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PHYSICOCHEMICAL PROPERTIES OF INNOVATIVE MULTICOMPONENT DIETARY SUPPLEMENTS

S u m m a r y

Background. In recent years, dietary supplements have become the subject of interest among consumers, food producers and the pharmaceutical industry. Dietary supplements are not medicinal products and are not considered as such. However, the majority of society in many countries treats dietary supplements as equal to medicinal products. The article draws attention to innovative dietary supplements containing many ingredients from exotic plants, which have not been used in supplements to such an extent so far. The aim of this study was to determine the physicochemical properties of multi-ingredient Purella Super Mix dietary supplements. These supplements are produced by Purella Food, and the series includes five products with various advertised effects on the human body. Purella Super Mix products were characterized in terms of their composition and the resulting importance for human health. A number of physicochemical analyzes of Purella Super Mix were carried out, which have not been published to this extent before. Water content, water activity, bulk density and the angle of repose and cone angle were measured, as well as the Hausner ratio and the Carr index were determined. These properties deliver important information both for the technological process and for subsequent stages of the product life cycle.

Results and conclusions. The results of the research on the physicochemical properties of innovative multi-ingredient dietary supplements can be used to predict the durability of these products and to design the packaging that takes into account the behavior of supplements during storage. The results obtained may also constitute the basis for further research on improving the composition and properties of multi-ingredient dietary supplements.

Key words: dietary supplements, physicochemical analysis, water content, water activity, flowability

Introduction

In recent years, there has been a rapid increase in the production of food products classified as dietary supplements. This trend intensified during the COVID-19 pandemic, as sales of products for common cold, flu, and immunity boosters increased significantly at that time [12].

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The term “dietary supplement” is defined slightly differently in various countries. The definition in the Dietary Supplement Health and Education Act (DSHEA), which was adopted in 1994 by the U.S. Food and Drug Administration (FDA), defines a dietary supplement as an orally taken product containing a “dietary ingredient” intended to supplement the diet. This Act defines the terms “dietary ingredient” and “new dietary ingredient” as components of dietary supplements [6].

The main legal act of the European Union (EU) is the Directive 2002/46/EC on the approximation of the laws of the Member States relating to food supplements. It defines “food supplements” as foodstuffs the purpose of which is to supplement the normal diet and which are concentrated sources of nutrients or other substances with a nutritional or physiological effect [16]. Similarly, the European Food Safety Authority (EFSA) defines dietary supplements as concentrated sources of nutrients or other substances with a nutritional or physiological effect intended to supplement the normal diet [23].

In 2005, Codex Alimentarius adopted Guidelines for vitamin and mineral dietary supplements [25]. They only apply to supplements containing vitamins and/or minerals where these products are regulated as food, and address the composition of supplements, including their safety, purity and bioavailability [24].

The above definitions show that dietary supplements are not medicinal products and are not considered as such. However, the majority of people in many countries treat dietary supplements as equal to medicinal products, and knowledge about them is still fragmentary [15, 56].

Distinguishing dietary supplements from other categories of foodstuffs such as conventional foods and medicinal products is not easy. Therefore, two questions demand an answer, what are the boundaries between dietary supplements and conventional foods, and the boundaries between dietary supplements and over-the-counter medicines? New products are constantly appearing on the dietary supplement market, and therefore it is reasonable to ask about their composition, properties and safety of use.

The aim of this study was the determination of physicochemical properties of multi-ingredient Purella Super Mix dietary supplements. These supplements are produced by Purella Food, and the series includes five products with different claimed effects. There is a lack of research in the literature on the physicochemical properties of multi-ingredient dietary supplements, which may be important for the management of their production, transport and storage. The physicochemical properties of supplements may influence their preparation for consumption, which in turn is of great importance to the consumer. Parameters such as water content, water activity, bulk density and the static and kinetic angles of repose constitute important information both for the technological process and for the subsequent stages of the product life cycle. Addi-

tionally, knowledge about the physicochemical properties of dietary supplements can be broadened by further research results, such as the color of supplements and its changes during storage.

Material and methods

The research material consisted of five multi-ingredient Purella Super Mix dietary supplements: Super Mix Detox, Super Mix Protein, Super Mix Antyox, Super Mix Energy and Super Mix Immunity. The declared composition of Purella Mix dietary supplements is presented in Table 1. The products in retail packaging were purchased on www.abc-store.pl.

Table 1. Purella Super Mix dietary supplement composition

Tabela 1. Skład suplementów diety Purella Super Mix

Product / Produkt	Composition / Skład
I – Detox / Detox	moringa powder bio 40 % / sproszkowana moringa bio 40 % chlorella powder 30 % / sproszkowana chlorella 30 % linseed 10 % / siemię lniane 10 % baobab dried fruit pulp bio 10 % / suszony miąższ owoców baobabu bio 10 % spirulina powder 10 % / sproszkowana spirulina 10 % PL-EKO-06
II – Proteins / Proteiny	hemp protein powder bio 30 % / proszkowane białko konopne bio 30 % powdered maca root bio 30 % / sproszkowany korzeń maca bio 30 % dried baobab fruit pulp bio 10 % / suszony miąższ owoców baobabu bio 10 % banana puree powder (banana puree, maltodextrin) 10 % / sproszkowany przecier bananowy (przecier bananowy, maltodekstryna) 10 % pea protein powder bio 10 % / sproszkowane białko grochu bio 10 % powdered raw cacao beans bio 10 % / sproszkowane ziarna surowego kakao bio 10 % PL-EKO-06
III – Antioxidants / Antyoksydanty	blueberry powder 30 % / sproszkowana borówka amerykańska 30 % camu camu berry powder bio 20 % / sproszkowane jagody camu camu bio 20 % chia – chia seeds (<i>Salvia hispanica</i>) bio 20 % / chia – nasiona szalwii hiszpańskiej (<i>Salvia hispanica</i>) bio 20 % lucuma fruit powder bio 15 % / sproszkowane owoce lucuma bio 15 % maqui berry powder bio 10 % / sproszkowane jagody maqui bio 10 % acai berry juice powder bio (acai berry juice bio, tapioca bio) 5 % / sproszkowany sok z jagód acai bio (sok z jagód acai bio, tapioka bio) 5 % PL-EKO-06
IV – Energy / Energia	maca root powder bio 40 % / sproszkowany korzeń maca bio 40 % banana puree powder (banana puree, maltodextrin) 30 % / sproszkowany przecier bananowy (przecier bananowy, maltodekstryna) 30 % acai berry juice powder bio (acai berry juice bio, tapioca bio) 15 % / sproszkowany sok z jagód acai bio (sok z jagód acai bio, tapioka bio) 15 % guarana powder bio 10 % / sproszkowana guarana bio 10 % lucuma fruit powder bio 5 % / sproszkowane owoce lucuma bio 5 % PL-EKO-06

V –Immunity / Odporność	ginger powder bio 35 % / sproszkowany imbir bio 35 % mango powder 20 % / sproszkowane mango 20 % powdered turmeric bio 15 % / sproszkowana kurkuma bio 15 % / lucuma fruit powder bio 15 % / sproszkowane owoce lucuma bio 15 % / camu camu berry powder bio 10 % / sproszkowane jagody camu camu bio 10 % maca root powder bio 5 % / sproszkowany korzeń maca bio 5 % / PL-EKO-06
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Source: own compilation based on information contained on unit packaging

Źródło: zestawienie własne na podstawie informacji umieszczonych na opakowaniu jednostkowym

The water content in the tested samples of non-pulverized dietary supplements was determined by thermal drying in a laboratory dryer at a temperature of 403.15 K (130 °C) under normal pressure until constant mass was obtained [37]. The content of water was expressed in g/100 g d. m. (dry mass).

Water activity was determined in the AquaLab 4TE device (version AS4 2.14.0 2017, Decagon Devices, Inc.) with an accuracy of ± 0.0003 at a temperature of 293.15 K (20 °C) [45].

The color of the tested dietary supplements was determined by measuring the trichromatic components $L^*a^*b^*$ CIE Lab using a Konica Minolta C-410 camera.

The physicochemical properties of Purella Super Mix supplements relevant to storage were assessed by determining the static and kinetic angle of repose [19, 47].

Both of these parameters are strongly related to the fluidization and flowability of powders, and Table 2 presents the interpretation of the static and kinetic angle of repose values in this context.

Table 2. Powder fluidization and flowability indices

Tabela 2. Wskaźniki fluidyzacji i sypkości proszków

Powder fluidization index Wskaźnik fluidyzacji proszków		Powder flowability index Wskaźnik sypkości proszków	
Tendency to fluidization Skłonność do fluidyzacji	Angle of repose [°] Kąt zsypania [°]	Flowability index Stopień sypkości	Cone angle [°] Kąt nasypu [°]
Very high / Bardzo wysoka	≤ 25	Very good / Bardzo dobra	≤ 30
Fairly high / Dosyć wysoka	$26 \div 40$	Quite good / Dosyć dobra	$31 \div 35$
Showing a tendency to flow / Wykazująca tendencję do płynięcia	$41 \div 50$	Good / Dobra	$36 \div 40$
Can flow at times / Czasami może płynąć	$51 \div 57$	Average / Przeciętna	$41 \div 45$
		Poor / Słaba	$46 \div 55$
Does not exhibit flow properties Nie wykazuje właściwości płynięcia	≥ 58	Bad / Zła	$56 \div 65$
		Very bad / Bardzo zła	$66 \div 90$

Explanatory notes / Objasnienia:

Source: compilation based on Jedlińska et al. 2013, Manual 2010

Źródło: zestawienie na podstawie Jedlińska et al. 2013, Instrukcja 2010

The characterization of flowability parameters of the powders was based on the determination of loose and tapped density, which are the basic characteristics necessary to determine the Hausner ratio and Carr index [1, 2, 46, 47]. Both the Hausner ratio and the Carr index have an empirically established relationship to the flowability of powder. The Hausner ratio is a parameter that determines the cohesion of powder.

The results obtained were presented as the average value of nine repetitions. The hypothesis about the differences in the average level of physicochemical parameters in the tested products was verified using the Fisher-Snedecor F test combined with a post-hoc analysis in which the least significant difference (LSD) test was used. These methods were used due to the fact that all compared empirical distributions were close to the normal distribution (which was checked with the chi-square goodness-of-fit test) [40]. All the hypotheses were verified at a significance level of $\alpha = 0.05$, based on the test probability value, p . It was assumed that $p \leq 0.05$ indicates a significant difference in the parameter between the examined objects. The Statistica 13.3 package was used in the analysis.

The storage quality was assessed using the static-desiccator method, determining sorption isotherms and determining the moisture balance between the tested sample and the atmosphere with the assumed relative humidity, which was regulated using saturated salt solutions. The products were placed in a hygostat with water activity in the range of $0.07 \div 0.92$ and stored for 90 days at 20 ± 1 °C. Mathematical descriptions of sorption isotherms were modeled by the BET (Brunauer, Emmett and Teller) isotherm in the range of $a_w = 0.07 \div 0.33$. The capacity of the monomolecular layer (v_m), energetic constant (c_e) and specific sorption surface were determined [53]. The results were analyzed using Jandel-Table Curve 2D v. 5.01 software. The fit of empirical data to the BET equation was characterized by the coefficient of determination (R^2), the sum of squares of deviations of theoretical from empirical values (SS), and the values of standard errors (RMS).

Commodity characteristics of Purella Super Mix dietary supplements

Characteristics of Super Mix Detox

Purella Super Mix Detox dietary supplement is a preparation intended to help cleanse the body. The basic ingredient of this supplement is moringa powder (40 %). The plant belongs to the *Moringaceae* family, which includes several species, the most widely used of which is *Moringa oleifera* Lam. *Moringa oleifera*, also called the “Miracle Tree”, is a tree with enormous nutritional and medicinal properties [30]. It is rich in macro- and microelements and other bioactive compounds that are important for the proper functioning of the body and the prevention of certain diseases [3, 44]. The leaves, flowers, seeds and almost all parts of this tree are edible and have therapeutic

properties, including antidiabetic, anticancer, antiulcer, antimicrobial and antioxidant properties [30].

The second ingredient in Purella Super Mix Detox in terms of content is chlorella at 30 %. *Chlorella vulgaris* Beijer is a representative of algae found in sea and inland waters, rich in numerous nutrients, including vitamins D, B₁₂, folic acid and iron [7].

Purella Super Mix Detox also includes linseed (10 %), dried baobab fruit pulp (10 %) and spirulina (10 %). Linseed contains significant amounts of biologically active ingredients, such as α -linolenic acid, lignans and dietary fiber. Due to its numerous nutritional properties, linseed has been classified as a “superfood” [42].

Baobab (*Adansonia digitata* L.) grows in many regions of sub-Saharan Africa and in Madagascar. Baobab products are important in nutrition and folk medicine. Mainly fruit pulp is used for this purpose, which can be eaten raw or added to dishes [13].

Spirulina (*Arthrospira platensis*) is another cyanobacterial algae that is part of Purella Super Mix Detox with hypolipidemic and hypoglycemic properties, also acting against hypertension [39]. Moreover, spirulina does not have cellulose cell walls, hence it is easily digested [27].

Characteristics of Super Mix Protein

Purella Super Mix Protein dietary supplement is a product with high protein content that is advertised to help maintain muscle mass and healthy bones. Purella Super Mix Protein dietary supplement is a product with a high protein content that may help maintain muscle mass. The main source of protein is 30 % hemp (*Cannabis sativa* L.) protein powder [41].

Another ingredient whose content in the product is 30 % is powdered maca root. *Lepidium meyenii* (maca) is a Peruvian plant from the *Brassicaceae* family that has been cultivated for over 2000 years and grows only in the Central Andes at an altitude of 4000 to 4500 m [28]. Maca has nutritional, energizing and fertility-enhancing properties, and has effects on sexual dysfunction, osteoporosis, benign prostatic hyperplasia, memory and learning, as well as protects the skin from ultraviolet radiation [29]. Clinical studies have shown the effectiveness of maca in sexual disorders and in increasing sperm count and motility [10].

Super Mix Protein also contains 10 % of other ingredients in powdered form, such as banana puree, pea proteins and raw cocoa beans [35]. Raw cocoa beans are characterized by high antioxidant activity, because most flavanols are reduced during cocoa processing, including fermentation, roasting and Dutch processing [26]. However, one must remember that it is a raw food product, which means it may be contaminated with pathogenic bacteria just like any other raw food.

The manufacturer declares that all ingredients of the Super Mix Protein supplement are organic and that the product has an organic food certificate. However, the name of the body certifying this product is missing on the packaging.

Characteristics of Super Mix Antyoks

Super Mix Antyoks dietary supplement is a product containing many ingredients of plant origin, including powdered blueberries, powdered camu camu berries, chia sage seeds (*Salvia hispanica* L.), powdered lucuma fruits, powdered maqui berries, powdered acai berry juice and tapioca.

Previous research shows that blueberries (*Vaccinium corymbosum* L.) are a rich source of bioactive compounds and demonstrate high antioxidant activity in vitro and in cellular systems in ORAC and PSC tests [55]. Camu camu fruits (*Myrciaria dubia* L.) are a significant source of vitamins and minerals. The berries of this plant are rich in vitamin C and β -carotene. Due to the low glycemic index, they are recommended for people suffering from diabetes [38]. *Salvia hispanica* L. is a subtropical plant known since pre-Columbian times. The qualities of the plant have been appreciated and, since 2009, chia seeds have been classified as novel food by an implementing decision of the EU Commission [57]. Lucuma fruit (*Pouteria lucuma* (Ruiz and Pav.) Kuntze) is little known on the European market. The fruit is mainly consumed in Peru and Chile, and known as a good source of fiber, minerals, β -carotene, phenolic compounds and niacin [17]. Maqui berries (*Aristotelia chilensis* (Mol.) Stuntz) are a source of anthocyanins. The relatively high content of anthocyanins and the presence of polar polyglycosylated derivatives make the fruits of *A. chilensis* an interesting source of anthocyanin extracts for food and pharmaceutical applications [22]. Acai berries (or assai) are one of the most popular fruits that are classified as functional foods in the world [14].

The addition of tapioca to this supplement is intended to provide the right consistency to the product, which is important for the consumer. Tapioca is obtained from cassava (*Manihot esculenta* Crantz), and its physicochemical and structural properties such as swelling, solubility, gelation, consistency (stickiness) mean that it is increasingly used by the food industry. Cassava contains significant amounts of fiber and does not contain gluten, which, for understandable reasons, arouses the interest of nutritionists and, above all, consumers suffering from diseases [11, 21].

Most of the ingredients of Super Mix Antioxidants, apart from blueberries, come from organic farming and the product is certified as an organic food product.

Characteristics of Super Mix Energy

Super Mix Energy dietary supplement is a product that is advertised as having a stimulating effect on the body. It includes powdered plant raw materials such as: maca root, banana puree, acai berry juice, guarana and lucuma fruit.

The importance and properties of maca root, banana puree, acai berries and lucuma fruit have already been discussed, as they are also included in other Super Mix supplements.

Guarana (*Paullinia cupana* Kunth var. *sorbilis* (Mart.) Ducke) has been traditionally eaten by indigenous communities in the Amazon region. It is often found in various types of energy drinks and stimulating teas due to its high caffeine content, which can reach up to 6 % in the seeds [48].

The anti-aging potential of guarana seed extract has also recently been demonstrated. The percentage of elderly people is globally growing and therefore effective therapies and therapeutic agents are being sought that can improve their quality of life [5].

Characteristics of Super Mix Immunity

The Super Mix Immunity supplement is a product that supports the immune system, mainly thanks to ginger and turmeric. It consists of powdered plant raw materials such as ginger rhizome, mango fruit, turmeric rhizome, lucuma fruit, camu camu berries and maca root. The importance and properties of lucuma fruit, camu camu berries and maca root have already been discussed earlier, as they are also included in other Super Mix supplements.

Ginger (*Zingiber officinale* Roscoe) has been used as a spice and remedy for over 200 years in traditional Asian medicine, especially in China. Ginger rhizome contains flavonoids and a number of phenolic compounds (gingerdiol, gingerol, gingerdione and shogaols) and sesquiterpenes. Moreover, it is a source of Fe, Mg, Ca and vitamin C. Ginger has anti-inflammatory, anti-apoptotic, anti-cancer, anti-pyretic, anti-platelet, anti-cancer, anti-hyperglycemic, antioxidant, anti-diabetic, anti-thrombotic and analgesic, cardiotonic and cytotoxic properties [31]. It is widely used for a cold, arthritis, cramps, a sore throat, rheumatism, muscle pain, vomiting, constipation, indigestion, hypertension, dementia, fever and infectious diseases [49]. Ginger oil is also used as a flavoring for soft drinks, as a spice in bakery products, confectionery, marinades, sauces and as a preservative [4].

The rhizome of turmeric (*Curcuma longa* L.) has been utilized for centuries for culinary purposes and food coloring, and also as an ingredient of various medicinal preparations, widely used in Ayurveda and traditional Chinese medicine. Intensive research on the health properties of turmeric conducted in recent years has shown that it has antioxidant, anti-inflammatory, neuroprotective, anticancer, hepatoprotective and cardioprotective effects [50]. The main bioactive substance in turmeric is curcumin, which, when combined with strengthening agents, provides many health benefits [32].

Results and discussion

In line with the changing lifestyle, especially in developed and developing countries, there is high demand for a wide range of powdered food products with emphasis on their quality [54]. The basic parameters that determine the intensity of chemical, physical and microbiological changes affecting the quality of products include water content and activity. At the same time, the level of initial water content and activity in products determines the ability to absorb and desorb it from/to the environment, depending on the potential variation of these parameters that are specific to a given product and its environment. Hence, these parameters determine the storage stability, which can therefore be a criterion for assessing the quality of Super Mix supplement mixtures in powder form.

Based on the assessment of the initial water content in the tested Super Mix I-V mixtures, statistically significant differences in the assessed parameter were found (Table 3).

Table 3. The initial water content and activity of the tested Super Mix I-V products

Tabela 3. Początkowa zawartość i aktywność wody w badanych produktach Super Mix I-V

Product / Produkt	Water content [g/100 g d.m.] / Zawartość wody [/100 g s.m.]	Water activity [-] / Aktywność wody [-]
I – Detox / Detox	6.89 ^b ± 0.05	0.369 ^d ± 0.001
II – Proteins / Proteiny	7.14 ^c ± 0.06	0.376 ^d ± 0.001
III – Antioxidants / Antyoksydanty	7.48 ^d ± 0.07	0.263 ^a ± 0.001
IV – Energy / Energia	6.56 ^a ± 0.06	0.314 ^b ± 0.001
V – Immunity / Odporność	8.03 ^e ± 0.09	0.350 ^c ± 0.002
<i>p</i>	0.000	0.000

Explanatory notes / objaśnienia:

The same letter symbols next to the mean values indicate no significant difference between the means in the LSD test / Jednakowe symbole literowe przy wartościach średniej wskazują na brak istotnych różnic między średnimi w teście NIR

The water content in the assessed Super Mix supplements was the highest for the Super Mix Immunity (V) product [8.03 g/100 g d.m.], and the lowest for the Super Mix Energy (IV) product [6.56 g/100 g d.m.]. Budzik (2023) found a higher water content for selected superfood powders [8]. In the camu camu powder the water content was 9.84 g/100 g d.m., in lacuma powder it was 8.75 g/100 g d.m., and in guarana powder it had the lowest value of 7.91 g/100 g d.m.

In the case of the initial water activity, it was found that the highest value of this parameter was characteristic of Super Mix Proteins (II) and Detox (I), and the water activity values obtained did not differ statistically significantly. The remaining tested products were characterized by a statistically significantly lower level of water activity.

The lowest level of water activity was observed in Super Mix Antioxidants (III) (Table 3).

Based on the results obtained, it can be supposed that the differences in the water content and activity in the tested Super Mix supplements were probably determined by the production technology, i.e., the parameters of the drying process used by the manufacturer, as well as the properties of individual ingredients and their percentage share in the Super Mix supplement mixtures.

The next assessed parameter was color, which is an important factor determining the choice of products by consumers [51]. In the case of Super Mix supplement mixtures, the color of the product, depending on the raw material composition, may contribute to the positive reception of the product or its rejection. The color assessment was carried out based on the analysis of the L^* index (brightness), a^* index (red color) and the b^* (yellow color) index. The values of the color parameters are presented in Table 3.

ANOVA followed by the LSD test showed that all studied products differed statistically significantly in terms of all assessed color indicators: L^* , a^* and b^* (Table 3). The value of the L^* parameter was the highest in the Super Mix Immunity product (V), and lower levels were recorded in the Super Mix Energy (IV), Mix Proteins (II) and Mix Detox (I). The Super Mix Antioxidants (III) had the lowest average level of the L^* index. The values of the a^* color index in the Super Mix Detoks product (I) reached negative values, which indicated and confirmed the green shade of the raw materials resulting from the chlorophyll content in the product ingredients (moringa Bio, chlorella and spirulina). The b^* parameter (color change from blue to yellow) had positive values for all tested Super Mix products (Table 4) and the values obtained for the individual Super Mixes assessed were statistically significantly different.

Based on the assessment of the L^* , a^* and b^* indicators, it was found that the obtained color parameters of the tested Super Mix products were determined primarily by the color of individual ingredients, related to the content of dyes included in the formulas.

Table 4. Color parameters

Tabela 4. Parametry barwy

Product / Produkt	L^*	a^*	b^*
I – Detox / Detox	$51.0^b \pm 0.36$	$-6.5^a \pm 0.06$	$14.7^b \pm 0.13$
II – Proteins / Proteiny	$61.7^c \pm 0.37$	$4.0^c \pm 0.10$	$18.6^c \pm 0.05$
III – Antioxidants / Antyoksydanty	$47.5^a \pm 0.32$	$6.7^c \pm 0.11$	$11.7^a \pm 0.20$
IV – Energy / Energia	$65.9^d \pm 0.24$	$4.8^d \pm 0.24$	$19.1^d \pm 0.03$
V – Immunity / Odporność	$69.7^e \pm 0.15$	$2.3^b \pm 0.13$	$47.6^e \pm 0.04$
<i>p</i>	0.000	0.000	0.000

Explanatory notes / objaśnienia:

The same letter symbols next to the mean values indicate no significant difference between the means in the LSD test/Jednakowe symbole literowe przy wartościach średniej wskazują na brak istotnych różnic między średnimi w teście NIR

Another parameter characterizing the quality of the tested Super Mixes was the kinetic angle of repose (Table 5). The angle of repose is the minimum angle that forms between the horizontal plane and the top layer of the inclined surface, at which the process of pouring of the bulk material begins. The smaller the values of the angle of repose are, the more susceptible the material is to spontaneous pouring. The cohesive forces between the particles of the tested products and the frictional resistance determine the angle of repose of the dry material. Additionally, the pouring of material is related to the product humidity, which is especially important in the case of mixtures – the higher the humidity is, the larger the angle of repose becomes [34]. The measurements of the angle of repose can be used to select appropriate materials for the construction of devices for packing bulk materials, as well as unit containers made of various types of packaging materials, that is, it is information that is important mainly for the manufacturer to design and select appropriate packaging [52]. Knowledge of the selected physicochemical properties is an important issue from the point of view of managing its transport and storage, in particular in production plants, as well as at home. The fluidic properties of foodstuffs, estimated on the basis of parameters such as the static and kinetic angle of repose and bulk density, play a particularly important role during the transport of products on the technological line. The static and kinetic angle of repose, on the other hand, determines the size of the storage area, storage capacity and silo unloading speed [52]. The LSD test showed that the angle of repose from the rough surface was the highest in the Super MIX Energy (IV) product (despite the lowest water content) and in the Super Mix Resistance (V) product. A lower level of the angle of repose value was recorded in the Mix Proteins (II) and the Mix Antioxidants (V). The lowest value of the angle of repose was found in Super Mix Detox (I), which had a low water content (Table 3). The obtained values of the angle of repose indicated that the tested super Mix supplements did not exhibit flow properties (Table 2).

An important parameter determining the quality, and more precisely – the flowability, of the tested products was the static angle of repose (cone angle). The higher the cone angle, the lower the flowability of the product. Products with a cone angle below 40° are characterized by fluidity and above 50° by low flowability [36, 47, 52]. In the tested products, the value of the filling angle ranged from 88.7 to 89.2° , indicating very poor flowability of the tested Super Mix products.

Table 5. Cone angle and the angle of repose of the tested Super Mix I–V products

Tabela 5. Kąt zsypania i nasypu badanych produktów super Mix I–V

Product / Produkt	Angle of repose [°] / Kąt zsypania [°]	Cone angle [°] / Kąt nasypu [°]
I – Detox / Detox	58.70 ^a ± 1.03	89.20 ^b ± 0.021
II – Proteins / Proteiny	78.00 ^c ± 3.22	89.20 ^b ± 0.048
III – Antioxidants / Antyoksydanty	69.30 ^b ± 1.03	88.70 ^a ± 0.035
IV – Energy / Energia	81.30 ^{cd} ± 1.03	89.00 ^b ± 0.022
V – Immunity / Odporność	82.00 ^d ± 1.03	89.00 ^b ± 0.034
<i>p</i>	0.000	0.000

Explanatory notes / objaśnienia:

The same letter symbols next to the mean values indicate no significant difference between the means in the LSD test / Jednakowe symbole literowe przy wartościach średniej wskazują na brak istotnych różnic między średnimi w teście NIR

Another quality characteristic of the tested Super Mix supplements related to the trade and storage of bulk goods was loose and tapped bulk density (Table 6). Loose bulk density and tapped bulk density are closely related to the porosity of the bulk material. This term includes two concepts – external intergranular porosity and internal porosity. The first is characterized by the presence of empty spaces between particles throughout the system, while the second describes a network of capillaries and pores that occur within a single particle. Fine-grained cohesive loose products in the form of powder are characterized by high porosity of the loosely packed bed, which is caused by the formation of large free spaces between the particles [20]. On the basis of the conducted research, it was shown that the highest loose and tapped density characterized the Super Mix Energy (IV), Super Mix Resistance (V) and Super Mix Antioxidants (III), and statistically significant differences in the assessed parameters were found between the products (Table 6).

Table 6. The selected physical properties of the tested products Super Mix I – V

Tabela 6. Wybrane właściwości fizyczne badanych produktów Super Mix I – V

Product / Produkt	q_L [g/cm ³]	q_U [g/cm ³]	HR	IC
I – Detox / Detox	0.462 ^a ± 0.004	0.661 ^a ± 0.003	1.43 ^d ± 0.005	30.20 ^d ± 0.26
II – Proteins / Proteiny	0.465 ^a ± 0.005	0.682 ^b ± 0.001	1.47 ^c ± 0.019	31.80 ^c ± 0.87
III – Antioxidants / Antyoksydanty	0.548 ^b ± 0.002	0.712 ^c ± 0.011	1.30 ^a ± 0.016	23.00 ^a ± 0.94
IV – Energy / Energia	0.582 ^d ± 0.005	0.817 ^c ± 0.017	1.40 ^c ± 0.020	28.70 ^c ± 1.03
V – Immunity / Odporność	0.562 ^c ± 0.007	0.754 ^d ± 0.003	1.34 ^b ± 0.013	25.50 ^b ± 0.72
<i>p</i>	0.000	0.000	0.000	0.000

Explanatory notes / objaśnienia:

q_L – loose density, q_U – tapped density, HR – Hausner ratio, IC – Carr index, The same letter symbols next to the mean values indicate no significant difference between the means in the LSD test / q_L – gęstość

luźna, q_U – gęstość utręszona, HR – współczynnik Hausner’a, IC – indeks Carr’a, Jednakowe symbole literowe przy wartościach średniej wskazują na brak istotnych różnic między średnimi w teście NIR

Hausner ratio (HR) and Carr index (IC) are related to loose and tapped bulk density. They also characterize the flowability of powders. The values of the $HR < 1.2$, $HR = 1.2 \div 1.4$ and $HR > 1.4$ correspond to low, medium and high cohesiveness of the product, respectively [18]. The Carr index (IC) also determines the flowability of a given product. Based on the IC value, it is assumed that materials with $IC < 18$ have very good flowability, medium flowability for $IC = 18 \div 25$, poor flowability with IC 25 to 30, and very poor flowability when $IC > 30$ [18]. Based on the obtained index values, it was found that the lowest HR and IC values were characteristic of product III, and the highest were found for product II. F-test confirmed the existence of statistically significant differences between the tested Super Mix products in terms of HR and IC values. Further, it was found that products I and II exhibited the properties of a coherent powder with high cohesiveness and very poor flowability. On the other hand, products III–V were characterized by medium cohesiveness and poor flowability (Table 6).

In order to determine the strength and direction of the correlations between the measured physicochemical parameters, the values of the Pearson’s linear correlation coefficient (r) were calculated. The null hypothesis about the lack of the linear correlation was verified, i.e., the hypothesis about the lack of a significant difference between the absolute value of the coefficient and the value of 0. This verification was performed using the Student’s t-test with a significance level of $p = 0.05$.

The results of the Student’s t-test indicated a statistically significant correlation between loose density and tapped density, angle of repose (directly proportional correlation), as well as loose density and Hausner ratio (HR), Carr index (IC), cone angle and water activity (inversely proportional relationship). Statistically significant correlation was found also between the tapped density and the angle of repose, cone angle and water activity. In the case of the Hausner ratio (HR) and the Carr index (IC), a directly proportional correlation was obtained with the cone angle and water activity. Of all the correlations between physicochemical features, the strongest one was found between the cone angle and the water activity of the tested Super Mix I–V products (Table 7).

The final stage of the research was the assessment of the storage quality of Super Mix products based on the determination of sorption isotherms. The sorption isotherms of the tested products (Fig. 1) represented the physical sorption process taking place on porous bodies and were characterized by a sigmoidal shape, showing similarity to type II isotherms in the classification of Brunauer et al. [8]. Sorption isotherms were characterized by continuity throughout the entire range of ambient water activity $a_w =$

0.07 ÷ 0.92, which indicated that in the tested products there was no change in the structure determined by the increased order of individual components.

Table 7. Correlations between physicochemical parameters (Pearson correlation coefficient values)

Tabela 7. Współzależności pomiędzy cechami fizykochemicznymi (wartości współczynnika korelacji Pearsona)

	qL [g/cm ³]	qU [g/cm ³]	HR	IC	Angle of repose [°] / Kąt zsypu [°]	Cone angle [°] / Kąt nasypu [°]	a _w [-]	Water content [g/100 g d.m.] / Zawartość wody [g/100 g s.m.]
qL [g/cm ³]	1.000	0.897*	-0.671*	-0.656*	0.596*	-0.715*	-0.655*	0.190
qU [g/cm ³]		1.000	-0.275	-0.255	0.707*	-0.399*	-0.386*	-0.097
HR			1.000	0.999	-0.095	0.884*	0.783*	-0.561
IC				1.000	-0.088	0.891*	0.788*	-0.566
Angle of repose [°] / Kąt zsypu [°]					1.000	-0.145	-0.032	0.225
Cone angle [°] / Kąt nasypu [°]						1.000	0.951*	-0.305
a _w [-]							1.000	-0.069
Water content [g/100 g d.m.] / Zawartość wody [g/100 g s.m.]								1.000

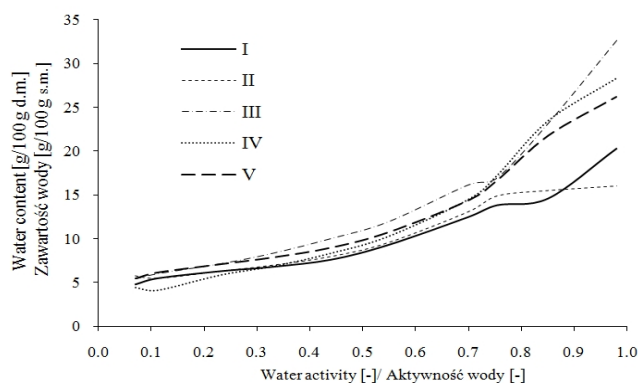


Fig. 1. The sorption isotherms of the tested products determined in an environment with $a_w = 0.07 \div 0.92$, over a period of 90 days, at 20 °C

Rys. 1. Izoterma sorpcji badanych produktów wyznaczona w środowisku o $a_w = 0,07 \div 0,92$, w czasie 90 dni, w temperaturze 20 °C

Analyzing the course of the determined sorption isotherms, it was found that in the Super Mix I, II and V products (in the area of the sorption isotherm I), in the water activity range $a_w = 0.07 \div 0.33$, the process of water desorption took place. In Super Mix III and IV products, the desorption process took place in the water activity range $a_w = 0.07 \div 0.23$ (Fig. 1). The highest water content in the first sorption area ($a_w = 0.07 \div 0.33$) was found for Super Mix V, 7.8411 [g/100 g d.m.]. In the case of areas II and III of the sorption isotherm, product III had the highest equilibrium water content (Fig. 1, Table 8).

Table 8. The equilibrium water content of the tested products stored in an environment with water activity $a_w = 0.07 \div 0.92$

Tabela 8. Równowagowa zawartość wody w badanych produktach przechowywanych w środowisku o aktywności wody $a_w = 0,07 \div 0,92$

Water activity of environment a_w [-] / Aktywność wody środowiska a_w [-]	The equilibrium water content after the storage process [g/100 g d.m.] / Równowagowa zawartość wody po procesie przechowywania [g/100 g s. m.]					<i>p</i>
	I – Detox / Detox	II – Proteins / Proteiny	III – Antioxidants / Antyoksydanty	IV – Energy / Energia	V – Immunity / Odporność	
0.07	4.77 ^b ±0.008	5.42 ^c ±0.005	5.46 ^c ±0.012	4.41 ^a ±0.002	5.45 ^c ±0.044	0.000
0.11	5.45 ^b ±0.014	5.44 ^b ±0.007	6.04 ^c ±0.004	4.39 ^a ±0.599	6.11 ^c ±0.005	0.000
0.23	6.27 ^d ±0.004	6.25 ^d ±0.006	7.19 ^c ±0.007	5.85 ^a ±0.003	7.06 ^b ±0.001	0.000
0.33	6.78 ^a ±0.001	6.95 ^c ±0.010	8.39 ^c ±0.002	6.84 ^b ±0.006	7.84±0.003	0.000
0.44	7.59 ^a ±0.003	7.94 ^b ±0.007	10.07 ^e ±0.001	8.33 ^c ±0.008	8.96 ^d ±0.002	0.000
0.55	9.24 ^a ±0.051	9.52 ^b ±0.004	11.99 ^e ±0.020	10.25 ^c ±0.002	10.68 ^d ±0.054	0.000
0.69	12.41 ^a ±0.023	13.01 ^b ±0.004	16.16 ^e ±0.026	14.44 ^d ±0.029	14.34 ^c ±0.007	0.000
0.75	13.73 ^a ±0.123	14.72 ^b ±0.007	16.20 ^c ±0.048	17.22 ^e ±0.002	16.67 ^d ±0.018	0.000
0.85	14.51 ^a ±0.120	15.76 ^b ±0.015	23.25 ^d ±0.036	23.49 ^e ±0.036	21.80 ^c ±0.011	0.000
0.92	19.95 ^b ±0.050	15.87 ^a ±0.013	32.75 ^d ±0.096	27.14 ^c ±0.094	26.91 ^c ±0.022	0.000

Explanatory notes / Objasnienia:

The same letter symbols next to the mean values indicate no significant difference between the means in the LSD test / Jednakowe symbole literowe przy wartościach średniej wskazują na brak istotnych różnic między średnimi w teście NIR

In order to estimate the selected parameters of the surface microstructure and the course of the sorption phenomenon, empirically determined water sorption isotherms were modeled with the BET isotherm in the range of $a_w = 0.07\text{--}0.33$. The parameters of the BET equation are presented in Table 9.

Table 9. The parameters of BET isotherm describing the sorption of water by the tested products in water activity range $a_w = 0.07 \div 0.33$

Tabela 9. Parametry izotermy BET opisującej sorpcję wody przez badane produkty w zakresie aktywności wody $a_w = 0,07 \div 0,33$

Product / Produkt	v_m	c_e	R^2	SS	RMS	PS
I – Detox / Detox	4.58	266.47	0.9964	0.07	0.06	161.05
II – Proteins / Proteiny	4.59	106.39	0.9995	4.35	1.89	161.25
III – Antioxidants / Antyoksydanty	5.72	111.09	0.9999	1.69	1.30	200.87
IV – Energy / Energia	4.80	41.40	0.9922	1.31	1.89	168.66
V – Immunity / Odporność	5.30	82.38	0.9986	0.02	0.02	186.34

Explanatory notes / Objasnienia:

v_m – the capacity of the monomolecular layer, c_e – energy constant, R^2 – correlation coefficient, SS – the sum of squares of deviations of theoretical values from empirical values, RMS – root mean square error, PS – specific surface area of sorption / v_m – pojemność warstwy monomolekularnej, c_e – stała energetyczna, R^2 – współczynnik korelacji, SS – suma kwadratów odchyleń wartości teoretycznych od empirycznych, RMS – średni błąd kwadratowy, PS – powierzchnia właściwa sorpcji

Based on the capacity of the monomolecular layer, v_m , it can be concluded that products III and V had a higher storage stability compared to the other products. The significant development of the surface of the monomolecular layer protects food products against deterioration of quality as a result of absorbing a certain amount of water [43].

The energy constant c_e reflects the difference between the desorption enthalpy of the monolayer and the enthalpy of vaporization of the liquid adsorbent. The obtained results of the c_e constant ($c_e \geq 2$) confirmed the sigmoidal shape of the sorption curve.

Based on the comparison of SS and RMS values, it was found that the BET model obtained analytically adequately described the sorption process of the tested Super Mix I–V products (Table 9). The root mean square error (RMS) did not exceed 10 % RMS, indicating good compliance of the model fit to the sorption data in the studied range of water activity (Table 9).

Based on the capacity of the monomolecular layer v_m , the specific sorption surface was calculated. The specific sorption surface, defined as the surface per unit mass [m^2/g], is one of the most important parameters characterizing the ability of adsorbents to adsorb gases, vapors and ions. Based on the results obtained, it was found that product III had the highest specific sorption surface, 200.87 m^2/g .

Conclusions

1. The article presents the measurements and analysis of the physicochemical parameters of novel multi-ingredient dietary supplements relevant to storage. These products were characterized taking into account their composition and declared

- properties. Particular attention was paid to ingredients from exotic plants that have not been used to such an extent in dietary supplements so far.
2. The assessment of the selected physicochemical properties of Super Mix multi-ingredient dietary supplements showed that the tested products differed statistically significantly in terms of initial water content and activity. Based on the obtained values of the angle of repose and cone angle, it was found that the products had no flow properties and were characterized by poor flowability, which was also confirmed by the obtained values of the Hausner ratio and the Carr index. Moreover, based on the HR and IC values, it was found that products I and II exhibited the properties of a coherent powder with high cohesiveness and very poor flowability, while products III–V were characterized by medium cohesiveness and poor flowability. Based on the analysis of sorption properties and on the obtained value of the capacity of monomolecular layer determined via the BET equation, it was found that product III was characterized by highest intrinsic storage stability.
 3. The results of the research on the physicochemical properties of multi-ingredient dietary supplements can be used to predict the durability of such products and to design packaging that takes into account the behavior of plant-based supplements during storage. The results obtained may also constitute the basis for further research on improving the composition and properties of multi-ingredient dietary supplements.

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FIZYKOCHEMICZNE WŁAŚCIWOŚCI INNOWACYJNYCH WIELOSKŁADNIKOWYCH SUPLEMENTÓW DIETY

Streszczenie

Wprowadzenie. W ostatnich latach suplementy diety są przedmiotem zainteresowania tak konsumentów jak i producentów żywności i przemysłu farmaceutycznego. Suplementy diety nie są produktami leczniczymi, i za takie nie są uznawane. Jednak większość społeczeństwa w wielu krajach traktuje suplementy diety na równi ze środkami leczniczymi. W artykule zwrócono uwagę na innowacyjne suplementy diety zawierające w swoim składzie wiele składników pochodzących z roślin tropikalnych, które do tej pory nie były w tak dużym stopniu wykorzystywane w suplementach. Celem niniejszej pracy było określenie fizykochemicznych właściwości wieloskładnikowych suplementów diety Purella Super Mix. Suplementy te są produkowane przez firmę Purella Food, a w skład tej serii wchodzi pięć produktów o różnicowanym działaniu. Dokonano charakterystyki produktów Purella Super Mix pod kątem ich składu i wynikającego stąd znaczenia dla zdrowia człowieka. Przeprowadzono szereg analiz fizykochemicznych Purella Super Mix, które do tej pory w takim zakresie nie były publikowane. Zbadano zawartość wody, aktywność wody, gęstość nasypową oraz kąt zsypania i nasypu, określono współczynnik Hausnera i indeks

Carr'a które stanowią ważną informację tak dla procesu technologicznego, jak i dla kolejnych etapów cyklu życia wyrobu.

Wyniki i dyskusja. Wyniki badań właściwości fizykochemicznych innowacyjnych wieloskładnikowych suplementów diety mogą być wykorzystane do prognozowania trwałości tych produktów oraz projektowania opakowań uwzględniających zachowanie suplementów podczas przechowywania. Uzyskane wyniki mogą również stanowić podstawę do dalszych badań nad doskonaleniem składu i właściwości wieloskładnikowych suplementów diety.

Słowa kluczowe: suplementy diety, analiza fizykochemiczna, zawartość wody, aktywność wody, sypkość

