the conclusion of a study on decoupling commissioned by the European Environment Bureau. Structural changes leading to decoupling are extremely unlikely to occur in the near future due to (1) increasing energy expenditures, (2) rebound effects, (3) problem shifting, (4) underestimated impact of services, (5) limited recycling potential, (6) insufficient and inappropriate technological change, and (7) cost shifting (Parrique et al., 2019). Commodity scientists have contributed to the research on energy, rebound effects, recycling, technology, ecodesign and in recent years, circular economy (Salerno-Kochan & Wojnarowska, 2023). Good commodity science training could thus help to identify misjudgements of this kind.

National commodity science development examples

In the early 19th century, business schools were established to train merchants, civil servants and artisans. The teaching of commodities and technology was an important part of the curriculum. These schools soon needed teachers with academic qualification. Before and shortly after the First World War, academic economic institutions with a strong commodity science curriculum were established in many countries: Bulgaria, Poland, Romania, Russia, Slovakia, Ukraine, China, Korea and Japan. Some of them already disappeared in the 1980s (Hungary, Germany, others survived until 2000 or beyond). In general, frequent name changes, e.g. in connection with (quality) management or marketing, were often a sign of the crisis of these institutions. In Austria, the institute changed its name three times after the Second World War (Warenlehre, Warenwissenschaft, Sustainable Product Management). A brief overview of the first modern commodity science academic places is given in Table1.

Tab. 1. The science of commodities at European Economic universities

The first kind of commodity science department was reported from the University of Padua (Italy) in 1549
In Austria, Vienna, an Imperial Export Academy was founded in 1898 and followed by a High School of Economics in 1919 (today’s Vienna University of Economics).
Poland: In 1924 the Institute of Commodities was established at the Higher Trade School in Krakow, which became Higher College of Trade (HCT) in 1925. Commodity science was the basis of the Higher School of Commerce, founded in 1926 by Poznań merchants. After World War II, commodity science was developed in many centers, such as Warsaw School of Economics, University of Technology and Humanities in Radom, Maritime University of Gdynia, University of Warmia and Mazury in Olsztyn and in a number of other agricultural schools and universities of technology.
Two examples from Romania: Bucharest Academy of Economic Studies, which was founded in 1913. In 1920 the Commercial Academy in Cluj-Napoca was founded, becoming Babes Bolyai University of our days. Both with curricula for commodity science.
Among Bulgarian universities the University of National and World Economy (UNWE) established in Sofia in 1920 and the Higher School of Commerce in Varna, which opened in 1920 have commodity science and technologies from their very beginning.
In 1907 the “Moscow Commercial Institute”, now “Plekhanov Russian University of Economics” was founded. During all this time subjects that are closely connected to commodity science have been taught here.
In Lithuania the first Institute to deal with commodities was Klaipėda Commerce Institute founded in 1934 and in 1940 a commodity science department was established at Vilnius University

Source: Waginger, 2008.

Conclusions

An analysis of the traditional scientific and practical works of commodity science shows that it has always been at the forefront or even ahead of the times. For example, the issues raised in the 1990s have only arrived today in mainstream economy and also policy (rather too late): Ecodesign, Circular Economy, Ecological Footprint, use of renewable rather than fossil energy, alternatives to meat consumption, non-toxic products, etc. The representatives of commodity science were mostly cosmopol-

itan and in many cases universally educated. Commodity science is an interdisciplinary science comprising technology, economy, social science and ecology.

The analysis addresses as well several reasons for the rejection of commodity science by mainstream economy. One reason may be that the growth paradigm and the concept of value added are in ideological conflict with commodity science: As the added value of a commodity increases, the entropy in the environment increases. This is a kind of degradation of the environment. For example, recycling cannot be carried out many times to obtain a good raw material again, it always requires energy input and there are creeping losses in quality. However, commodities have not only an environmental but also a social life cycle. A circular economy has to consider this. The economic (monetary and marketing) life cycles of products are quite different, and they usually try to optimise monetary conditions. This has created many of the problems we face today: supply chain problems, resource problems, environmental problems etc.

Finally, we would like to quote the statement of the late Mitsuharu Mitsui “I know that commodity science is now disappearing in Germany and Italy as well as Japan. I don’t think that consumer economics in the United States is popular. As long as we take importance on B/C (benefits/costs) of education at high schools and universities, marketing seems to be a more reasonable (efficient) subject than commodity science or consumer economics in the present industrial capitalist societies. This phenomenon will last for the next several decades. The only hope to maintain the academic and practical subject of commodity science may be found in the local communities’ environment management. Although I would like to signify fertile commoditization in the local economy in further ways, my time to develop and hone the related academic concepts is running out. Life is so short for a Japanese lonely commodity scientist”.

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THE TWILIGHT OF COMMODITY SCIENCE. PART II. CONTEMPORARY SITUATION OF COMMODITY SCIENCE IN SELECTED COUNTRIES

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DOI: 10.56091/CTQS.Qual-31

Dedicated to the memory of Professors Gerhard Vogel, Mitsuharu Mitsui, Josef Hölzl and Wolfgang Haupt*

**Josef Hölzl (1925–2022) – Professor at the Institute of Technology and Commodity Science, WU Wien (1972–1991)*

Gerhard Vogel (1944–2022) – Professor at the Institute of Technology and Commodity Science, WU Wien (1994–2012)

Mitsuharu Mitsui (1947–2022) – Professor at the Department of Business Administration at Kobe University (1976–2004)

Wolfgang Haupt (1954–2023) – Director of the commercial academy and commercial school as well as the advanced course "eco Telfs" (Tyrol, Austria) 2005–2019

Abstract

Commodity science is currently in a twilight period in many countries as is being replaced by quality science and marketing. We will consider whether knowledge of the properties of commodities and their embeddedness in environmental and social systems is necessary for a marketing specialist and any other economist.

Keywords: commodity science, commodity science history

Introduction

The first part of the article reviews the history of commodity science, identifying critical turning points and examining developments in various countries. Based on the interpretation of communications and reports from IGWT members, it appears that commodity science has entered a kind of twilight sleep, with three main trends:

- Commodity science is being completely abolished: Switzerland, Belgium, Germany, Austria, and Slovak Republic.
- Commodity science is merging with environmental and social sciences: Austria before the shut down of the institute, Italian institutions, partly Poland/Poznan.
- Commodity science is being assimilated: e.g. by (quality) management and marketing: Korea, Japan, partly Poland/Krakow, Romania/Bucarest, Cluj Napoca, Lithuania.

The relationship to quality management should be viewed differently, as a large part of commodity science is dedicated to quality management (Salerno-Kochan & Wojnarowska, 2023). Ruževičius even

understood commodity science as a precursor science of quality management sciences and saw little conflict in this close relationship (Ruževičius, 2010).

A brief overview of the academic institutions in modern commodity science is provided in Table 1 in Part I. The contemporary situation of commodity science in selected countries will be characterized based on personal communication (Giungato, 2023; Karkalíková, 2023; Karavayev, 2023), papers, correspondence and minutes from Institutes, manuscripts (Mitsui, 2010) and articles (Salerno-Kochan et al., 2020)

National commodity science situations examples

As it would be far too exhaustive to list all the stories, the following are a few selected examples of their fate (Waginger, 2008).

Austria

In Austria, for example, commodity science can be traced back to the establishment of polytechnic and commercial schools and the Vienna World Exhibition of 1873, which led to the founding of the Imperial Export Academy of Vienna in 1898. In 1919, the Academy became the University of World Trade (now WU Wien) (Hözl, 1982).

As in other European countries after the First World War, the beginning of commodity science as an economic discipline in Austria was rather hopeful. The importance of commodity science is reflected in the fact that it occupied the entire third floor of the university building in 1919, with laboratories and showrooms for the raw materials collection. During the Second World War, research in commodity science and technology was driven by resource scarcity and the search for alternative materials and technologies (e.g. biofuels). Books from this period are written in a rather nationalistic style. Also some professors in Austria, had been politically active before and during the Second World War. Maybe this was a reason for a doubtful image of the discipline for some time. Although almost all the professors at the Austrian commodity science Institute at WU Vienna had to defend the discipline within the university, they managed to survive. Prof. Hözl (Head of the Institute 1972–1991) succeeded in establishing a separate curriculum for the training of teachers of commodity science at grammar schools, and his successor prof. Vogel (Head of the Institute 1994–2012) introduced a specialisation in waste management into the commodity science curriculum. In a new curriculum after 2003/4, commodity science even became a very successful specialisation (field of competence) within microeconomics. All these achievements were abolished after the implementation of the Bologna reform in 2006. The Senate found that commodity science no longer fit into an economic studies programme, and the institute went into twilight. When Professor Vogel retired in 2012, the Institute

was closed. Two of the remaining academic staff were taken on by the Institute of Multilevel Governance and Development until their retirement. The last commodity science lecture was held in 2018 (Convolut & P).

Bulgaria

The situation of commodity science in Bulgaria (Varna) is at first sight reassuring. Pashova (2018) presented history of commodity science in Bulgaria. The emergence and development of commodity science, and in particular a brief history of the specialty commodity science at the University of Economics-Varna, Bulgaria as well as the state of the commodity science in modern market conditions is described by Stoykova and coauthors (Stoykov et al., 2020). The actuality of the training of specialists with high qualification in the field of commodity science in the Department of Commodity Science is presented and the possibilities for their wide realization are outlined. The factors that determine the relevance and importance of the commodity science, and the perspective directions for its development are outlined.

Germany

In Germany in the 19th century, Johann Beckmann's ambition to train economists and farmers in technology and raw materials science was implemented. First, polytechnic schools were founded (in Karlsruhe in 1825, then in Munich and Stuttgart), most of which became higher technical colleges or universities in the 20th century. The first commercial school was founded in Braunschweig in 1896, followed by a commercial school in Leipzig in 1898, and schools in Frankfurt, Cologne, Mannheim, etc. Before the First World War, technology was one of the compulsory subjects in business studies, e.g. chemistry and physics with special emphasis on technology at the Leipzig commercial college.

After the Second World War, language, business and technology programmes were established in higher commercial colleges. The emphasis on technology remained until the 1950s, but by the 1970s it was very often only an optional subject in most commercial teacher training programmes. The reasons for the decline of the subject were that the increasing variety and development of technology and commodities made the purchase of demonstration equipment and collection items too expensive. In addition, students' skills and interest in science were decreasing, and the state was neglecting teacher training in this area (Vatter, 1970).

In academic economics, commodity science has increasingly been dissolved into business subjects such as procurement, sales and marketing. Technology was integrated into subjects such as industrial management or product development. This happened, for example, at the University of Cologne,

Faculty of Management, Economics and Social Sciences, under the leadership of Professor Udo Koppelman (1939–2023) (Koppelman, 1970).

Commodity science developed from a scientific approach to a product-related approach and then to an approach based on specific customer behaviour and its implications for marketing (Troll, 2005).

In Germany, as in Austria, there is no longer a Chair of Technology and Commodity Studies at the university level.

Italy

The situation of Merceologia in Italia seems to be similar to that in Poland, but the changes caused by the change of the FOS/OECD classification took place earlier. Most of the commodity scientists come from economic faculties as the discipline for historical reasons belong to the old economic school of commerce (subsequently converted into economic faculties), but some of them come from chemical or other faculties. You can obtain the degree of Professor of the 1st or 2nd degree in the field as still there is the Scientific Habilitation Committee - Abilitazione Scientifica Nazionale (ASN) in commodity science (Scienze merceologiche 13/B5 or the SECS P/13 scientific sector). In the declaration of the SECS P/13 sector, is stated that the goal of commodity science is to gather the research fields that study the production of goods, analysing and assessing both resources and production/transformation technologies together with the impacts on quality and environment, including environmental managements and certification systems. Actually there are 103 professor and assistant professor belonging to the commodity science scientific sector distributed in about 30 universities in the country. The “Società Italiana di Merceologia” (SIM) currently AISME (Accademia Italiana di Scienze Merceologiche) has been active since 1978. Last year's convention, held in Bari (27–28 October) at the Aldo Moro University, was the XXX Congresso Nazionale di Scienze Merceologiche (<https://www.aisme2022.it/>). It gathered 140 participants who presented 21 plenary lectures, and 119 scientific communication and posters in the field of: Quality management systems, process and product certification, safety and traceability, sensory analysis, labeling, quality and consumer relations (Giungato, 2023).

However, in recent years, communication with other European commodity institutions has been and remains poor. It is regrettable to say that after the last IGWT symposium in Rome, Italian activists failed to reorganize this association.

Japan

The following is a short extract from a report of Mitsuharu Mitsui, Professor, School of Business, University of Hyogo (Mitsui, 2010)

Mitsuharu Mitsui was Professor of commodity science at the Department of Business Administration at Kobe University of Commerce in Japan for about 30 years, from 1976 to 2004. Around 2000, commodity science was integrated into marketing at the level of Japanese business schools. Only a few university professors maintained commodity science in their curriculum. Mitsui then taught commodification for local businessmen and service management for undergraduates, and commodity science in a master's programme at the University of Hyogo, but switched to service management in 2010. When he retired in 2012, this position was also closed. According to him, commodity science has always been favoured by the weak. Until the 1950s, weak people and organisations in Japan were satisfied and supported by their encyclopedic (well-categorised) knowledge. Once Japan began to enjoy high economic growth in the 1960s, the paradigm of commodity science shifted steadily and marketing has been promoted ever since.

Poland

In Poland, the science of commodities gave rise to higher economic universities in Cracow and Poznań. However, commodity science disappeared from the list of scientific disciplines in Poland in 2018. This was motivated by the fact that commodity science is not included in the list of scientific disciplines of the Organization for Economic Co-operation and Development (OECD). In 2004 OECD adopted the Field of Science And Technology (FOS) system of sciences, covering six fields of science and one field of art (OECD). It was the result of adapting the so-called "The Frascati Manual for a changing reality". The OECD claimed that the existing FOS classification does not fully reflect changes in the science and technology area, especially with regard to emerging technology fields such as ICT, biotechnology and nanotechnology. Commodity science is missing from this list. OECD claims that it was not possible to develop a FOS classification that satisfied the needs of all actors involved. As a result, the final classification represents a compromise between different viewpoints and user needs.

In the FOS list by OECD in point 5. Social sciences there is a subsection 5.2 Economics and Business, which includes:

- Economics, Econometrics;
- Industrial relations;
- Business and Management.

Poland had to adapt the division of scientific disciplines to this classification. Academic circles faced the necessity of pooling disciplines, especially in Polish economic universities. As a result of pooling, the number of scientific disciplines was reduced from over 100 to about 30. At the same time, a broad discussion on this classification took place.

Unfortunately, commodity science as an independent scientific discipline could not be saved.

In a scientific discipline 5. Social sciences, two sub-disciplines were created:

- economics and finance, and
- management and quality sciences.

Management and quality sciences are an artificial combination of management issues with the old scientific discipline of commodity science. As a result of this “provisional”, according to Sułkowski & Lenart-Gansiniec (2021) construction, practically all significant faculties and departments of commodity science were forced to revise not only their names, but also the scope of their scientific and research activities. However, despite a series of discussions with decision-makers in the field of management sciences, like members of Council of Scientific Excellence, the official scope of this new discipline has not yet been established! The difficulty of institutionally defining the boundaries of management and quality sciences is also visible in scientific policy, for example in disputes related to the approval of the title of doctor or habilitated doctor in cases where members of the commission consider belonging to a discipline to be doubtful. Unfortunately, this applies mainly to commodity scientists.

A significant contribution to the discussion on the scope of this discipline is the article by Salerno-Kochan and co-authors (2020). It is a voice in the ongoing discussion in scientific circles related to the discipline of management and quality science on defining sub-disciplines and research areas. The authors set the goal of systematizing quality sciences as a sub-discipline with particular emphasis on the relevant areas of research. Three areas of research in the practical stream of science were proposed, such as:

- systemic quality management,
- quality in the full product life cycle, and
- consumer determinants of product quality.

Two areas of research in the theoretical trend were defined, i.e. the methodology of quality sciences and critical studies of quality sciences. According to the authors of this article, the ontology of this sub-discipline should also be considered.

The aforementioned authors also presented the substantive areas and thematic scopes of research in individual areas of quality science, emphasizing their complementarity and pointing to their separateness from management science. Complementarity mainly concerns the implementation of product management processes that enable the recognition of buyer needs and consumer satisfaction surveys, thus constituting a starting point for improving product quality. In turn, distinctiveness is associated with a multidimensional approach to product quality, using interdisciplinary knowledge in the field of social sciences and natural and technical sciences, thanks to which it is possible to shape and identify product features in accordance with the requirements and expectations of the consumer. However, so far it has been difficult to convince policy-makers from management sciences to this approach.

The discussion on commodity science (Zalewski, 2013), the interdisciplinarity of quality sciences (Borys, 2012) and their place and importance in the classification of scientific fields and disciplines as well as economic practice (Przybyłowski & Grudowski, 2018) has been going on in Poland for many years. Unfortunately, with disastrous results, because commodity science as such is disappearing in practically all universities where they existed and developed so far, moving towards quality sciences.

Nevertheless, the Polish Commodity Science Society and the Commodity Sciences – Quality Sciences Committee at the Polish Academy of Sciences (Poznan Branch) still exists and operates.

Romania

In Romania the first name of Commodity Knowledge appeared for the first time in a book published in 1879 entitled “Commodities trading Manual for knowledge of goods” author N. Mallian – Professor of Accounting and Commercial goods Public School in Craiova. Also in Romania in 1895, Arsenie Vlaicu published in Brasov the first manual of classic Commodities entitled “Commodity and technology professional trade schools and private study” (Burda & Chirimbu, 2016). The situation of commodity science in Romania is also changing. There are still Dep. Com. Sci in several universities, but in most universities this studies have been transformed into other specialties or incorporated into other faculties. As Onete and coauthors (2010) claim, recently commodity science in Romania is turning into consumer science, its study object extending to the product itself and its reality, towards the study of the relations between consumer, community and environment, by taking into consideration the socio-economic and scientific aspects of the product impact on the consumer.

Slovak Republic

At the University of Economics in Bratislava, established in October 1940 the Department of Commodity Science (named Institute of Commodity Science, Raw Material, Mechanical and Chemical

Technology Science) was part of the College of Commerce in Bratislava. In 1945 it was renamed as Institute of Commodity Science and Technology. In 1960, as a result of its division, the Department of Commodity Science began to take shape and was officially established in 1964. In the early 1990s, the department's name changed to the Department of Commercial Commodity Science.

The educational objective in the field of commodity science was to provide fundamental information and knowledge about the utility characteristics of food and non-food products. The department focused on product quality, safety, innovation, and environmental aspects of products, product packaging and storage, as well as technical barriers to trade. It also addressed the implementation and improvement of quality management systems, environmental management, and integrated management systems to enhance the competitiveness of businesses. In addition to the theoretical part, teaching also included laboratory exercises, utilizing available laboratory equipment such as microscopes, refractometers, pH meters, and more. The department conducted educational activities at all three levels of higher education, and it was the first among the departments of the Faculty of Commerce that offers e-learning courses. The results of its scientific research were realized through domestic and international projects such as Tempus, Leonardo da Vinci, Erasmus+, Horizon+ and others. The department had a strong collaboration with international institutions focused on commodity science. In the academic year 1997/1998, the department's name was changed to the Department of Commodity Science and Product Quality, which ceased to exist in 2021 as a result of reorganization within the Faculty of Commerce (Karkalíková, 2023).

Ukraine

The training of commodity science experts in Ukraine has deep roots and a unique, almost hundred-year-old experience: from a few schools at the beginning of the 20th century to today, when more than 100 educational institutions of various levels of accreditation, subordination and ownership are successfully training commodity science experts (Mazaraki, 2008).

As a result of the transformational processes in the education and economy of Ukraine, the reform of the system of state market supervision, consumer rights protection, the improvement of the legislative and institutional environment for the implementation of the Association Agreement between Ukraine and the EU, the need to develop new sales markets for Ukrainian goods, the field of commodity science competence is also expanding significantly, the tasks and functions of commodity science scientists are changing, and the social significance of scientific products of commodity science is increasing.

From the traditional assessment of the quality of goods, the specialists of this area in particular approached the management of the quality and safety of goods on the basis of the introduction of international requirements, detection of falsification, conducting expert evaluation, law-making activities, and in fact – to systematic work on the formation of a high level of quality of life of citizens as consumers (DSTU, 2000; Mazaraki et al., 2014).

The key to the stable development of trade in Ukraine is providing the industry with highly qualified personnel. The leading role in this process belongs to the system of higher education. The State University of Trade and Economics (SUTE) as the legal successor of the Kyiv National University of Trade and Economics (KNUTE) is a leader in the training of specialists in commodity science, and heads the development of relevant standards of higher education in Ukraine.

Today, SUTE is a multi-disciplinary higher education institution of the European level and has been training specialists in the fields of economics, entrepreneurship, trade, quality management and many others for over 75 years both in the base institution (Kyiv) and in 13 separate structural units (3 institutes, 8 colleges, 2 schools) in 9 cities of Ukraine. Commodity science is taught as a separate discipline that forms the professional competencies of future specialists (SUTE, 2023).

SUTE trains specialists in 62 educational programs of the first (bachelor's) level (including 5 taught in English), 65 programs of the second (master's) level (including 14 taught in English), 15 programs of the third (educational and scientific) level (as of September 2023). The educational process is aimed at meeting the needs of the labor market, the formation of competitive advantages and innovative competences in the students of education. For more than ten years, national state education standards have been in effect – educational and professional programs, regulations for diagnosing students' knowledge. A complete set of industry standards of higher education, built on a competency-based approach, has been created.

Among other higher education institutions of Ukraine, which provide for the study of the discipline of commodity science, the following should be noted: Vinnytsia Institute of Trade and Economics of SUTE, Kyiv National University of Construction and Architecture, Lviv University of Trade and Economics, National University of Food Technologies (Kyiv), National Pharmaceutical University (Kharkiv), Odessa National Technological University, Poltava University of Economics and Trade, Uzhhorod Institute of Trade and Economics of SUTE, Alfred Nobel University (Dnipro), Kherson National Technical University, Chernivtsi Institute of Trade and Economics of SUTE and others (Mazaraki & Prytulska, 2016).

Separate departments of commodity science, expertise, product safety and quality management have been established in the mentioned universities and institutes, or in which commodity science is studied as a separate discipline.

The text is based on the presentation of Prof. Taras Karavayev during the 2nd IQMSc conference (Karavayev, 2023).

Conclusion

As already mentioned, in many countries, commodity research is currently under pressure from the mainstream economy, which is leading to a number of irreversible changes.

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