

## EVALUATION OF PHYSICOCHEMICAL AND USABLE PROPERTIES OF HAIR SHAMPOOS CONTAINING SUPERCRITICAL CARBON DIOXIDE STRAWBERRY SEED EXTRACT

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**Abstract:** This paper presents the results of a study of selected physicochemical properties for prototype hair shampoos containing strawberry seed extract obtained under supercritical CO<sub>2</sub> conditions. Four formulations were developed and prototypes of hair shampoos were created. It was found that the properties of the shampoos depended on the concentration of the hydrophobic extract used. As a function of the extract concentration, the viscosity of the cosmetic, the foam stability index values and the ability to emulsify fatty soils are lowered. The introduction of the extract into the cosmetic composition resulted in a reduction in the cosmetic lightness and also in a yellow-green color of the shampoo.

**Keywords:** shampoo, extract, strawberry seeds, quality.

### 1. INTRODUCTION

The basic treatment for proper hair and scalp hygiene is shampooing. Hair shampoos are most often cosmetics in a liquid form, although there are also other forms of cleansing cosmetics for the scalp and hair on the market, such as bars, powders, sprays [Zięba et al. 2019a,b; Ociczek and Zięba 2020]. The base of traditional liquid hair shampoos is water, in which the proportion in the formula may be as high as several dozen percent. Water in a cleansing cosmetic acts as a solvent for the key ingredients responsible for such things as removing dirt from the surface of the hair and scalp (sebum, dust, environmental contaminants, residues from styling cosmetics).

There are also other ingredients in the shampoo composition, such as conditioning, foaming, healing, sequestering properties, etc. Each shampoo formulation also contains viscosity modifiers, pH-regulating compounds and microbial growth inhibitors [Ananthapadmanabhan et al. 2004; Kumar and Mali 2010]. The composition of modern shampoo formulations should ensure that,

in addition to properly removing dirt, it should also fulfil additional functions, such as: providing shine, softness and fluffiness without causing the hair to become electrified, facilitating combing of the dry and wet hair, facilitating hair styling, etc. [Ananthapadmanabhan et al. 2004; Kumar and mali 2010; Zięba et al. 2019a,b; Ocieczek and Zięba 2020].

Consumer demands are now focused on the natural composition of cosmetics, driven by concerns for the health of the users and the environmental impact of the product. As a result, producers are focusing on optimizing the composition of the products, reducing the proportion of synthetically derived raw materials in the formulations in favor of components derived from natural sources. One interesting possibility of using raw materials of natural origins in the composition of shampoos is the use of extracts obtained under CO<sub>2</sub> supercritical conditions [Michorczyk, Vogt and Ogonowski 2015; Sikora et al. 2015].

Extraction conducted under supercritical CO<sub>2</sub> conditions is an innovative and competitive method for commonly used technologies. Carbon dioxide has a high penetration capacity into the plant material. The process of CO<sub>2</sub> extraction under supercritical conditions takes place at a relatively low temperature of about 50°C. The main extracting agent (compressed carbon dioxide) is volatilized after the process, and the resulting product has a very high level of purity. In addition, the relatively low temperature of the process allows valuable ingredients (e.g. vitamins, proteins, phytosterols) to be retained from a cosmetic point of view [Janiszewska and Witrowa-Rajchert 2005; Michorczyk, Vogt and Ogonowski 2015; Sikora et al. 2015].

Strawberries (*Fragaria Ananasa*) have traditionally been considered a raw material for the food industry. However, the literature on the subject confirms that this plant can also be of interest to the cosmetic and pharmaceutical industries [Gündüz 2016]. In Poland, the strawberry harvest in 2022 was 185,000 tons, accounting for a 31% share of the national berry harvest [www.gov.pl]. The strawberry is a seasonal crop, with fruit harvested in Poland between May and July. The fruit of the plant is used in industry mainly for its taste and nutritional values. Strawberry fruit is a source of fiber, vitamins C and K, as well as minerals (K, Ca, P, Mg, Mn) [Gündüz 2016]. Strawberry seed oil is a valuable source of antioxidants, gamma-tocopherol, linoleic acid, alpha-linolenic acid and oleic acid, and therefore may be a potential raw material for the cosmetic industry [Gündüz 2016; Minutti-López Sierra et al. 2019].

An extensive study on the presence of bioactive components in the oils extracted by CO<sub>2</sub> extraction from the berry seeds was conducted by Milala et al. [2018]. They used strawberry, chokeberry and raspberry seeds as plant material. The researchers showed that strawberry seed oil contained carotenoids at a level of 115 mg/L and the total tocopherols content was about 550 mg/kg. The oil was also reported to contain the fatty acids:  $\alpha$ -linolenic (about 30% of the content), linoleic

(about 49% of the content), palmitic (about 4.3% of the content), oleic (about 15% of the content) and stearic (about 1.5% of the content).

Villamil-Galindo et al. [2023] obtained strawberry extracts using ultrasound-assisted extraction (UAE). The plant materials were strawberry agroindustrial by-products. The bioactive potential of the extracted compounds was evaluated in cellular models. The main objective of this study was to investigate the UEA kinetic parameters of phenolic compounds from strawberries and their stability during in vitro fermentation. The bioactive potential of the extracted compounds was investigated and evaluated in cellular models. The researchers found that the total phenolic compound (TFC) content of the extracts used was in the range of 5.1–12.2 g GAE/kg, depending on the UAE conditions used. Additionally, the extracts from strawberry agro-industrial by-products displayed promising bioactivity, including anticancer potential in colorectal cancer cell lines and anti-inflammatory properties.

Gasparrini et al. [2015], on the other hand, investigated the properties and applicability of strawberry extract in a sunscreen preparation. The results of their experiments confirmed that the strawberry extract used had a high antioxidant capacity, high content of anthocyanins and vitamin C and beta-carotene, resulting in a protective effect on skin cells against UVA-induced damage. They confirmed their hypotheses in their other studies [Gasparrini et al. 2017].

Interesting research on the use of bioactive components extracted from strawberry seeds was conducted by Wójciak et al. [2024]. The results of their research indicated the antioxidant properties of the phenolic-rich fraction isolated from strawberry seeds. The results of their experiments also demonstrate the ability to scavenge free radicals in human skin fibroblasts and the possibility of using these properties in various types of cosmetics.

The literary data [Gasparrini et al. 2015; 2017; Michorczyk, Vogt and Ogonowski 2015; Sikora et al. 2015; Wójciak et al. 2024] indicate that the properties of strawberry fruit and raw materials extracted from it are interesting from a cosmetic point of view. The aim of this study was to determine selected physicochemical and usable properties of hair shampoos containing strawberry extract obtained under supercritical carbon dioxide conditions as an ingredient.

## 2. MATERIALS AND EXPERIMENTAL METHODS

### 2.1. Materials

The shampoo formulas contained: Disodium Laureth Sulfocinate (Texapon SB, Basf, Germany), Cocamidopropyl Betaine (Dehyton K, Basf, Germany), Cocamide DEA (Rokamid KAD, PCC Rokita S.A., Poland), *Fragaria Ananassa* Strawberry Seed Oil (ECO<sub>2</sub>, Łukasiewicz Research Network – New Chemical Syntheses Institute, Poland), Glycerin (Glycerin, Pure Chemical, Poland), PEG-40

Hydrogenated Castor Oil (Rokacet HR40, PCC Rokita S.A., Poland), Xanthan Gum (Kelzan, CP Kelco, Germany), Citric Acid (Citric Acid, POCH S.A., Poland), Sodium Benzoate and Potassium Sorbate (KEM BS, Pol-Nil S.A., Poland).

## 2.2. Formulations

Four models of hair shampoo formulations (O, A, B, C, D) containing different concentration of *Fragaria Ananassa* Strawberry Seed Oil were developed.

The qualitative and quantitative compositions of the raw materials studied are shown in Table 1.

**Table 1.** Composition of model hair shampoos

Ingredient (according to INCI)	Formulation			
	O	A	B	C
	Concentration [% wt.]			
Disodium Laureth Sulfococinate	10.5			
Cocamidopropyl Betaine	2.0			
Cocamide DEA	2.0			
<i>Fragaria Ananassa</i> Strawberry Seed Oil	0	0.25	0.50	1.00
Glycerin	2.0			
PEG-40 Hydrogenated Castor Oil	2.0			
Xanthan Gum	0.7			
Citric Acid	to pH 5.5			
Sodium Benzoate and Potassium Sorbate	0.75			
Water	up to 100.0			

Source: own study.

The manufacture of the shampoo required several steps, including the preparation of pre-mixes of raw materials that are difficult to mix with water, which forms the base of the cosmetic. To this end, a pre-mix of Glycerin and Xanthan Gum was prepared by weighing out these ingredients and then vigorously blending them together. In small portions, the resulting mixture was introduced into the weighed water heated to approximately 40°C. The resulting mixture was stirred with a mechanical stirrer until the ingredients were combined. Similarly, a pre-mix of *Fragaria Ananassa* Strawberry Seed Oil was prepared with PEG-40 Hydrogenated Castor Oil acting as a solubilizer in the shampoo formulation. The pre-mix of hydrophobic extract and solubilizer prepared in this way was introduced in small portions into the previously prepared mixture of water, Glycerin and Xanthan Gum. Then appropriately measured amounts were introduced into the resulting mixture: Disodium Laureth Sulfococinate, Cocamidopropyl Betaine and Cocamide DEA. The preparation was cooled to room temperature while stirring until homogeneous. A preservative was added and the pH of the cosmetic was adjusted to 5.5.

### **2.3. Dynamic viscosity**

Dynamic viscosity was measured using a Brookfield DV-I+ viscosity meter (Brookfield, USA). Measurements were taken at 20 rpm, at room temperature (approximately 22°C). A total of three measurements were performed, after which the results were averaged.

### **2.4. Ability to emulsify fatty soils**

The assessment of the ability to emulsify fatty soils imitating sebum by the preparations was conducted according to the methodology described in the literature [Podkova-Zawadzka, Wasilewski and Zięba 2021] on the basis of Polish Standard PN-C-77003. The method determines the ability of shampoo to emulsify rapeseed oil used as an imitation of sebum, colored with Sudan red. The purpose of the dye is to facilitate detection of the point at which the formulation does not emulsify fatty soils (0 points – separation of clear drops or a layer of clear oil on the tube collar, 1 point – clear ring (more than 5 mm thick) of emulsified oil in the tube collar, 2 points – clear ring (3 to 5 mm thick) of emulsified oil in the tube collar, 3 points – clear ring (1 to 3 mm thick) of emulsified oil in the tube collar, 4 points – clear ring (less than 1 mm thick) of emulsified oil in the tube collar, 5 points – hardly recognizable ring in the tube collar, unstable emulsion in the volume phase, 6 points – no fringe in the flask neck, homogeneous emulsion).

## **2.5. Foam Stability Index (FSI)**

The method of measurement was in line with Polish Standard PN-ISO 696:1994 (Surface active agents – Measurement of foaming power – Modified Ross-Miles method). The foam volume was measured, produced by 500 ml of the sample solution (1 wt.%) falling from a height of 450 mm into a cylinder (1000 ml) containing 50 ml of the same solution. Measurements were conducted at 22°C. The final result was the arithmetic mean of three independent measurements. The Foam Stability Index (FSI) was calculated from the equation:

$$\text{FSI} = (V_{10} / V_1) \cdot 100\%$$

where:

- $V_{10}$  – foam volume after 10 min,
- $V_1$  – foam volume after 1 minute.

## **2.6. Color**

Color tests were performed using a CR 400 colorimeter from Konica Minolta. Each formulation was assessed in the C.I.E. system based on the measurement of three trichromatic components:  $L^*$ ,  $a^*$  and  $b^*$ . Each color was determined through three components:

- $L^*$  – lightness (intensity of color brightness; by comparing their  $L^*$  values, colors can be classified as either light or dark),
- $a^*$  – value between red and green,
- $b^*$  – value between yellow and blue [Klimaszewska et al. 2021].

## **2.7. Data analysis**

The points in the figures represent average values from a series of three or five independent measurements. The distribution was used to calculate confidence limits for the mean values. Confidence intervals, which constitute a measuring error, were determined for a confidence level of 0.90. Error values are presented in the figures.

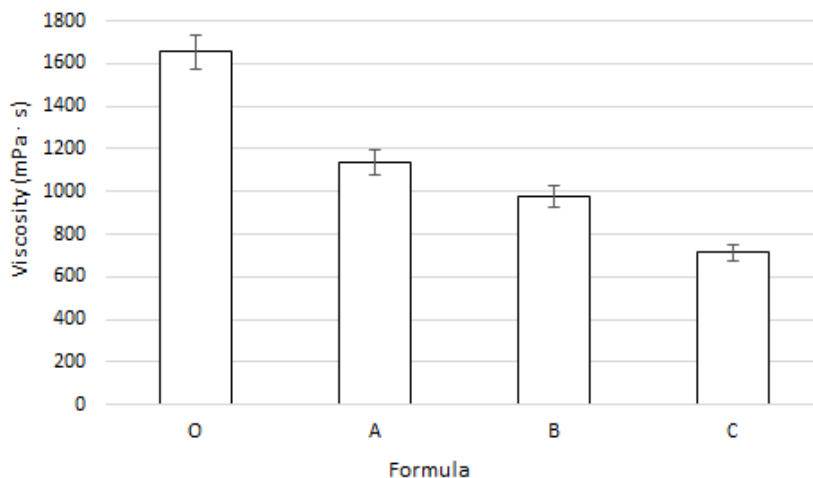
# **3. RESULTS**

## **3.1. Dynamic viscosity**

Dynamic viscosity ( $\eta$ ) is a very important parameter in assessing the quality of hair shampoos. A high  $\eta$  value is often equated by consumers with a high content of active substances and also with a high washing capacity of the cosmetic.

The introduction of certain substances into the formulation can sometimes affect the viscosity of the preparation.

The viscosity of hair shampoos with *Fragaria Ananassa* Strawberry Seed Oil was measured and the results are illustrated in Figure 1.



**Fig. 1.** Dynamic viscosity of hair shampoo prototypes with *Fragaria Ananassa* Strawberry Seed Oil

Source: own study.

The viscosity of the shampoo without the addition of *Fragaria Ananassa* Strawberry Seed Oil was 1653 mPa·s (Fig. 1). Enrichment of the formulation with the smallest share of the extract ( $c = 0.25\%$  wt.) resulted in a reduction of the measured value by almost one third in relation to the standard cosmetic (formulation O). Subsequent increases in the concentration of strawberry extract determined a further decrease in  $\eta$ , and the lowest viscosity was obtained for the sample containing 1% wt. of the proposed plant additive ( $\eta = 713$  mPa·s).

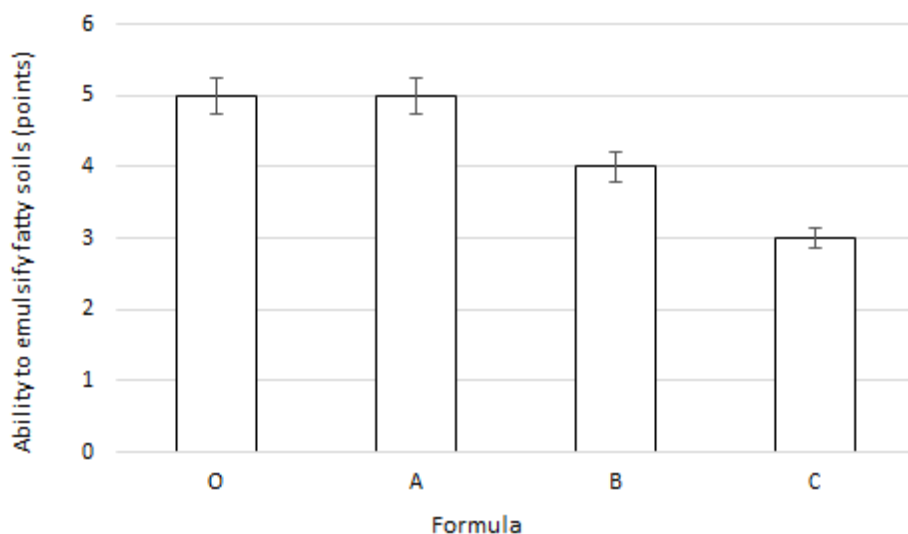
Godeto et al. [2023] used extracts received from the seeds of selected plants (*Reccinus Communis L.* seed; and *Lepidium Sativum L.* seed) to develop herbal shampoos. The variables in the formulations were the anionic surfactant content, concentration and type of plant extract. In this study, the viscosity of the shampoos was measured under different speed conditions, ranging from 0.3 to 12 rpm. The viscosity regulator in the compositions was Acacia Gum, used at a concentration of 4% wt. The  $\eta$  values of the shampoos decreased as a function of increasing rotational speed. In the case of shampoos with the addition of *L. Sativum* extract,  $\eta$  values similar (1265 and 1890 mPa·s) to those obtained in the present study were obtained for cosmetics without the extract and containing strawberry extract at a concentration of 0.25% wt.

For shampoos without added plant extracts, the viscosity of the product is usually around one thousand to several thousand mPa·s. In the case of commercial shampoos containing plant extracts, very often the viscosity of the cosmetic is several hundred mPa·s, while achieving a viscosity of several thousand mPa·s is problematic. Extracts are chemically very complicated mixtures of mainly organic compounds. Organic compounds can block the formation of structures (micelles) in the shampoo, which are responsible, among other things, for regulating the viscosity of the cosmetic. This is most often the case when the viscosity modifier of the cleansing cosmetic is Sodium Chloride. Problems with adjusting the concentration and type of viscosity modifier also apply to systems containing plant extracts, where the viscosity is regulated by plant gums, for example.

### 3.2. Ability to emulsify fatty soils

The surface of the scalp and hair can be affected by different types of dirt, including sweat, sebum, environmental pollutants, styling product residues and so on. The elementary task of a shampoo is to remove dirt, including that of a hydrophobic nature, from the surface of the scalp and hair. One of the steps in the washing process is the emulsification of the dirt in the washing bath.

The ability of the shampoo to remove fatty soils was assessed and the results are shown in Figure 2.



**Fig. 2.** Ability to emulsify fatty solids of hair shampoo prototypes with *Fragaria Ananassa* Strawberry Seed Oil

Source: own study.



The results of the ability to remove fatty soils for shampoos containing strawberry seed extract obtained under supercritical CO<sub>2</sub> conditions show a high ability score of 5 points for the base shampoo (formulation O) and the shampoo with a 0.25% wt. share of plant extract. The ability to emulsify fatty soils decreased with increasing proportions of the plant additive in the formulation up to the value of 3 points observed for formulation C. In practice, this score means a clear ring (less than 1 mm thick) of emulsified oil in the tube collar. The results of tests on the ability to remove greasy soiling may indicate that the use of the proposed shampoos in practice will not result in excessive degreasing of the skin and hair.

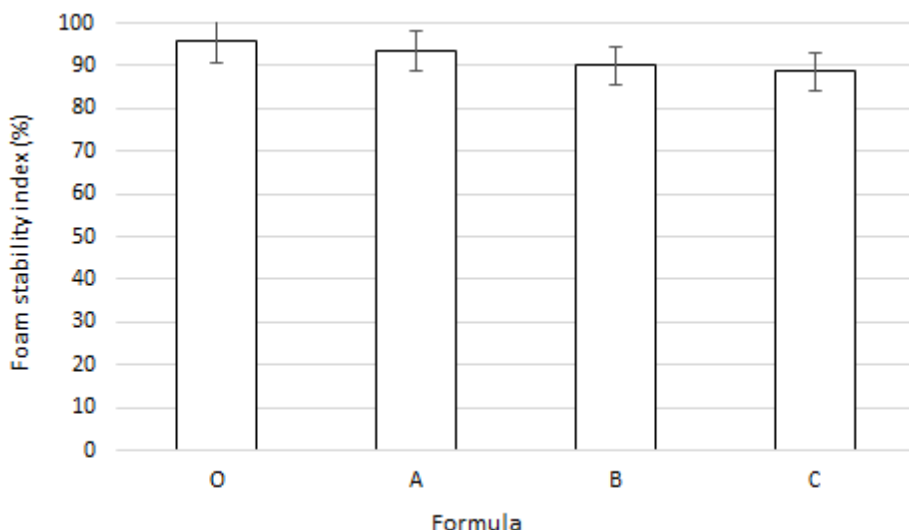
Wasilewski, Seweryn and Bujak [2016] evaluated the ability to emulsify fatty solids by dishwashing liquids containing blackcurrant seed extract obtained under supercritical CO<sub>2</sub> conditions. This work used a methodology in which the measurements of fat emulsification capacity was made by increasing the mass of added fatty soils by 0.2 g to the moment when the formulation no longer emulsifies fat. The researchers found that a decrease in the emulsifying capacity of fat soiling correlated with an increase in the proportion of currant extract in the dishwashing liquid formulation. They found the highest emulsification capacity of 20.2 g/l for the washing up liquid without an extract and the lowest (16.4 g/l) for the liquid containing 0.5% wt. extract. Despite using a different methodology, the trend observed by this team was the same as in the present study.

The lower the ability to emulsify fatty solids, the lower the ability to remove fatty soils from the hair and scalp. Shampoos with a lower capacity to remove fatty soils (3–4 points) also fulfil their cleaning role. However, it is important to remember that the cleansing capacity of the shampoo should be adapted to the hair type and scalp type. For example, in the case of children's skin or people with dry, atopic skin, excessive removal of sebum from the skin surface can lead to irritation, e.g. dryness, redness and itchy skin. Shampoos with a high cleansing potential is suitable for people with oily scalps and hair prone to oiliness.

### **3.3. Foam Stability Index (FSI)**

The ability to form a rich and long-lasting foam is a very important criterion for assessing the quality of hair shampoos. Consumers expect cleansing cosmetics to create a thick, creamy foam. However, too rich a foam can cause rinsing problems from the hair surface and scalp.

The Foam Stability Index (FSI) of the manufactured hair shampoo prototypes was assessed and the results are illustrated in Figure 3.



**Fig. 3.** Foam Stability Index of hair shampoo prototypes with *Fragaria Ananassa* Strawberry Seed Oil

Source: own study.

The FSI value observed for the basic shampoo was 95.6%. The addition of plant extract resulted in a uniform decrease in the measured value to FSI = 88.6% (formula C). Thus, a decrease in the measured parameter of approx. 8% with respect to the base shampoo (formula O) was obtained. In all cases, high values were obtained, ensuring that the consumer expectations were met.

FSI measurements for bath formulations containing Supercritical CO<sub>2</sub> extract from strawberry seeds were also conducted by Sikora et al. [2015]. The formulations of the cosmetics created by the team contained, among other things, strawberry seed extract (c = 0–2% wt.) and their viscosity was modified with sodium chloride. The developed bath products showed FSI values of approximately 93%, irrespective of the extract content in the formulation, and therefore as high as those obtained in the present study.

### 3.4. Color

Plants and the raw materials extracted from them contain a multitude of chemical compounds in their composition. Some of these have coloring properties (e.g. azulene, chlorophyll,  $\beta$ -carotene). The essences vary greatly in terms of their chemical content and may also have coloring substances in their composition [Bujak et al. 2021; 2022], which can impart color to the product when introduced. Color measurements were conducted for the original shampoos containing *Fragaria*

*Ananassa* Strawberry Seed Oil and for the cosmetic without the extract in the formulation.

The results of the measurements are shown in Table 2.

**Table 2.** Colorimetric characteristics of shampoo samples having with different concentrations of *Fragaria Ananassa* Strawberry Seed Oil

Formula	Parameter		
	L*	a*	b*
O	75.28 ±0.04	-0.12 ±0.01	-0.74 ±0.01
A	73.13 ±0.03	-0.34 ±0.01	0.81 ±0.02
B	72.64 ±0.03	-0.41 ±0.01	1.67 ±0.02
C	71.09 ±0.03	-0.54 ±0.01	1.98 ±0.02

Source: own study.

The brightness values (L\*) of the hair shampoo samples ranged from L\* = 71.09 to 75.28. It should be noted that the highest brightness (L\* = 75.28) was recorded for the cosmetic product without the added extract. With an increase in the percentage of extract in the formulation, a slight decrease of approx. 5% in the measured parameter was observed in relation to the base sample (sample O).

For the a\* parameter, the values ranged from a\* = -0.12 for the shampoo without extract to a\* = -0.54 for the C labelled cosmetic (C<sub>ECO2</sub> = 1% wt.). It can therefore be assumed that the color of the shampoos shifted slightly towards green as the proportion of the proposed plant material increased. In the case of b\* values, it was observed that they raised as the proportion of *Fragaria Ananassa* Strawberry Seed Oil in the shampoo formulation increased. For the reference cosmetic (symbol O), b\* was equal to -0.74, while for the shampoo containing the plant raw material, the value of the measured parameter b\* ranged from 0.81 (C<sub>ECO2</sub> = 0.25% wt.) to 1.98 (C<sub>ECO2</sub> = 1% wt.). Samples with added extract thus took on a yellow color. From a practical point of view, it can be summarized that shampoos to which the hydrophobic extract *Fragaria Ananassa* Strawberry Seed Oil was introduced were characterized by a green-yellow color, which was imperceptible to the human eye.

Confirmation of the possibility of imparting color to cosmetics through the use of plant derivatives is provided by research conducted by Bujak et al. [2021; 2022]. The team evaluated the effect of introducing aqueous-ethanol extracts from plants (e.g. field poppy, ternatene clitoria, safflower, evening primrose) into cosmetic creams. The researchers noted a significant effect of the extracts used on L\*, a\* and b\* values. The greatest effect on the measured parameters was recorded for the use of *C. Ternatea L.* extract, the use of which resulted in a blue-violet coloring of the sample and values of L\* = 68.19, a\* = 6.23, b\* = 18.92.

Thus, the researchers obtained  $a^*$  and  $b^*$  values that were higher than those obtained in this study.

#### 4. CONCLUSIONS

The aim of this study was to determine selected physicochemical and performance properties of hair shampoos containing strawberry seed extract obtained under supercritical carbon dioxide conditions as an ingredient. The following tests were conducted: dynamic viscosity, ability to emulsify fatty soils, Foam Stability Index and color evaluation of shampoos containing the plant additive.

The following conclusions were drawn from the laboratory results:

- an increase in the concentration of *Fragaria Ananassa* Strawberry Seed Oil results in a decrease in the  $\eta$  value of shampoos for hair. For optimal use and application properties, lower concentrations ( $c = 0.25\%$  wt.) of this extract should be considered in the formulation;
- the results of the evaluation of the ability to emulsify fatty soils showed an adequate ability of the shampoo prototypes to emulsify soiling. It should be noted that this ability decreased as a function of the increasing proportion of hydrophobic extract in the washing cosmetics;
- the shampoos developed were characterized by a very high capacity to form a stable foam. FSI values decreased as a function of increasing extract concentration;
- introducing the hydrophobic extract of *Fragaria Ananassa* Strawberry Seed Oil into the shampoo formulation resulted in lower  $L^*$ ,  $a^*$  and  $b^*$  values, and the developed cosmetics were characterized by a green-yellow color, but differences in individual parameters were imperceptible to the human eye. Basic parameter determinations ( $L^*$ ,  $a^*$ ,  $b^*$ ) can serve as a starting point for subsequent calculations (hue or general color difference) in the future.

In conclusion, the hydrophobic extract of *Fragaria Ananassa* Strawberry Seed Oil can form an alternative ingredient in hair shampoos. In order to make the shampoos more viscous, it would be necessary to use a higher concentration of viscosity adjuster or replace it with another modifier.

In considering the emulsification of fatty soils it is thought that the developed cosmetics could be dedicated to children or people with dry, sensitive skin; however, to determine this, it is planned to extend the experiment to evaluate the effect of the developed cosmetics on the moisturization level.

## 5. ACKNOWLEDGEMENTS

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