

## APPLICATION OF *ARONIA MELANOCARPA* FRUIT POWDER OBTAINED BY AN INNOVATIVE LOW-TEMPERATURE DRYING METHOD FOR FACIAL CARE MASKS

Emilia Klimaszewska<sup>1\*</sup>, Marta Ogorzałek<sup>2</sup>, Natalia Korolska<sup>3</sup>,  
Ryszard Tomasiuk<sup>4</sup>

<sup>1,2,3</sup> Casimir Pulaski University of Radom, Chrobrego 27, 26-600 Radom, Poland,  
Department of Cosmetology, Faculty of Medical Sciences and Health Sciences

<sup>4</sup> Casimir Pulaski University of Radom, Chrobrego 27, 26-600 Radom, Poland,  
Department of Basic Medical Sciences, Faculty of Medical Sciences and Health Sciences,  
ORCID 0000-0003-1570-5196, e-mail: r.tomasiuk@urad.edu.pl

<sup>1</sup> ORCID 0000-0002-5621-5911, e-mail: e.klimaszewska@urad.edu.pl

<sup>2</sup> ORCID 0000-0002-1896-1876, e-mail: m.ogorzalek@urad.edu.pl

<sup>3</sup> Student, e-mail: nataliakorolsk98@gmail.com

\* Corresponding author

**Abstract:** The purpose of the study was to develop recipes and create skin care masks based on *Aronia Melanocarpa* Fruit Powder, obtained by an innovative low-temperature drying method, and to evaluate the physicochemical and functional properties of such products. It was noted that as the concentration of fruit powder in the tested formulations increased, the value of dynamic viscosity increased, while the yield point decreased. It was found that an increase in the concentration of plant powder in the formulation caused a change in the saturation and hue of the analysed masks towards red and yellow. After application of the developed masks to the skin, TEWL decreased, with the lowest value recorded for the mask with the highest concentration (0.5%) of *Aronia Melanocarpa* Fruit Powder.

**Keywords:** skin care cosmetics, low temperature drying, chokeberry, TEWL, physicochemical properties.

### 1. INTRODUCTION

Chokeberry (*Aronia*) was already known in ancient folk culture as a medicinal substance [Svarc et al. 2019]. According to the literature, it originates from, and it is even stated that its "homeland", is North America [Wawer, Eggert and Hołub 2015; Doroszko, Janda and Jakubczyk 2018]. The fruit belongs to the family Rosaceae and subfamily Maloideae [Wilczyński et al. 2017; Doroszko, Janda and Jakubczyk 2018; Sadowska et al. 2019].

In Poland, the first seedlings appeared in 1976, and work on the cultivation of this fruit was initiated by Professor Wiesław Grochowski. Experimental work in Kraśnik and near Janów Lubelski was conducted by Maria Cichowicz, M.Sc. and Piotr Eggert, M.Sc. [Wawer, Eggert and Hołub 2015]. Chokeberry fruits are rich in vitamins, such as C, which is mainly found in this fruit, as well as E, B1, B2, B3, B5, B6, and K [Wilczyński et al. 2017; Doroszko, Janda and Jakubczyk 2018]. They also contain minerals, among which are molybdenum, manganese, copper, boron, iodine, and cobalt [Doroszko, Janda and Jakubczyk 2018]. They contain many active substances, such as polyphenols, which include anthocyanins, tannins, phenolic acids and flavonoids [Tao et al. 2017; Wilczyński et al. 2017; Sadowska et al. 2019].

According to sources [Wawer, Eggert and Hołub 2015; Wilczyński et al. 2017; Doroszko, Janda and Jakubczyk 2018], the dry weight of chokeberry fruit is about 17.0–29.0%, and has the following approximate chemical composition:

- water: 74.0–83.0%;
- sugars, i.e. glucose and fructose: 6.2–10.8%;
- fat: 0.14%;
- protein: 0.7%;
- fibre: 5.62%;
- acids, which are given in terms of malic acid: 0.7–1.3%.

Due to its rich properties, chokeberry has found use not only in the food and pharmaceutical industries, but also in the cosmetics industry. Through its antioxidant activity, absorbing harmful UV radiation as well as removing free radicals, this fruit strongly supports the fight against poor skin condition, fine wrinkles and photoaging. The strongest antioxidant properties are characterized by the following vitamins: A, C, and E, as well as carotenoids and also polyphenolic compounds [Wawer, Eggert and Hołub 2015; Wilczyński et al. 2017]. Through its high content of polyphenols, which inhibit lipid degradation, chokeberry has not only found application in food preservation, but can also be used in the cosmetics industry as a substitute for artificial preservatives [Wilczyński et al. 2017]. Chokeberry owes its dark berry colour, which is practically black, to the anthocyanins it contains [Wawer, Eggert and Hołub 2015].

After analysing the presented properties with which the chokeberry fruit is characterized, it is proposed to use it in skin care preparations for all skin types, with a special distinction for mature, problematic skin with erythematous changes, as well as inflammatory conditions [Milutinović et al. 2021]. The authors of the study [Ćujić et al. 2017] found that *Aronia Melanocarpa* Fruit Powder may have a beneficial effect and potential with aging skin, thus slowing the appearance of wrinkles and their existing alleviation. In addition, they found that chokeberry extract may be preferred in cosmetic products for treating aging skin.

In the present study, an attempt was made to develop recipes and create care masks containing *Aronia Melanocarpa* Fruit Powder obtained by an innovative

method of drying with simultaneous micronization. The technology for obtaining this powder was developed by Admor Sp.j. The raw materials obtained by the innovative Monochronic Drying & Powdering (MDP) technology pass the phytosanitary and microbiological inspections. They have the appropriate documents, as well as certificates that are strict with the law. The innovative low-temperature drying method is one that has not been used so far on such a large scale in the fruit and vegetable processing industry. This technology is new and has so far been used in Poland only on a semi-technical scale, but it is expected to be used on an industrial scale in the next few years, as it is a method that competes with the methods used so far. This method is much cheaper and there are no such requirements that the powders obtained by this process be stored in special packaging, as the powders have a suitable shelf life for storage. This technology ensures that finely dispersed powders are obtained that are natural and contain high values of important parameters for the consumer [Sadowska et al. 2015; Stevanus et al. 2017]. The raw materials involved in this technology are used in their entirety and come from quality-controlled crops. The process involves crushing the fruits and vegetables to very small sizes, with simultaneous evaporation of the water. This takes place at temperatures below 40°C, where this value as well as the drying time depend on the type of material on which the process is carried out. The powders produced in this way are natural and contain no additives, although they do contain the sensory characteristics of the fruits and vegetables that were used for this method [Sadowska et al. 2015]. The powders obtained by the innovative low-temperature drying method are characterized not only by a very good sensory quality, but also by a high content of bioactive substances as well as nutritional substances, which include polyphenols, antioxidant components, minerals, and vitamins [Sadowska et al. 2015; Stevanus et al. 2017].

It is noteworthy that no publications have been reported in the literature on the use of *Aronia Melanocarpa* Fruit Powder obtained by this innovative method in skin care masks.

It is anticipated, given these valuable properties of chokeberry and the new method of obtaining it in powder form, that it could be a very valuable raw material in skin care masks.

## 2. MATERIALS AND METHODS

### 2.1. Formulations

Different formulations (Tab. 1) of facial care masks were developed based on *Aronia Melanocarpa* Fruit Powder obtained by an innovative low-temperature drying method.

**Table 1.** Formulations of facial care masks based on *Aronia Melanocarpa* Fruit Powder obtained by an innovative low-temperature drying method

INCI Name	Concentrations [%w/w]			
	<i>Symbol of face masks</i>			
	M1	M2	M3	M4
Cetearyl Olivat / Sorbitan Olivat	6			
Cetearyl Alcohol	3			
Triethylhexanoin	6			
Salvia Hispanica (Chia) Seed Oil	2			
Butyrospermum Parkii (Shea Butter)	1			
Glycerine	3			
Sodium Benzoate and Potassium Sorbate	1			
Aqua	at 100			
Citric Acid	to pH 5.5-6.0			
<i>Aronia Melanocarpa</i> Fruit Powder	0	0.1	0.3	0.5

Source: own study.

Four formulations of skin care masks were created, differing in the proportion of the *Aronia Melanocarpa* Fruit Powder obtained by an innovative low-temperature drying method. For this purpose, the components of the oil phase (Cetearyl Olivat / Sorbitan Olivat, Cetearyl Alcohol, Triethylhexanoin, Salvia Hispanica (Chia) Seed Oil and Shea Butter) were weighed and combined by stirring on a magnetic stirrer, and heated on a water bath to 70°C. The mixture was stirred until all the ingredients were completely dissolved. Next, the components of the aqueous phase (Glycerine, Aqua) were weighed and combined by stirring on a magnetic stirrer, and also heated on a water bath to 70°C. The mixture was stirred until all the ingredients were completely dissolved. The two phases were combined by adding the aqueous phase to the fatty phase. The whole mixture was stirred on a magnetic stirrer for about 10-15 minutes, until the two phases were combined. It was then cooled to a temperature of about 30°C and homogenized using a Silent Crusher-M-homogenizer, from Heidoplph, at about 30°C at 10 rpm for 5 minutes. Then *Aronia Melanocarpa* Fruit Powder in the appropriate concentration and the preservatives

Sodium Benzoate and Potassium Sorbate were added, and then combined by vigorous mixing. The pH was adjusted with Citric Acid to a pH of about 5.5–6.0.

Next, the stability of the masks stored for a period of 7 days was evaluated alternately at 40°C for one day and at 5°C also for one day. The test was performed in a Pol-Eko hothouse, as well as an Amica refrigerator. Centrifugal tests at 2,000 rpm using a Rotofix 32 A centrifuge from Zentrifugen were also carried out for the mask prototypes made. These tests lasted 30 minutes. A visual evaluation of the facial mask prototypes was then carried out. No adverse changes were found in the appearance of any of the tested masks.

## **2.2. Dynamic viscosity**

The dynamic viscosity test of the care masks was performed at 22°C using a Brookfield rheometer type HA DV III Ultra. The dynamic viscosity evaluation was performed using Helipath type spindles at a spindle speed of 5 RPM. For each formulation, 5 independent measurements were made and then the obtained value was averaged.

## **2.3. Yield point**

The yield points of the tested masks were determined using a Brookfield HA DV III Ultra rheometer equipped with a set of vane spindles at 20°C. EZ-Yield Software was used in the study. Measurements were recorded every second of the test, running at a constant spindle speed: 1 rpm.

## **2.4. Colour evaluation**

The colour evaluation of the care masks was performed at 22°C using a Konica Minolta CR-400 colorimeter and the CIE LAB method, which is based on the measurement of three trichromatic components: L, a\*, b\*. The L measurement is the brightness and intensity of the image, the a\* component is the value between the colours red and green, and the b\* component is the value between the colours: yellow and blue. The final result from each of the tested cosmetic prototypes was the arithmetic average of the five measurements.

## **2.5. Transepidermal water loss**

Transepidermal water loss (TEWL) was examined using a Tewameter TM 300. Measurements were taken on clean, degreased skin (baseline) and 1.5 h after

applying of the facial masks. Measurements were taken using 10 women, between the ages of 35 and 40 years.

## 2.6. Statistical analyses

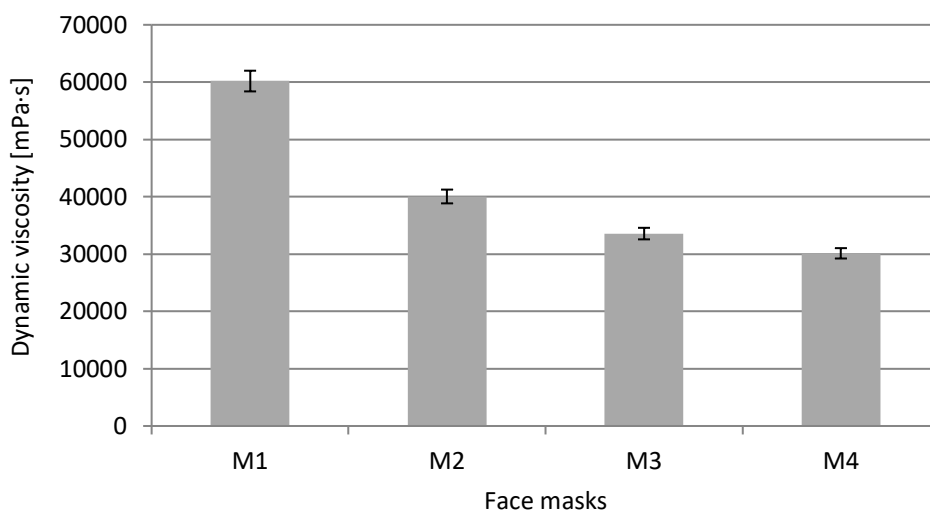
The bars in the graphs represent the arithmetic means of the values from three independent measurements. Student's t-test was used to analyse statistical differences between mean values ( $\alpha = 0.05$ ). Confidence intervals representing the measurement error for a confidence level of 0.95 were determined.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

## 3. RESEARCH RESULTS

### 3.1. Dynamic viscosity

Dynamic viscosity is an important characteristic in the quality of cosmetic masks. It primarily affects the ease of dispensing the product from the package, as well as enabling proper application to the skin.

Figure 1 shows the dynamic viscosity of facial masks that differ in the concentration of *Aronia Melanocarpa* Fruit Powder.



**Fig. 1.** Dynamic viscosity of face masks with varying concentrations of *Aronia Melanocarpa* Fruit Powder

Source: own research.

The dynamic viscosity ( $\eta$ ) of the reference mask (M1) without chokeberry in powder form was 60,000 mPa·s. As the concentration of *Aronia Melanocarpa* Fruit Powder increased, a decrease in  $\eta$  was observed. The results for masks M2, M3 and M4 containing the aronia powder content was, respectively: 0.1; 0.3 and 0.5%, oscillating between about 40,000 and 30,000 mPa·s.

Referring to the literature, it can be noted that the values of dynamic viscosity for cosmetic masks fall into a wide range, from about 10,000 to 350,000 mPa·s. This wide range depends primarily on the physicochemical form of the masks as well as on the concentration and type of rheology modifiers used.

For example [Draelos and Thaman 2007], the dynamic viscosity for cosmetic masks in the form of a gel was in the range 10,000–200,000 mPa·s. Higher values of  $\eta$  between 9700 and 30,000 mPa·s were obtained by the authors [Klimaszewska et al. 2021] for masks in the form of emulsions with the inclusion of kamchatka berry powder. Another study [Asthana et al. 2021] showed that peel-off masks were characterized by even higher dynamic viscosity values, above 140,000 mPa·s and even 240,000 mPa·s [Berings et al. 2013].

Therefore, it can be concluded that the results for the dynamic viscosity of facial masks with *Aronia Melanocarpa* Fruit Powder obtained by an innovative drying method with simultaneous micronization are within the range presented in the literature for this type of cosmetic product.

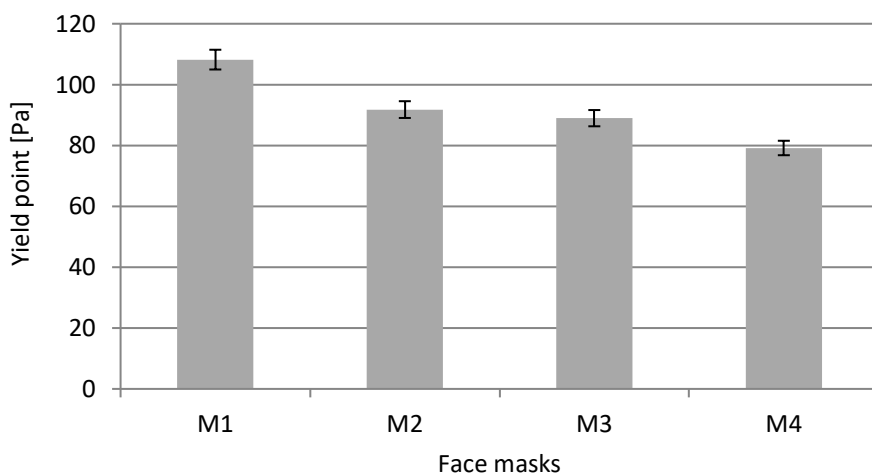
### 3.2. Yield point

The yield point is the minimum value of shear stress above which a mask will flow. It plays an important role for the cosmetic manufacturer in terms of optimal choice of packaging and dosage method, which in turn affects the efficiency and ease of use of the product by the consumer. Lower values of the parameter indicate a lighter texture and potentially better spreadability of the product on the skin.

Figure 2 shows the yield point for cosmetic masks with different concentrations of *Aronia Melanocarpa* Fruit Powder.

The values of the yield point for face masks with *Aronia Melanocarpa* Fruit Powder range from about 79 to 108 Pa. The highest value of this parameter was recorded for the mask without chokeberry (M1).

The addition of this raw material led to a decrease in the value of the flow limit by about 15–27% compared to the reference mask (M1), with the lowest value (79 Pa) characterized by the M4 mask with the participation of 0.5% *Aronia Melanocarpa* Fruit Powder.



**Fig. 2.** Yield point of face masks with varying concentrations of *Aronia Melanocarpa* Fruit Powder

Source: own research.

As in the case of dynamic viscosity, the flow limit values reported in the literature for cosmetic masks fall within a very wide range [Ngomo et al. 2014; Klimaszewska et al. 2016a]. They depend on the form of the product, the composition, including the concentration and types of rheology modifiers.

For example, the values of the flow limit of cosmetic masks in the form of emulsions containing different concentrations of blackberry seed extract ranged from 30 to 70 Pa [Klimaszewska et al. 2016a,b].

In turn, the values of the flow limit of cosmetic masks containing kamchatka berry in powder form presented in the work [Klimaszewska et al. 2021] ranged from 16 to 50 Pa.

At the same time, the formulation containing 0.9 wt% Kamchatka berry obtained the lowest value of the yield point and had the best spreadability on the skin among the tested facial masks.

Higher values of the melt limit (about 130 Pa) were recorded for the mask with the rheology modifiers: carrageenan gum and xanthan gum, in a mass ratio of 5:5 [Klimaszewska et al. 2016b]. High values of the yield point, around 400 Pa, are recorded for clay-based masks, such as Cameroonian mineral clays [Ngomo et al. 2014]. It should be noted, however, that too high values of the melt flow limit can translate into difficulties in spreading the products on the skin [Kulawik-Pióro et al. 2020].



### 3.3. Colour evaluation

Colour is one of the most important characteristics of raw materials, intermediates and final products in the field of many industries. The CIE Lab model is the current, internationally most widely used method for measuring and ordering colours. Based on the results obtained (Tab. 2), the relationships between the concentration of *Aronia Melanocarpa* Fruit Powder in individual face masks and the values of the component colours of the CIE Lab model were determined.

**Table 2.** Results of colorimetric evaluation of face masks

Face mask	Parameter		
	L	a*	b*
M1	47.27 ±1.42	-0.84 ±0.03	0.21 ±0.01
M2	42.16 ±1.26	2.62 ±0.08	1.06 ±0.03
M3	37.81 ±1.13	5.01 ±0.15	1.76 ±0.05
M4	33.63 ±1.00	6.48 ±0.19	1.23 ±0.04

Source: own research.

Based on the obtained results for parameter L (brightness), it was found that as the concentration of *Aronia Melanocarpa* Fruit Powder in the formulation of the face masks increases, the brightness of the samples decreases, with values ranging from 33.63 to 47.27. The highest value of parameter L (47.27) was recorded for mask M1 without *Aronia Melanocarpa* Fruit Powder, while the lowest value of brightness L (33.63) was obtained by mask M4 containing 0.5 wt.% *Aronia Melanocarpa* Fruit Powder. The obtained values of parameter a\* determined the limit of red and green colour, while parameter b\* determined the limit of the yellow and blue colour increase with the increase of the concentration of *Aronia Melanocarpa* Fruit Powder in the formulation of face masks. In the case of parameter a\*, the results were between -0.84 and 6.48. Thus, sample M1 without *Aronia Melanocarpa* Fruit Powder was characterized by a shade of a green colour (negative values of parameter a\*). The addition of *Aronia Melanocarpa* Fruit Powder caused a change towards a red buff colour (positive values of the a\* parameter). In analysing the obtained results of the b\* parameter, it was found that all masks obtained positive values of this parameter, informing about their yellow colour.

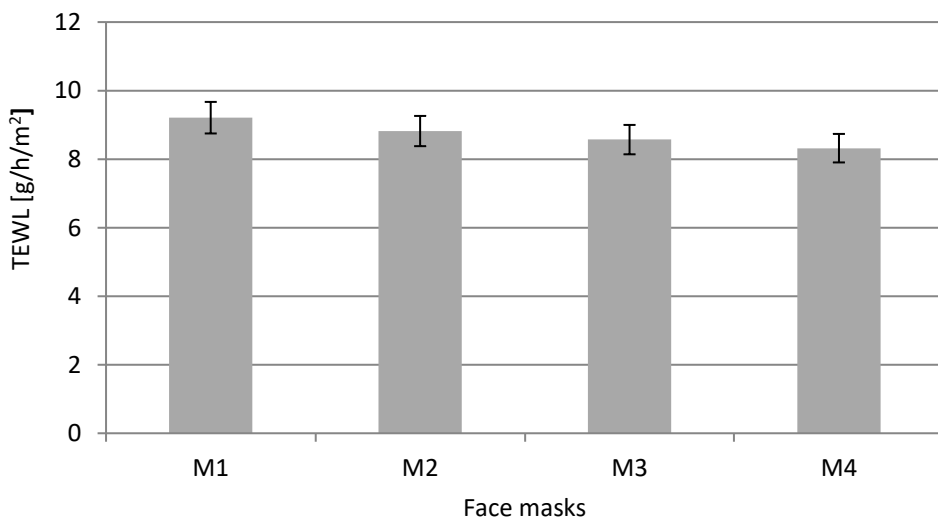
Similar observations were obtained by the authors of a paper [Bujak et al. 2021], where they analysed the colour evaluation of cosmetic emulsions with the participation of plant extracts, i.e. globe amaranth (*Gomphrena globosa* L.), butterfly pea (*Clitoria ternatea* L.), safflower (*Carthamus tinctorius* L.), pomegranate (*Punica granatum* L.), and corn poppy (*Papaver rhoeas* L.). They observed a change in the colour of the emulsion from white (base sample) to yellow, red and blue-violet with the extracts tested. Studies discussing the effect of blue honeysuckle powder added to the formulation of face masks on the colour change of the cosmetic are also known

[Klimaszewska et al. 2021]. The authors showed that an increase in the concentration of blue honeysuckle powder in cosmetic masks caused a dominance of the red colour, inducing a change in the saturation and hue. Thus, it can be concluded that natural raw materials affect the colour of the final product, which unfortunately can also change during storage.

### 3.4. TEWL

Transepidermal water loss is extremely important for assessing the performance of the skin's water barrier. Too much water vapor released from the skin may indicate poor condition of the epidermal barrier. TEWL values ranging from 0 to 10 g/h/m<sup>2</sup> indicate very healthy skin.

The results of the transepidermal water loss after the application of a brewing mask with different concentrations of *Aronia Melanocarpa* Fruit Powder are shown in Figure 4.



**Fig.4.** Transepidermal water loss after the application of face masks with varying concentrations of *Aronia Melanocarpa* Fruit Powder

Source: own research.

TEWL values after the application of the tested M1-M4 masks varied between 8.32–9.21 g/h/m<sup>2</sup>, which means that all the tested prototypes show beneficial effects on the skin. However, it should be noted that as the concentration of *Aronia Melanocarpa* Fruit Powder in the face masks increases, the loss of water from the

skin is reduced. Therefore, it can be concluded that this ingredient has a beneficial effect on the skin by preventing water loss from the epidermis.

#### 4. CONCLUSIONS

This article presents the results of a study on the development of facial care masks with *Aronia Melanocarpa* Fruit Powder obtained by an innovative low-temperature drying method. The effect of chokeberry concentration (0–0.5%) on selected properties of the obtained masks was verified, i.e. dynamic viscosity, yield point, colour evaluation and transepidermal water loss. Based on the results obtained, it was found that:

- The dynamic viscosity of the tested masks ranges from 30 129 to 60 187 mPa·s. As the percentage of *Aronia Melanocarpa* Fruit Powder increases, the  $\eta$  values decrease.
- The results of the yield point correlate with those obtained for the dynamic viscosity of the masks. The lowest yield point values were recorded for products with the highest concentration of *Aronia Melanocarpa* Fruit Powder. Therefore, it can be assumed that these masks will spread well on the skin.
- As the concentration of *Aronia Melanocarpa* Fruit Powder in the face masks increases, their brightness decreases – parameter L, and the values of parameter  $a^*$  and parameter  $b^*$  increase. Thus, an increase in the concentration of the plant powder in the formulation caused the dominance of red and yellow colours, resulting in a change in saturation and hue.
- After application of the masks to the skin, transepidermal water loss ranged from 8.32 to 9.21 g/h/m<sup>2</sup>, which means that all the tested masks showed beneficial effects on the skin. The lowest water loss was recorded for the masks with the highest concentration of *Aronia Melanocarpa* Fruit Powder.

To conclude, it can be connoted that *Aronia Melanocarpa* Fruit Powder obtained by the innovative method of drying with simultaneous micronization can be successfully used in the formulations of cosmetic masks. The proposed solution may be an interesting alternative for cosmetic manufacturers.

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