

Article

The Role of Burdock and Black Radish Powders Obtained by Low-Temperature Drying in Emulsion-Type Hair Conditioners

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Abstract: The aim of this study was to evaluate the potential role of burdock and black radish powders in emulsion-type hair conditioners. The studied plant powders were obtained by low-temperature drying. This method allows plants to retain many valuable nutrients, including vitamins or phytosterols, which have a positive effect on the condition of hair and skin. For the selected plant powders, the content of vitamin C and total polyphenolic content, as well as the degree of reduction of DPPH free radicals, were determined. Burdock and black radish powders proved to contain polyphenolic compounds and exhibited antioxidant activity, which is particularly evident in burdock powder. The plant material under study was also proven to contain vitamin C. The following stage of this study involved designing the formulations and preparing seven hair conditioner emulsions containing different plant-based powders at various concentrations. In the next step, the cosmetic prototypes were evaluated for their physicochemical and functional properties. The hair conditioners were found to have satisfactory functional characteristics, including dynamic viscosity, yield stress, and consistency. Colorimetric analysis showed that an increase in the concentration of burdock and black radish powders obtained by low-temperature drying in hair conditioners resulted in a more saturated color compared to the reference sample. The test results indicated that an increase in the concentration of the plant-derived powders contributes to an increase in the intensity of the yellow color of the samples.

Keywords: cosmetic emulsions; hair conditioners; plant powders; burdock; black radish



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1. Introduction

In the contemporary world, the appearance of hair is believed to be a very important factor across many societies. Healthy sleek hair is recognized as one of the major determinants of beauty. In addition, hair is a significant contributor to people's well-being and self-esteem. Hair condition and appearance are affected by a range of factors, including exposure to solar radiation and environmental pollution [1,2], but also stress and age [3]. Recent years have seen a growth in the market for cosmetic products designed for hair care, nourishment, and regeneration [4]. Hair conditioning products increase hair volume, softness and shine, reduce frizz, improve combability, stimulate new hair growth, and cleanse the hair [5]. Consequently, new raw materials with proven benefits for hair care are constantly being sought. Consumers are particularly interested in hair conditioners containing natural plant-derived substances. Plants are a rich source of active substances that improve the appearance and growth of hair and have a beneficial effect on the scalp.

Burdock (*Arctium lappa*) is a plant of the family *Asteraceae*. It is found across Europe, North America, South America, and Asia, where its therapeutic properties have been used

for hundreds of years [6]. Burdock is a plant that grows up to 1 m tall. Young cylinder-shaped roots produce branches with a thickness of 3 to 6 cm [6]. The parenchyma ranges in color from white to white-yellow, depending on the age of the plant [2]. According to the literature reports [7,8], burdock is a source of plenty of bioactive compounds, including phytoestrogens, flavonoids (e.g., luteolin, rutin, quercetin), fatty acids (e.g., linolenic acid, stearic acid), carboxylic acids (e.g., caffeic acid, chlorogenic acid), and saccharides (e.g., fructose, sorbitol, galactose, mannitol, mannose), which are present in seeds, fruit, roots, and leaves. Burdock is also rich in vitamins B1, B2, C and A, and contains minerals including magnesium, sodium, potassium, calcium, zinc, copper, and iron. Based on the presence of active ingredients, multiple scientific studies have shown burdock to have prebiotic (inulin in the root) [9], antioxidant (caffeic acid in the stems, leaves, and rhizodermis) [10], antibacterial (betaeudesmol in the fruit) [11], and anticancer (tannin in the root, and arctin in the leaves, fruit and root) properties. In addition, burdock shows favorable dermatological properties. Phenolic compounds identified in the leaves of the plant are known to improve the condition of skin affected by eczema, psoriasis, acne, or skin rashes [12]. Worthy of mention is a paper published by Fierascu et al. [13], where an extract obtained from burdock plants exhibited important antiradical activity. From a cosmetic point of view, the findings reported by Ahangarpour et al. [14] appear to be relevant as well. The researchers found that hydroxycinnamic acid derivatives contained in burdock were responsible for the plant's anti-inflammatory and antibacterial properties, as well as its protective effects against ultraviolet radiation.

Black radish (*Raphanus sativus* var. *niger*), also called Black Spanish radish or Erfurter radish, is a plant of the family *Brassicaceae*. It has been cultivated in Europe, Asia, and Africa for many years. Black radish has a large tuberous root encased in a black skin, and a white flesh. *Raphanus sativus* var. *niger* plants have thick, coarsely hairy, lyrate-pinnate leaves from which, at later stages in the plant's development, a tall stem extends, topped with purple or white inflorescence. The edible part of the plant is the tuberous root. According to the literature reports [15], black radish is rich in water-soluble vitamins, especially B1, B2, B3, B5, B6, B9, and C, and contains minerals including potassium, magnesium, calcium, iron, manganese, zinc, phosphorus, and fluorine. Moreover, *Raphanus sativus* var. *niger* is a good source of dietary fiber, carbohydrates, and sugars. Also important is the content of volatile substances, including hexyl isothiocyanate (18.4%), 4-methylthiobutyl isothiocyanate (17%), and 4-methylpentyl isothiocyanate (8.4%) [14]. In addition to flavonoids, alkaloids, tannins, and phenolic compounds, black radish contains unique bioactive glucosinolates (glucoraphasatin and glucoraphanin) and isothiocyanates [15]. For some time now, there has been a rise in the popularity of black radish juice, which has been credited with antioxidant [15], antibacterial [15,16], antiviral [15], anti-inflammatory [15–17], anticancer [15–17], antimutagenic [15–19], antidiabetic [15–19], and diuretic properties [15]. It is also used to treat bronchitis, diarrhea, gynecological disorders, and jaundice [16–19].

Today, the cosmetics industry is dominated by multi-component products based on heterophasic systems. Because of their unique properties, emulsions are recognized as the most valuable cosmetic form. Emulsions are systems comprising two immiscible liquids, with one dispersed in the other in the form of droplets, usually with diameters ranging from 0.05 to 0.001 mm [20].

In recent years, a trend has emerged to eliminate silicones from cosmetic formulations, including hair conditioners. For example, Liu [21] studied the effects of various silicone-free conditioners on Mongolian hair damaged by bleaching and compared the results of physicochemical tests with the changes in properties achieved after using a classic silicone-containing formulation. It was found that effective conditioning of damaged hair could be achieved without silicones in advanced cosmetic emulsions based on ingredients such as octyldodecyl myristate or glycerol oleate.

The present study was undertaken to study emulsion-type hair conditioners formulated with the addition of burdock and black radish powders obtained by an innovative method of low-temperature drying with simultaneous micronization (Monochronic Drying

& Powdering, MDP). Monochronic Drying & Powdering is a technology that comprises raw material drying at low temperatures (approx. 40 °C) with simultaneous micronization to ensure, among others, an appropriate level of fragmentation and low water content in the raw material [22]. MDP is very beneficial from a cosmetic point of view because it allows plants to retain many valuable nutrients, such as vitamins or phytosterols, which have a positive effect on the hair and skin.

The aim of this study was to empirically verify the role of burdock and black radish powders in emulsion-type hair conditioners. For the purpose of this study, formulations of model hair conditioners in the form of emulsions were developed. The variables included the type and concentration of plant powders (burdock and black radish) obtained by MDP. For the plant-based material under study, the content of vitamin C and total polyphenolic content, as well as the degree of reduction of DPPH, free radicals were determined. In addition, the finished cosmetic emulsions containing plant powders were evaluated for formulation stability, viscosity, yield point, texture (hardness and adhesive force), and color.

2. Materials and Methods

2.1. Plant Material

Plant material in the form of ready-to-use plant powder was bought from Ecoherba Poland. Plants were cultivated in the Białowieża Forest (Podlaskie Voivodeship, Poland). Powdered plant material was obtained through an innovative technique of low-temperature drying with simultaneous micronization (MDP). Selected plant parts (root tuber: *Raphanus sativus* var. *niger*, leaves: *Arctium lappa*) were used in the drying process to acquire the powder.

2.2. Formulations

Hair conditioner formulations were developed based on the available published data [5,23–32] with slight modifications. A total of seven emulsions were prepared, containing different plant powders at varying concentrations. The quantitative and qualitative compositions of the hair conditioner formulations are shown in Table 1.

Table 1. Qualitative and quantitative composition of hair conditioner prototype formulations.

Phase	Ingredients [According to INCI]	Symbol of Hair Conditioner						
		HC_0	HC_1	HC_2	HC_3	HC_4	HC_5	HC_6
		Concentration [% w/w]						
I	Cetrimonium Chloride				2.0			
	Ceteareth-20				2.5			
	Cetearyl Alcohol				4.0			
	Glyceryl Stearate				1.5			
	Paraffinum Liquidum				2.0			
	Ethylhexyl Hydroxystearate				1.0			
II	Aqua				at 100			
	Glycerin				1.0			
III	Sodium Benzoate and Potassium Sorbate				1.0			
	Citric Acid				to pH = 5.5			
	<i>Arctium lappa</i> Powder	-	0.25	0.5	1.0	-	-	-
	<i>Raphanus sativus</i> var. <i>niger</i> Powder	-	-	-	-	0.25	0.5	1.0

The following materials were used as ingredients in the hair conditioners: Cetearyl Alcohol (BASF, Piekary Śląskie, Poland), Cetrimonium Chloride (BASF Poland), Ceteareth-20 (BASF Poland), Glyceryl Stearate (CRODA, Kraków, Poland), Paraffinum Liquidum (Chempur, Piekary Śląskie, Poland), Ethylhexyl Hydroxystearate (BASF Poland), Sodium

Benzoate and Potassium Sorbate (Pol Nil S.A., Warszawa, Poland), Citric Acid (HSH Chemie, Warszawa, Poland), Glycerin (BASF Poland), *Arctium lappa* Powder (Ecoherba, Hajnówka, Poland), and *Raphanus sativus* var. *Niger* Powder (Ecoherba Poland).

Formulation Method

Each sample of a hair conditioner prototype containing a specific concentration of plant material was prepared separately (Figure 1). The ingredients of the oil phase (I) were weighed in a beaker. Next, the ingredients were mixed on a magnetic stirrer and heated in a water bath to approximately 70 °C until fully dissolved. The same procedure was performed for the hydrophilic components (phase II). The water and oil phases were combined while stirring continuously (2–3 min). The mixture was cooled down by continuous stirring to a temperature of approx. 30 °C and then homogenized. At a temperature of 25 °C, appropriate weighed amounts of plant powder (phase III) were added, and the content was thoroughly mixed. A preservative was added, and the pH regulator was adjusted to 5.5, where necessary. The cosmetic samples were stored for 24 h at room temperature (approximately 22 °C) before performing the tests.



Figure 1. Photo of hair conditioners' prototypes.

2.3. Content of Active Ingredients in Plant Powders

The content of vitamin C in the plant powders was determined according to the Polish Standard PN-A-04019:1998 [33]. The method consists of extracting ascorbic acid from the product with oxalic acid, followed by oxidation to dehydroascorbic acid in an acidic environment using the standard blue indicator dye 2,6-Dichlorophenolindophenol (DCIP). Absorbance measurements were conducted at $\lambda = 500$ nm. The excess volume of the 2,6-Dichlorophenolindophenol solution added to the test sample, corresponding to its absorbance value, was read from the calibration curve. In the next step, the content of vitamin C was calculated in mg per 100 g of the test sample.

The total polyphenol content was determined with the Folin–Ciocalteu method by performing absorbance measurements using the reducing properties of polyphenolic compounds. Absorbance measurements were conducted at $\lambda = 765$ nm. The concentration of gallic acid corresponding to the absorbance of the test sample was read from the calibration curve, and the total polyphenol content was calculated per gallic acid equivalent in mg/100 g of the sample. The test methodology is described in detail in a previous study [34].

The degree of reduction of DPPH free radicals was determined with the method proposed by Yen and Chen, which is based on evaluating the degree of reduction of stable 2,2-diphenyl-1-picrylhydrazyl (DPPH) radicals by antioxidants contained in the plant extracts under study. The DPPH solution is purple in color, but it turns yellow with the progressing reduction of the reagent. The decrease in the absorbance of the test samples, measured at a wavelength of $\lambda = 517$ nm, corresponds to the efficiency of free radical scavenging. The principle of the method is described in depth in a previous study [35].

The ability of the antioxidant studied to prevent the oxidation reaction was calculated using Formula (1):

$$\% \text{ inhibition} = \frac{100 \cdot (A_0 - A)}{A_0} \quad (1)$$

where:

A —mean absorbance of the antioxidant-containing test solution;
 A_0 —mean absorbance of the DPPH radical solution.

2.4. Stability Tests

The stability of the hair conditioner samples was evaluated in a centrifuge test using a ROTOFIX 32A centrifuge from Hettich (Kirchleingern, Germany). The formulation samples were placed in a centrifuge operating at a rotational speed of 2000 rpm for 10 min. The next stage involved a visual evaluation of the formulation's stability. The emulsion prototypes were also subjected to stress temperature tests to visually assess the stability of cosmetics stored alternately at elevated (40 °C, 1 day) and reduced (5 °C, 1 day) temperatures. The tests were performed in an ST-68 type incubator and a refrigerator from Amica (Hongkong, China). The study took 8 days (4 full cycles) to complete.

The results were verified by testing the emulsions studied using a Turbiscan Lab Cooler from Formulacion (L'Union, France). Transmittance (T) and backscatter (BS) correlation profiles, as well as the Turbiscan Stability Index (TSI), were obtained [22]. Both parameters were calculated using Formulas (2)–(4):

$$BS \approx 1/\sqrt{\lambda^*} \quad (2)$$

$$\lambda^*(\phi, d) = \frac{2d}{3\phi(1-g)Q_s} \quad (3)$$

$$TSI = \sqrt{\frac{\sum_{i=1}^n (X_i - X_{BS})^2}{n-1}} \quad (4)$$

where:

λ^* —is the photon transport mean free path in the analyzed dispersion;
 ϕ —is the volume fraction of emulsion particles;
 d —is the mean diameter of particles;
 g, Q_s —are the optical parameters given by the Mie theory;
 X_i —is the average backscattering for each minute of measurement;
 X_{BS} —is the average X_i ;
 n —is the number of scans [29].

2.5. Dynamic Viscosity

Viscosity is a parameter determining whether cosmetic products are easy to dispense and spread on the skin. Dynamic viscosity was measured using a Brookfield DV-I+ viscosity meter fitted with an appropriately sized spindle. Measurements were taken at 10 rpm at room temperature (approximately 22 °C). Three measurements were performed, and the results were averaged. The research methodology is described in a previous study [22].

2.6. Yield Stress

The values of yield stress in the hair conditioner formulations were measured with a HADV III Ultra viscometer from Brookfield Engineering Laboratories (Middleboro, MA, USA), equipped with a set of vane spindles [22]. The measurements were recorded in each second of the test progressing at a constant rotational speed of the spindle (1 rpm). The yield stress corresponded to the maximum value of shear stress, which, when exceeded, induced product fluidization. The measurement results were recorded and processed using EZ-Yield Software version 1.2. Measurements were taken at room temperature, i.e., approximately 22 °C. A total of five measurements were conducted, and their results were averaged.

2.7. Mechanical Properties (Texture)

The consistency of the test samples was determined using a Brookfield CT3 texture analyzer (Key Industries, Toronto, ON, Canada). The test was performed by immersing a steel spherical probe (TA18) to a depth of 10 mm into the sample. The test duration was 200 s for each sample. Three measurements were taken for each sample, following which the arithmetic mean was calculated. The results were processed by TexturePro CT software version 1.4. The test methodology is described in detail in the literature [10,22].

2.8. Color Assessment

Color tests were performed using a CR 400 colorimeter from Konica Minolta (Tokyo, Japan). 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 [22,36,37]:

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.

Next, on the basis of the data obtained, differences in color saturation (ΔC_{HC}) were calculated using Formula (5):

$$\Delta C_{HC_0-HC_X}^* = C_{HC_0}^* - C_{HC_X}^* \quad (5)$$

where:

$\Delta C_{HC_0-HC_X}^*$ —difference in color saturation between the base hair conditioner formulation and the hair conditioner containing plant powder;

HC₀—base hair conditioner formulation;

HC_X—hair conditioner with plant powder at a concentration of X;

X = 0.25; 0.5, 1.0.

The color saturation of the base hair conditioner and the hair conditioner containing plant powder was calculated using Formulas (6) and (7):

$$C_{HC_0}^* = \sqrt{\left(a_{HC_0}^*\right)^2 + \left(b_{HC_0}^*\right)^2} \quad (6)$$

$$C_{HC_X}^* = \sqrt{\left(a_{HC_X}^*\right)^2 + \left(b_{HC_X}^*\right)^2} \quad (7)$$

A total of five measurements were carried out, and their results were averaged.

2.9. Statistical Analysis

The points in the charts represent mean values from a series of three or five independent measurements. The Student's 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. Standard deviations are presented in the figures.

3. Results and Discussion

3.1. Content of Active Ingredients in Plant Powders

Plants have been used as ingredients in cosmetics, medicines, and food products for a great number of years. The interest in plant-based products can be attributed to the fact that plants contain a wealth of valuable chemical compounds with antioxidant, anti-inflammatory, antimicrobial or preservative properties that can be used to good effect in various products. For practical reasons, it is desirable to formulate cosmetics based on

raw materials exhibiting antioxidant properties, such as polyphenols [38,39]. An alternative indicator of antioxidant capacity is oxidative activity (measured degree of DPPH free radical reduction).

Another cosmetically relevant active ingredient is vitamin C. In addition to other benefits, it is claimed to contribute to the sealing of blood vessel walls or the relief of skin redness, accelerate the healing processes, and have mild exfoliating properties [40,41].

Table 2 summarizes the results of the measurements of the polyphenol content, oxidative activity, and vitamin C content in black radish and burdock powders obtained by low-temperature drying with simultaneous micronization.

Table 2. Results of measurements of total polyphenolic content, oxidative activity, and vitamin C content.

Plant Powder	Total Polyphenolic Content [mg GAE/100 g DM]		Oxidative Activity [% Inhibition]		Vitamin C Content [mg/100 g DM]	
	Mean Value	SD	Mean Value	SD	Mean Value	SD
Black radish	15.46	1.23	13.83	0.57	266.34	0.000
Burdock	41.52	1.25	68.75	2.91	4.14	0.447

As the results in Table 2 show, the total polyphenol content in black radish powder was 15.41 [mg GAE/100 g DM], while the degree of DPPH free radical reduction equaled 13.83%. In burdock powder, the total polyphenol content was 41.52 [mg GAE/100 g DM], and the degree of DPPH free radical reduction was 68.75%. Moreover, the content of vitamin C in black radish powder was found to be 266.34 [mg/100 g DM]. In contrast, the content of vitamin C in burdock powder obtained by low-temperature drying with simultaneous micronization was 4.14 [mg/100 g DM]. The results of studies on burdock and black turnip obtained by other researchers are discussed below.

Enkhtuya et al. [42] evaluated the content of polyphenolic compounds in extracts obtained from dried black radish root. The plant-derived material was dried at 40, 50, 60, and 70 °C. The following extracts were obtained: 99.5% ethanol extracts, 50% ethanol extracts, and water extracts. Based on the experiments, the authors found the most favorable content of polyphenols in all the extracts obtained from plant material dried at 70 °C, totaling 165 [mg GAE/100 g DM] for the 99.5% ethanol extract, 790 [mg GAE/100 g DM] for the 99.5% ethanol extract, and 892 [mg GAE/100 g DM] for the aqueous extract. Hanlon et al. [43] reports the results of quantification of total polyphenol content with the Folin–Ciocalteu method. The authors used frozen plant material, which was then freeze-dried. The total content of polyphenols in black radish pulp was 1794 [mg GAE/100 g DM]. Lugasi et al. [18] conducted a study to assess the antioxidant properties of black radish juice using the method proposed by Hatano et al. The black radish juice was made under industrial manufacturing conditions and stored at a temperature of 18 °C until chemical analysis. Based on the measurements, the antioxidant activity of black radish juice varied between 7.3% (for the juice concentration of 0.05 wt%) and 88.1% (for the juice concentration of 1 wt%). In their study, Enkhtuyai and Tsend [15] carried out experiments for dried peeled and unpeeled root of black radish, and juice extracted from the peeled and unpeeled root of black radish. The test results showed the highest degree of free radical reduction in the juice extracted from the peeled black radish root (61.8%), and the lowest degree of free radical reduction (21.9%) in the juice extracted from the unpeeled root of *Raphanus sativus* var. *niger*. In their study of the active ingredients present in the aqueous extracts of black radish, Hanlon et al. [43] showed a vitamin C content of 728 [mg/100 g DM]. This value is significantly higher than that obtained in the present study.

Zhang et al. [10] studied the content of polyphenols in unpeeled burdock root powder (BRP), peeled burdock root powder (PBRP), and burdock root peel powder (BRPP). The experiments revealed that the total polyphenol content varied depending on the part of the plant used for obtaining the powder: 15,131 [mg GAE/100 g DM] for BRP; 12,753 [mg

GAE/100 g DM] for PBRP; and 72,214 [mg GAE/100 g DM] for BRPP. The results show that the highest content of polyphenol compounds was present in the root peel of *Arctium lappa*, while the lowest value of the parameter was measured in peeled burdock root. Moreover, Ariádine Reder Custódio de Souza et al. [44] found that the oxidative activity of the burdock extracts obtained in their study ranged from 12.61 to 46.41%, which is nearly six times lower than the oxidative activity determined in the study reported here.

The content of vitamin C in burdock powder obtained by low-temperature drying with simultaneous micronization reported in our work is not high compared to the literature-reported data. For example, the study conducted by Zhang et al. [10] to determine the content of vitamin C in whole burdock root powder (BRP), peeled burdock root powder (PBRP), and burdock root peel powder (BRPP) found a high content of this active ingredient in the examined burdock material, ranging from 149 to 1231 [mg/100 g DM].

The results obtained are significantly influenced by factors including the form of the black radish plant material (dried, frozen, fresh), whether the plant or extract was tested, and the specific part of the plant studied. It is evident from the literature data that the highest content of active ingredients is typically obtained from fresh black radish plant material. Regarding burdock, discrepancies between the study's results and those in the literature could be attributed to factors such as the plant's country of origin, cultivation location, and other variables.

3.2. Stability Tests

Formulation stability is an absolute prerequisite in cosmetics, especially in emulsion-type products. Centrifuge and temperature test results show that the stability of cosmetic formulations is not adversely affected by the addition of the plant powders evaluated in this study. No change in sample appearance and no layer separation were observed after the tests.

Klimaszewska et al. [22] formulated stable emulsion-type facial masks, incorporating haskap berry (honeyberry) powder as one of the ingredients. The samples retained their stability after undergoing centrifugation (2000 rpm) and storage in a refrigerator and incubator at similar temperatures for a period of 30 days.

Tafuro et al. [45] studied the effects of adding Zea Mays Starch and Zinc Oxide at different proportions to the formulations of cosmetic emulsions containing a sugar derivative as a rheology modifier. Centrifuge tests conducted at a set speed of 3000 rpm for 30 min revealed a positive stability result for a sample containing only the additives at a 1:1 ratio.

A Turbiscan device makes it possible to detect signs of emulsion instability already at a very early stage of storage. Furthermore, it allows for the identification of the type of instability (coalescence, flocculation, creaming, etc.). The visual evaluation of the formulated samples was verified on the basis of transmittance (T) and backscattering (BS) correlation profiles. The profiles obtained for the formulated hair conditioners are shown in Figure 2, Figures A1–A6 (Appendix A).

Based on the determined profiles (Figure 2, Appendix A: Figures A1–A6), both the baseline emulsion and the hair conditioners formulated with the addition of black radish and burdock plant powders were found to be stable. Since the emulsions were very viscous, it was difficult to transfer them to the measuring vessels. During the transfer, the emulsions became aerated. Then, as a result of the temperature, they became deaerated during storage. Hence, there are slight deviations in graph slope, which, however, are not significant.

In the studies conducted by Wasilewski et al. [46], cosmetic emulsions were formulated with solid particles, including ground oat grain and ground orange peel. In general, the emulsion samples were characterized by a stable form (as determined by centrifuge and temperature tests). After a 60-day Turbiscan test, only minor signs of emulsion destabilization were seen, including sedimentation presumably caused by the settling of ground plant particles. The phenomenon was notable especially for the fragments of orange peel.

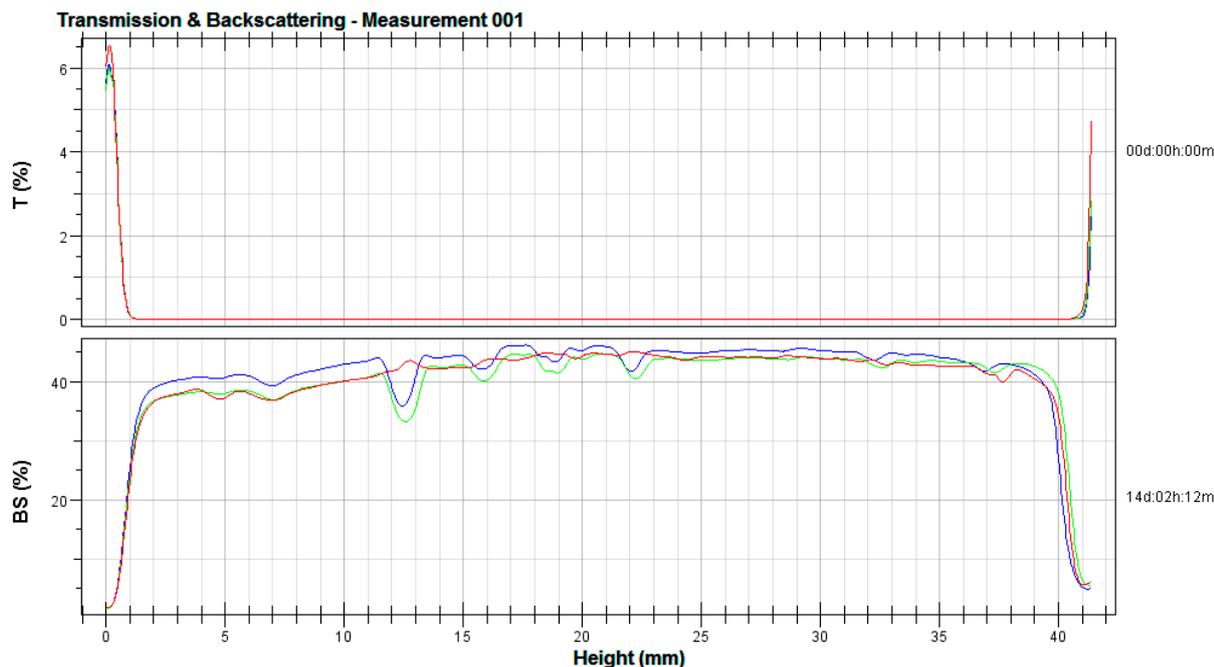


Figure 2. Dependence of transmittance (T) and back scattering (BS) on sample height for a prototype HC_0; dark blue line—evaluation of the preparation immediately after application into tubes (time “0”), green line—stability after 7 days, red line—stability after 14 days.

Jeon and Hong [47] conducted studies evaluating emulsion stabilization using a specific type of clay (montmorillonite) and applying different methodologies—visual and rheological—for stability assessment. The authors found that in a mixture of hydrophilic and hydrophobic clays, the emulsion exhibited synergistic stabilization due to the formation of an interfacial structure by the clay particles. Kowalska et al. [48] evaluated the stability of emulsions stored at 25 °C during a 30-day test. The emulsions were formulated with the addition of watermelon seed oil and orange fibers. The authors identified a range of phenomena, including creaming, coalescence, and flocculation in the test samples at different stages of storage. The longest period of stability was recorded for the sample containing orange fibers (0.5 wt%) and watermelon seed oil (6.12 wt%).

Another measure of emulsion stability is the Turbiscan Stability Index (TSI), which takes values from 0 to 100. The higher the TSI value, the greater the degree of emulsion destabilization. The TSI values obtained for the hair conditioner formulations are shown in Figure 3.

The TSI values (Figure 3) determined for the formulated hair conditioners vary in the ranges of 0.95 to 2.4 and 1.25 to 3.2 at one and two weeks after preparation, respectively. A review of the study data shows that the cosmetic without added plant powders (HC_0) had a TSI value of 1.95 after one week of storage at 38 °C. The TSI rose to 2.95 after two weeks. The highest TSI values were recorded for the conditioner containing burdock powder at a concentration of 0.5% (HC_1): $TSI_{7\text{ days}} = 2.4$ and $TSI_{14\text{ days}} = 3.2$. Nevertheless, these TSI values are still low, indicating that the emulsion has a relatively high stability, and the differences are not significant.

Based on the literature on the subject, plant-derived raw materials can impact the stability of the cosmetic form in a variety of ways. For example, Nizioł-Lukaszewska et al. [49] conducted a study to evaluate the application of dogwood fruit extract in cosmetic emulsions stored at 40 °C for a period of 14 days. The TSI values measured with a Turbiscan device were at the level of approx. 2.4 after seven days of storage and approx. 3.3 after 14 days of storage. They were thus close to the values obtained in the present study.

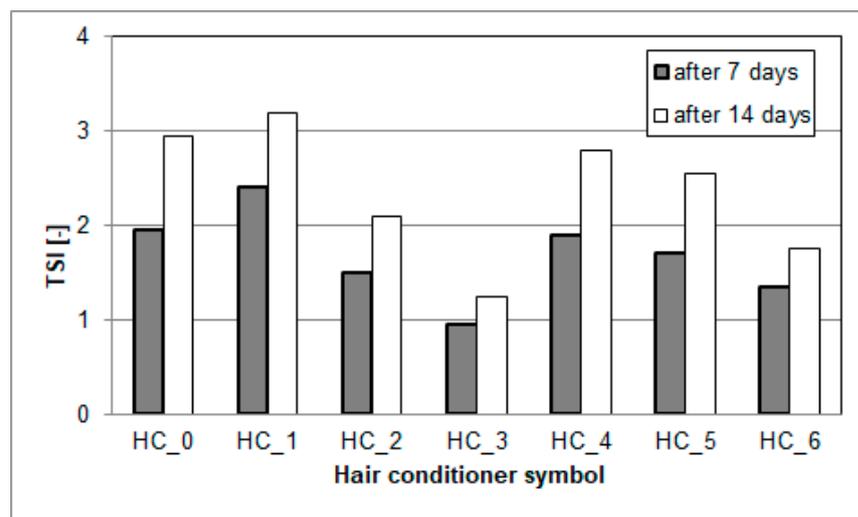


Figure 3. Turbiscan stability index (TSI) values of analyzed hair conditioners after 7 and 14 days.

Moreover, Wasilewski et al. [46] studied the effects of adding ground orange peel (*Citrus aurantium dulcis* Peel Powder) and oat bran (*Avena Sativa* Bran) to cosmetic emulsions on their properties. Based on the measurements of samples stored at 40 °C, carried out for 60 days, the TSI values varied around and above 8.

3.3. Dynamic Viscosity

The dynamic viscosity (η) of hair conditioners is an important determinant of cosmetic quality. It has an impact on the functional properties of cosmetics, i.e., ease of dispensing from the package or spreadability on hair. Consumers commonly assert that high viscosity is an indication of high content of active ingredients in hair conditioners. However, the dynamic viscosity of cosmetics is determined not only by the active ingredients but also, and primarily, by the presence of viscosity regulators and emulsifiers (surfactants).

The dynamic viscosity (η) of the hair conditioners was found to range from 30,120 to 41,110 mPa·s (Figure 4). Both the type and concentration of burdock and black radish powders were shown to affect the η values of the hair conditioners under study. Additionally, burdock powder incorporated into the hair conditioner formulations led to an increase in dynamic viscosity of approximately 2 to 26% compared to the reference formulation. Dynamic viscosity was found to rise in proportion to increasing concentrations of burdock powder. In this series of emulsions, the highest level of dynamic viscosity (38,066 mPa·s) was recorded for the conditioner containing burdock powder at a concentration of 1%. An addition of black radish powder to the hair conditioners led to slightly higher η values relative to the results obtained for the formulations with burdock powder. The test results ranged between 38,600 and 41,110 mPa·s. Similarly to the conditioners containing burdock powder, the higher the concentration of black radish powder, the higher the dynamic viscosity.

The results of dynamic viscosity measurements obtained for emulsion-type hair conditioners reported in the literature encompass a wide range. Similarly to other cosmetic emulsions [50], hair conditioners are classified as pseudoplastic non-Newtonian liquids (product viscosity decreases with increasing shear rate). According to other authors [48] the dynamic viscosity of the cosmetic emulsions they studied varied from approximately 8 to 26,000 mPa·s. For example, the viscosity of the cosmetic emulsions evaluated by Klimaszewska et al. [51] was approximately 25,000 mPa·s. The values of dynamic viscosity measured in the hair conditioners containing plant-derived powders show an order of magnitude consistent with the results reported in the literature.

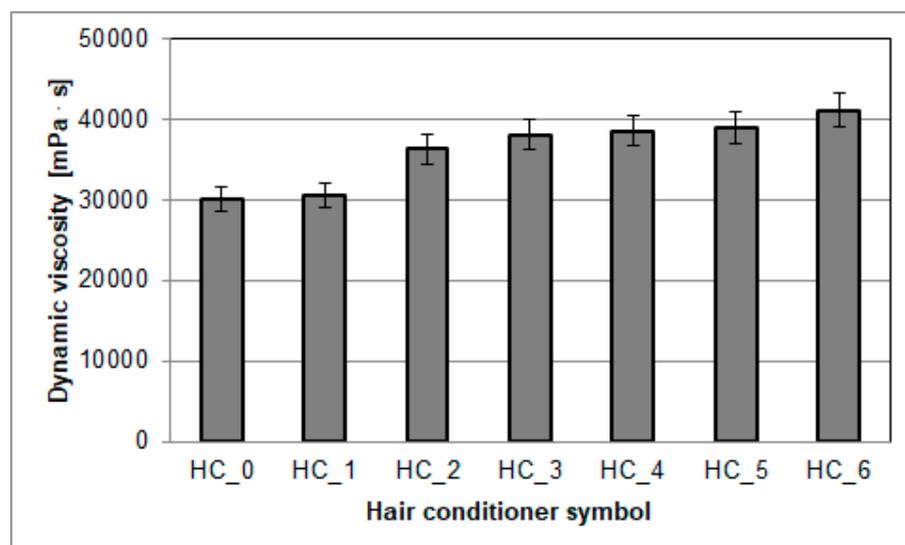


Figure 4. Dynamic viscosity of analyzed hair conditioners.

The rise in viscosity as a function of the increasing concentration of both plant powders under study can be explained by the fact that when solid particles in the form of a powder are incorporated, the viscosity of the continuous phase of the emulsion also rises (the plant powder acts as a “filler” for the continuous phase), thus increasing its dynamic viscosity and the stability of the cosmetic emulsion.

3.4. Yield Stress

The rheological properties of emulsion-type hair conditioners have a significant influence on their functionality. One of the important rheological parameters in emulsions is yield stress, which is the lowest shear-stress value at which a material will behave like a fluid. Generally, it is recognized that the lower the yield stress, the “lighter” the consistency of a cosmetic product and the more easily it spreads on the skin and hair [52]. From the point of view of consumers purchasing hair conditioners, the value of the parameter should not be too low, as this would result in the product running off the hair during application, or too high, which would make it difficult to apply the cosmetic on the hair. Yield stress results are also important for the manufacturers of hair conditioners. Determining the optimum yield stress value for different types of cosmetics helps select an appropriate packaging type and method of dispensing the product from the package. The effects of yield stress on the functional characteristics of emulsion-type cosmetic products have been reported in the literature [53]. For example, in their study of cosmetic creams, Savić et al. [48] found that the formulation with the highest yield stress had the best structure and stability of the emulsion system. The results of the yield stress measurements of the hair conditioners under study are shown in Figure 5.

The yield stress test results (Figure 5) obtained for the evaluated hair conditioners correlate significantly with the results of dynamic viscosity measurements (Figure 5). Again, the type (burdock, black radish) and concentration (0.25; 0.5 and 1.0 wt%) of the plant-derived powders were found to have an impact on the yield stress of the emulsions. There was a clear tendency for an increase in yield stress with rising concentrations of plant-based powders, both derived from burdock and black radish. Incorporating burdock powder (0.25 wt%) into the formulations of hair conditioners caused an increase in yield stress relative to the reference formulation (with no added powder: HC_0) by as much as 43%. For black radish powder, the increase reached 25%. Hair conditioners containing 1.0 wt% of burdock powder (HC_3) and black radish powder (HC_6) were characterized by the highest yield stress of 295 Pa and 280 Pa, respectively. Also, the addition of burdock powder to the hair conditioners led to a slightly higher value of yield stress compared to

the results noted for the formulations containing black radish powder. Lower values of the test parameter obtained for cosmetic emulsions translate into a lighter consistency and potentially better product spreadability on the skin. Similar studies were also conducted by Klimaszewska et al. [22], who investigated facial masks containing *Lonicera caerulea* Fruit Powder at various concentrations. The authors found that the formulation containing 0.9 wt% of blue honeysuckle powder, which had the lowest yield stress, was characterized by the best spreadability on the skin of all the facial masks tested. Also, in their study, Lukic et al. [50] found a similar correlation between the yield stress results and viscosity results of the studied emulsions to the formulated hair conditioners. The highest yield stress was shown in the most viscous sample, with values declining in proportion to decreases in emulsion viscosity induced—in this case—by a modification in the oil phase.

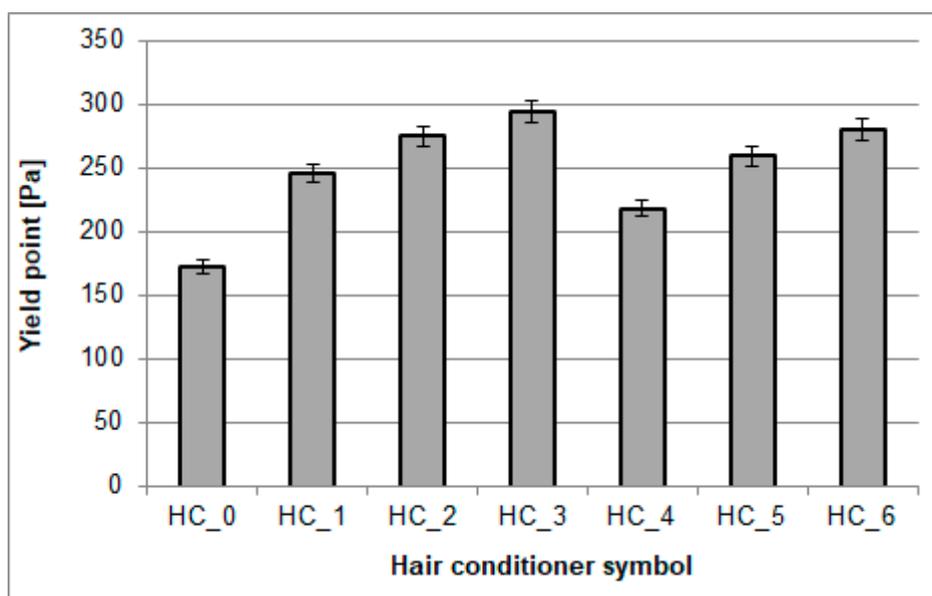


Figure 5. Yield point of analyzed hair conditioners.

Summing up, the results of yield stress tests reveal that the proposed plant-derived powders added to hair conditioners affect the ease of dispensing products from the package and improve spreadability on the hair surface at concentrations as low as 0.25 wt%. Furthermore, incorporating plant-based powders into hair conditioners may potentially contribute to reducing the content of emulsifiers or rheology modifiers in such formulations.

3.5. Mechanical Properties of Emulsions (Texture)

Texture analysis is a method very commonly used in the cosmetics, pharmaceutical, and food industries. Texture analysis relies, among others, on the mechanical techniques for performing quantitative measurements of product characteristics. Mechanical tests cause deformation of the tested surface of the product and define its selected parameters, [54,55] including hardness (maximum force at deformation), and adhesion or cohesion (maximum negative force recorded at product deformation).

The values of hardness and adhesive force obtained for the analyzed hair conditioners are shown in Figure 6.

For the reference hair conditioner (HC_0), the measured value of hardness was 8.5 g (Figure 6). For burdock powder, hardness varied within the range of 10.2 ÷ 12.45 g, while for the conditioners with added black radish powder, the parameter varied from 11.76 g to 14.8 g. Thus, the addition of burdock powder and black radish powder into the emulsions led to an increase in the measured parameter by up to nearly 35% compared to the emulsions without any added plant-derived powders.

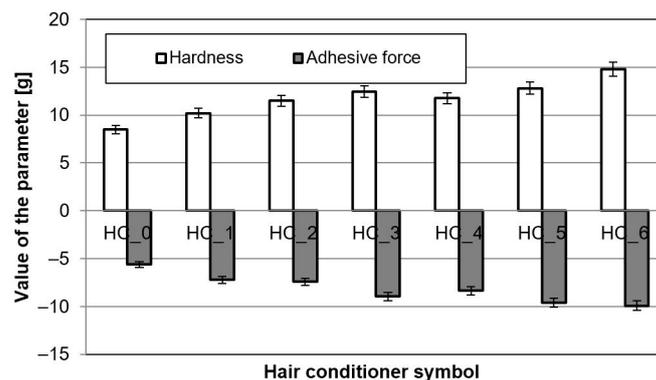


Figure 6. Hardness and adhesive force of analyzed hair conditioners.

A similar tendency was observed for adhesion (Figure 6). Within a given series of conditioners containing a plant-derived powder, the adhesive force increased, reaching a value that was nearly 50% higher (-9.9 g for HC_6) compared to the adhesive force measured for the baseline emulsion (-5.6 g for HC_0).

From the viewpoint of application properties, the values of hardness and adhesive force are thus advantageous, especially in terms of ease of dispensing a cosmetic product from its package. Since hair conditioners are usually packed in squeezable tubes or jars, these results indicate that the formulated hair conditioners would be easy to dispense from the packaging. Furthermore, the values of adhesive force recorded during the experiment show that users would have no difficulty spreading the product on their hair.

Kowalska et al. [48] evaluated the texture of emulsions containing, among other ingredients, xanthan gum and orange fibers as a function of the concentration of these additions in the emulsion formulation, and as a function of time. The authors proved that the hardness of the samples declined as a function of the decreasing percentage content of xanthan gum in the formulation, and rose as a function of the increasing orange fiber concentration to levels as high as 105 g. An analysis of the results of adhesive force measured in the prepared samples revealed an analogous trend. The values of adhesive force were as high as -77 g, and increased with time. Gilbert et al. [56] evaluated the effects of natural and synthetic polymers on the texture of formulated cosmetic emulsions, among other factors. The polysaccharides (e.g., carob, xanthan) used by the authors in the cosmetic emulsion formulations were shown to affect the values of emulsion hardness, which varied from 4 g to 6 g. The adhesive force measured in the studied creams was found to be in the range of -1.5 – 2.5 g.

3.6. Color Assessment

Chemically, plants are rich in compounds that can be used as coloring agents in the food or cosmetic industries, among an array of other applications [57,58]. The present study included an assessment of the effect of the concentration of burdock and black radish powders on changes in the color of the cosmetic emulsions formulated with the addition of these ingredients. The results of the assessment are listed in Table 3.

The addition of plant powders to the cosmetic emulsions was found to induce changes in all measured parameters (Table 3). The highest value of the L^* parameter was recorded for the baseline sample HC_0 (71.54). Even at the lowest proposed concentration ($c = 0.25$ wt%) of the plant powders, a decrease in the measured parameter was noted, and the downward trend for the L^* value was maintained with the increasing percentage content of both plant-derived powders. The lowest degree of whiteness was observed in the sample containing 1% of the *Raphanus sativus* var. *niger* powder ($L^* = 56.84$). The value was 20% lower compared to an emulsion without any plant powder.

Table 3. Color parameters of analyzed hair conditioners.

Hair Conditioner Symbol	Parameter			
	L*	a*	b*	ΔC^*_{HC}
HC_0	71.54	−1.54	−1.62	-
HC_1	70.51	−1.52	2.26	0.63
HC_2	67.16	−1.47	3.38	1.49
HC_3	66.71	−1.39	5.37	3.31
HC_4	66.63	−0.48	4.01	1.80
HC_5	63.12	0.07	4.79	2.55
HC_6	56.84	0.57	8.67	6.45

The parameter a^* was altered by the incorporation of plant-derived materials into the formulation as well. For the baseline emulsion, $a^*_{HC_0}$ was equal to -1.54 . In the hair conditioners containing *Arctium lappa* and the *Raphanus sativus* var. *niger* powders, the values of a^* ranged from -1.52 to -1.39 and -0.48 to 0.57 , respectively. Hence, as the percentage content of the powders in the formulation rose, the intensity of the green color in the emulsions decreased and that of the red color increased.

The b^* value determined for the reference emulsion (HC_0) equaled -1.62 . On the other hand, in the cosmetics containing burdock powder and black radish powder, the values of the b^* parameter were positive. Notably, however, the higher the concentration of the powder in the formulation, the greater the measured value. The highest value of b^* (8.67) was obtained for a cosmetic product containing black radish powder (HC_6). In practical terms, these test results mean that an increase in the concentration of the plant-derived powders contributes to an increase in the intensity of the yellow color of the samples.

The tests (Table 3) showed changes in the saturation of the color (ΔC^*_{HC}) of the formulated hair conditioners. The differences in ΔC^* varied depending on both the type of the plant-based powder used and its concentrations in the formulation. The difference in color saturation in the conditioners containing *Arctium lappa* powder was 0.63 in the formulation containing the ingredient at a concentration of 0.25 wt% and increased over five times ($\Delta C^*_{HC_3} = 3.31$) in the conditioner with added powder at a concentration of 1 wt%. A similar change trend was noted for the cosmetics formulated with the addition of the *Raphanus sativus* var. *niger* powder, though the intensity of the changes was greater. Color saturation in the samples containing black radish varied from 1.80 to as much as 6.45 . To recapitulate, a rise in the concentration of burdock and black radish powders obtained by low-temperature drying in hair conditioners resulted in a more saturated color compared to the reference sample.

Bujak et al. [59,60] assessed the effects of adding aqueous–ethanol extracts obtained from six different plants to cosmetic emulsions, including the impact on their color. The authors noted a significant effect of the extracts on the L^* , a^* , and b^* values, and on the values of ΔC . The greatest impact on the measured parameters was noted for the *C. ternatea* L. extract. The application of that extract imparted a blue-purple color on the sample and affected the values of $L^* = 68.19$, $a^* = 6.23$, and $b^* = 18.92$. The value of the difference (ΔC^*) reached 24.2 .

Nizioł-Lukaszewska et al. [49] also evaluated the effects of incorporating a Cornus MasL. extract into cosmetic emulsions. The authors showed that the addition of dogwood extract to the emulsion caused no significant changes in sample brightness (from $L^* = 77.62$ to $L^* = 76.54$) or the b^* value (from $b^* = 77.62$ to $b^* = 76.84$). In turn, the a^* value changed from $a^* = -0.52$ to $a^* = 0.94$ after extract addition.

4. Conclusions

Based on this study's results, it can be stated that burdock and black radish powders obtained by low-temperature drying contain polyphenolic compounds and exhibit antioxidant activity, which is especially prominent in burdock powder. The two plant-

derived materials studied also contain vitamin C; however, the content of the vitamin is not high in either of the ingredients. The hair conditioner formulations developed for the purpose of this study allow us to obtain stable emulsions. The stability of the hair conditioner prototypes was verified by performing centrifuge tests and thermal tests, as well as analyzing TSI values and transmittance (T) and backscattering (BS) correlation profiles. The dynamic viscosity of the hair conditioners was found to range from over 30,000 to over 40,000 mPa·s. This study showed that the measured parameter rose as a function of the increasing percentage content of plant-derived additives in the formulation. Moreover, the value of yield stress determined in the model emulsions rose along with increasing concentrations of burdock and black radish powders in the conditioner. The yield stress measured in the formulations varied from 173 to 295 Pa, which should ensure easy dispensing of the emulsion from the package and good spreadability on the hair surface. Additionally, the hardness of the formulations under study was found to increase with rising concentrations of the *Arctium lappa* powder and the *Raphanus sativus* var. *niger* powder in the conditioners. The values of the parameter were in the range of 8.5 to 14.8 g. The adhesive force was estimated to vary from −5.6 to −9.9 g. These results correlate with those obtained in the dynamic viscosity and yield stress tests. The results of viscosity, hardness, and yield stress tests indicate that the incorporation of burdock and black radish powders into emulsion-type hair conditioners may potentially contribute to reducing the content of emulsifiers or rheology modifiers in such formulations. Also, the type of plant-based powder triggered a change in the saturation of the color of the emulsion. An increase in the concentration of the plant-derived powders contributed to an increase in the intensity of the yellow and red color of the samples.

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Conflicts of Interest: The authors declare no conflicts of interest.

Appendix A

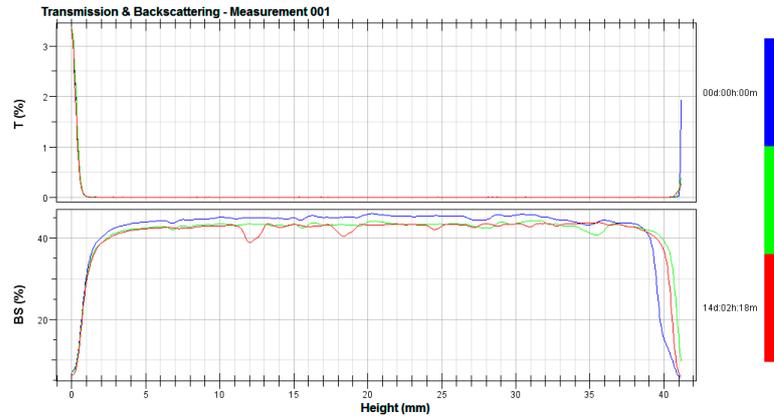


Figure A1. Dependence of transmittance (T) and back scattering (BS) on sample height for a prototype HC_1; dark blue line—evaluation of the preparation immediately after application into tubes (time “0”), green line—stability after 7 days, red line—stability after 14 days.

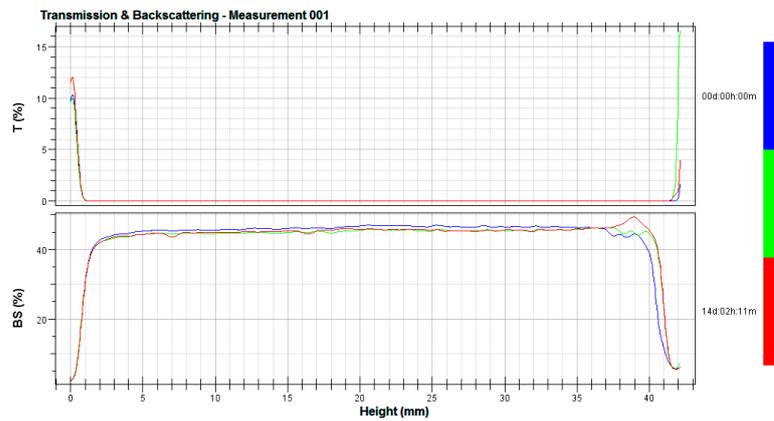


Figure A2. Dependence of transmittance (T) and back scattering (BS) on sample height for a prototype HC_2; dark blue line—evaluation of the preparation immediately after application into tubes (time “0”), green line—stability after 7 days, red line—stability after 14 days.

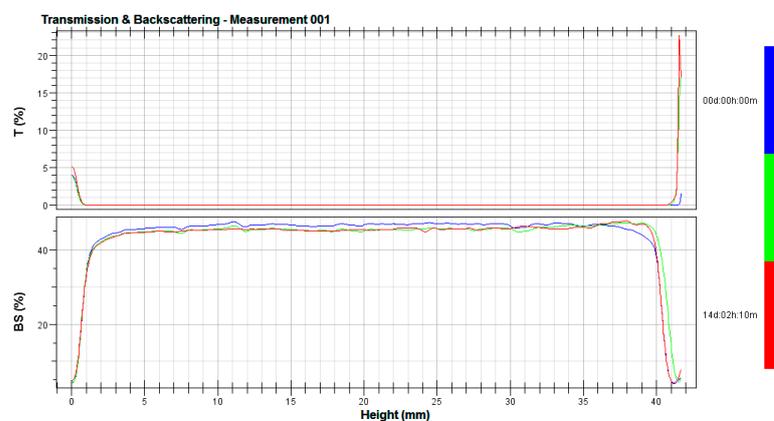


Figure A3. Dependence of transmittance (T) and back scattering (BS) on sample height for a prototype HC_3; dark blue line—evaluation of the preparation immediately after application into tubes (time “0”), green line—stability after 7 days, red line—stability after 14 days.

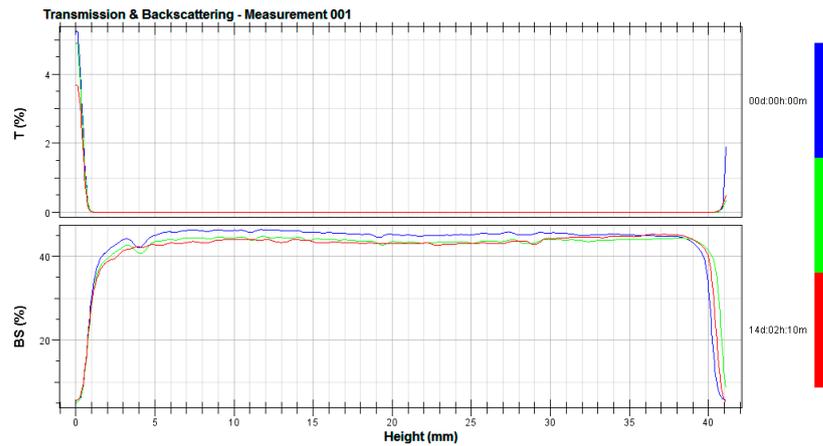


Figure A4. Dependence of transmittance (T) and back scattering (BS) on sample height for a prototype HC_4; dark blue line—evaluation of the preparation immediately after application into tubes (time “0”), green line—stability after 7 days, red line—stability after 14 days.

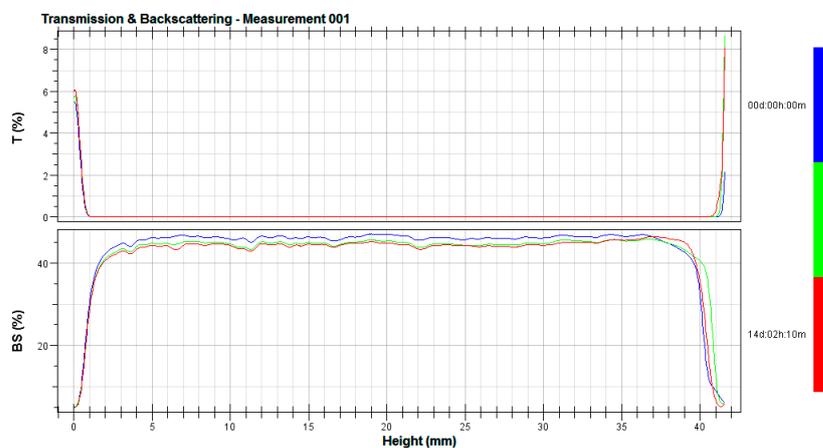


Figure A5. Dependence of transmittance (T) and back scattering (BS) on sample height for a prototype HC_5; dark blue line—evaluation of the preparation immediately after application into tubes (time “0”), green line—stability after 7 days, red line—stability after 14 days.

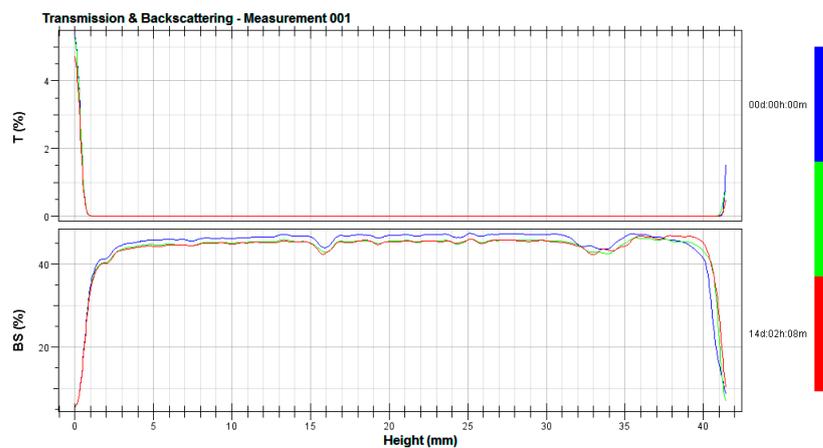


Figure A6. Dependence of transmittance (T) and back scattering (BS) on sample height for a prototype HC_6; dark blue line—evaluation of the preparation immediately after application into tubes (time “0”), green line—stability after 7 days, red line—stability after 14 days.

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