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# VALORISATION OF *ARONIA MELANOCARPA* POMACE FOR DEVELOPMENT OF FUNCTIONAL INGREDIENTS WITH HIGH NUTRITIONAL VALUE AND ANTIOXIDANT CAPACITY

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***Abstract***

*At the industrial level, the fruits of Aronia melanocarpa are processed mainly in the form of juice, resulting significant quantities of Aronia pomace, which is noted by the content of polyphenols, antocyanins, dietary fibers, pectic substances, minerals and vitamins. In this paper are presented the results of the research performed to achieve a functional ingredient (powder) from Aronia waste resulting in fruit juice industry. For this purpose, Aronia pomace (from the conventional and organic culture of our country) was subjected to a convective drying process at 50°C or lyophilisation at -55°C to protect the bioactive compounds to a moisture content to allow their milling and turning them into powder and their stability in terms of quality. Powder achieved from Aronia pomace is characterized by total dietary fiber content (55.42-58.67%), minerals (1.97-2.73%), total sugar (12.72-15.89%), vitamin C (9.05-26.08 mg/100 g), and total polyphenols (50.26-96.92 mg GAE/g). At the same time, powder achieved from Aronia pomace has antioxidant capacity. Due to its complex biochemical composition and antioxidant potential, the functional ingredient achieved from Aronia pomace can be used to fortify bakery and pastry products.*

***Key words***: *Aronia, pomace, powder, total polyphenols, dietary fiber.*

## INTRODUCTION

*Aronia melanocarpa* fruits are some of the richest sources of bioactive compounds, having high biological and nutritional values (Horszwald et al., 2013; Janković et al., 2016; Chrubasik et al., 2010). At the same time, these fruits are a good source of polyphenols, which have high antioxidant activity. The main group of polyphenols found in this plant are anthocyanins, which are responsible for astringent taste and intense coloring (Caruso et al., 2016; Kulling and Rawel, 2008). Many recent studies have demonstrated the positive multidirectional effects of these compounds on health. Anthocyanins positively influence the circulatory system and the functioning of the heart. They stimulate insulin secretion and improve retinal functioning (Bermúdez-Soto et al., 2007; Lala et al., 2006; Li et al., 2017). Jurikova et al. (2017) mention that the fruits of *Aronia melanocarpa* are an important source of antioxidants, especially polyphenols, such as phenolic acids (neochlorogenic and chlorogenic acids) and flavonoids (anthocyanins, proanthocyanidins, flavanols and flavonols), especially, cyanidine-3-galactoside and cyanidine-3-arabinoside, as well as units of (-) epicatechin. Due to the high content of these bioactive compounds, the fruits of *Aronia melanocarpa* have a wide range of positive effects, such as a strong antioxidant activity and potential medicinal and therapeutic benefits (gastroprotective, hepatoprotective, antiproliferative or anti-inflammatory effects). These also could contribute to the the prevention of chronic diseases, including metabolic disorders, diabetes and cardiovascular diseases, due to the impact on lipid profile, of plasmatic glucose à *jeun* and blood pressure. Popularity of *Aronia melanocarpa* fruits is increasing due to its association with potential health benefits, such as: antioxidant, antibacterial, antiviral, antimutagenic, anticancer, cardioprotective, hepatoprotective, gastroprotective, antidiabetic and anti-inflammatory properties. Benefiacial effects of *Aronia melanocarpa* fruits have been demonstrated by *in vitro* and *in vivo* studies (Cvetanović et al., 2018; Kokotkiewicz, Jaremicz, & Luczkiewicz, 2010). Due to the astringent taste, to be accepted by consumers, *Aronia melanocarpa* fruits are dehydrated and then are ground and transformed into powders and used to obtain teas, instant juices, fruit yogurt, desserts and food supplements (Sadowska et al., 2019).

After obtaining the *Aronia* juice, the juice of and the juice of *Aronia and* elderberry (*Sambucus nigra* L.) result important quantities of waste (*Aronia* pomace, elderberry pomace), which is noted for its content in polyphenols, anthocyanins, food fibers, pectic substances, minerals (K, Ca, Mg, Fe) and vitamins (Grunovaite et al., 2016; Rodrigues et al., 2018). Due to the high content of polyphenols, waste *Aronia* fruits and elderberry (*Sambucus nigra* L.) fruits, they have *antioxidant potential* and have beneficial effects on the human body, to prevent diseases caused by oxidative steess, such as cardiovascular diseases, neurodegenerative diseases, cancer, diabetes and osteoporosis (Dufour et al., 2018).

In this paper are presented the results of the research performed to achieve a functional ingredient (powder) from *Aronia* waste resulting in fruit juice industry.

## MATERIALS AND METHODS

### Samples

*Aronia melanocarpa* pomace resulted by *Aronia melanocarpa* fruits processing into juice within the Pilot Experiments Plant for Fruits and Vegetables Processing in IBA Bucharest, using a juicer extractor (Philips). Within experiments were used *Aronia melanocarpa* fruits, purchased from Romanian farmers. Within experiments were used *Aronia melanocarpa* fruits from conventional (II) and organic (I and III) culture. Till processing, *Aronia melanocarpa* pomace was stored under refrigeration (3°C). *Aronia melanocarpa* pomace was subjected to dehydration by two procedures: convective dehydration with hot air at 50°C and lyophilisation at -55°C. Dehydration process of *Aronia melanocarpa* pomace, was achieved to a moisture (3.20-6.18%) which allows their milling and conversion into powder and, at the same time, their stability in terms of quality. Milling of dried semi-finished products was performed by using Retsch mill. The achieved functional ingredients (powders) were packed in glass containers, hermetically sealed, protected by aluminum foil against light and stored in dry and cool areas (temperature of maximum 20ºC), till to the biochemical analysis. Figure 1 shows *Aronia melanocarpa* pomace powders obtained by dehydration at 50°C, and Figure 2 those obtained by lyophilisation.

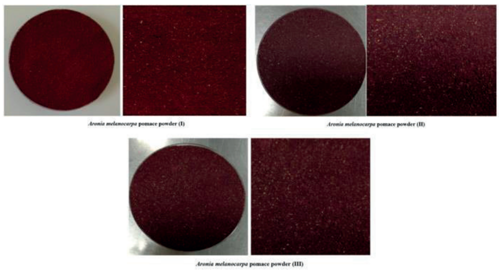

Figure 1. *Aronia melanocarpa* pomace powder (dehydration at 50°C)

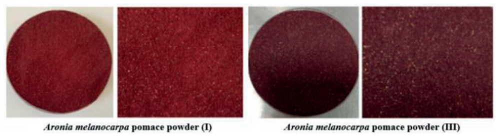


Figure 2. *Aronia melanocarpa* pomace powder (lyophilisation at -55°C)

### Methods

*Sensory analysis*

Sensory analysis (appearance, taste and smell) was performed by descriptive method.

*Physico-chemical analysis*

Measurement of the colour parameters of samples was performed at room temperature, using a CM-5 colorimeter (Konica Minolta, Japan), equipped with SpectraMagic NX software, to register CIELab parameters (the Commission Internationale de l'Eclairage - CIE), $L^*$, $a^*$ and $b^*$: $L^*$ - colour luminance (0 = black, 100 = white); $a^*$ - red-green coordinate (-a = green, +a = red); $b^*$ - yellow-blue coordinate (-b = blue, +b = yellow).

Moisture determination was performed with Ohaus Moisture Analyzer MB45 at temperature 105°C.
Protein content was determined by the Kjeldahl method with a conversion factor of nitrogen to protein of 6.25 (AOAC Method 979.09, 2005). Fat content was determined according to AOAC Method 963.15, and ash content according to AOAC Method 923.03 (AOAC, 2005).
In order to determine minerals samples were mineralized by calcination, with the addition of hydrochloric acid and hydrogen peroxide. The minerals sodium (Na), potassium (K), calcium (Ca), magnesium (Mg) and zinc (Zn) were determined by Atomic Absorption Spectrophotometer (type *AAnalyst* 400, Perkin–Elmer). Iron (Fe) was determined by Graphite Furnace Atomic Absorption Spectrophotometer (type *AAnalyst* 600, Perkin–Elmer).
Total sugar content was determined according to Schoorl method.
Total dietary fibre (TDF) was determined by enzymatic method using the assay kits: K-TDFR ''Total dietary fibre'' (AOAC Method 991.43). Determination of pectic substances (expressed as calcium pectate) was achieved by gravimetric method.
Determination of vitamin C content was performed by high performance liquid chromatography (Accela, Thermo Scientific) coupled with high resolution mass spectrometry (LTQ Orbitrap XL Hybrid Ion Trap-Orbitrap Mass Spectrometer, Thermo Scientific) using hippuric acid as internal standard (Catană et al., 2017).
Determination of vitamin E (α-tocopherol) content was performed by high-performance liquid chromatography (HPLC-DAD) (Popović et al., 2015).

*Total polyphenol content*
Total polyphenol content was conducted according to Horszwald and Andlauer (2011) with some modifications (concerning extraction media, time and mode of extraction, extract volumes of the used sample and reagents, using UV-VIS Jasco V 550 spectrophotometer), based on calibration curve of gallic acid achieved in the concentration range 0 to 0.20 mg/mL. The extraction of phenolic compounds was performed in methanol:water 50:50, and the absorbance of the extracts was determined at a wavelenght $\lambda = 755$ nm. Results were expressed as mg of Gallic Acid Equivalents (GAE) per g *Aronia melanocarpa* pomace powder.

*Antioxidant capacity*
The DPPH scavenging radical assay was conducted according to Horszwald and Andlauer (2011) with some modifications (concerning extract volumes of the used sample and reagents, using UV-VIS Jasco V 550 spectrophotometer). The reaction was performed in dark for 30 min (at ambient temperature) and after this time the absorbance was read at 517 nm. It was achieved the calibration curve Absorbance = f(Trolox concentration), in the concentration range 0-0.4375 mmol/L and the results were expressed as mg Trolox Equivalents per g *Aronia melanocarpa* pomace powder.

*Microbiological analysis*
The water activity (*A*w) was determined by an instrument Aquaspector AQS-2-TC, Nagy. The measurements were performed at 25ºC. Yeasts and molds were determined by the method SR ISO 21527-1:2009. *Enterobacteriaceae* were determined according to the SR EN ISO 21528-1:2017 method and *Escherichia coli* by SR ISO 16649-2:2007 method. *Salmonella* was determined by the method SR EN ISO 6579-1:2017.

## RESULTS AND DISCUSSIONS

*Sensory analysis*
As a result of the sensory analysis, it was found that the powders achieved from *Aronia melanocarpa* pomace, have colours from cherry to dark cherry and, respectively, brown, and have pleasant taste and smell, characteristic of these fruits.
As a result of the instrumental colour analysis, it was found that the powders obtained from *Aronia melanocarpa* pomace, by dehydration with hot air, at 50°C, are darker in colour compared to those obtained by lyophilisation, at -55°C, registering lower luminance values, respectively, $L^* = 38.55$, in the case of sample II (Figures 3, 4).

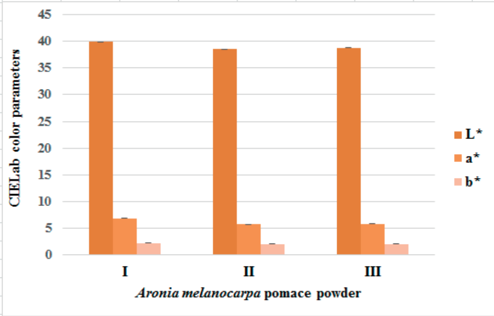


Figure 3. Colour parameters of the powders achieved from *Aronia melanocarpa* pomace (dehydration at 50°C)

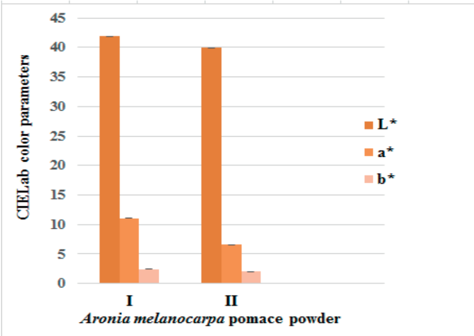


Figure 4. Colour parameters of the powders achieved from *Aronia melanocarpa* pomace (lyophilisation at -55°C)

Also, the minimum values of parameters a* (red colour coordinate) and b* (yellow colour coordinate) were also recorded for powders obtained from *Aronia melanocarpa* pomace, by dehydration with hot air, at 50°C (a* = 5.71 and b* = 1.98, values recorded in the case of sample II).

*Physico-chemical analysis*

Composition of the powders achieved from *Aronia melanocarpa* pomace is presented in Table 1.

Table 1. Physico-chemical composition of powders achieved from *Aronia melanocarpa* pomace

| Functional ingredient | Water (%) | Ash (%) | Protein (%) | Fat (%) | Total sugar (%) | Total fibre (%) |
|---|---|---|---|---|---|---|
| **Dehydration with hot air at 50°C** | | | | | | |
| Sample powder I | 5.79±0.14 | 2.63±0.03 | 6.81±0.06 | 3.11±0.03 | 15.40±0.08 | 56.85±1.05 |
| Sample powder II | 6.18±0.15 | 1.97±0.02 | 6.38±0.06 | 3.30±0.04 | 12.72±0.06 | 55.42±1.03 |
| Sample powder III | 5.22±0.13 | 2.01±0.02 | 5.67±0.05 | 3.19±0.04 | 15.52±0.08 | 57.60±1.06 |
| **Lyophilization at -55°C** | | | | | | |
| Sample powder I | 3.20±0.08 | 2.73±0.03 | 7.03±0.06 | 3.21±0.03 | 15.89±0.08 | 58.50±1.08 |
| Sample powder III | 3.85±0.10 | 2.04±0.02 | 5.80±0.05 | 3.27±0.04 | 15.80±0.08 | 58.67±1.09 |

Following the physic-chemical analysis, it was found that the powders achieved from *Aronia melanocarpa* pomace, are noted for their content in protein (5.67-7.03%), total ash (1.97-2.73%), total sugar (12.72-15.89%) and total fiber (55.42-58.67%), the obtained results being in accordance with those from specialty literature (Reiβner et al., 2019; Sidor and Michalowska, 2019).

Powders achieved from *Aronia melanocarpa* pomace (I) has the highest total ash content (2.63% in the case of powder achieved by dehydration with hot air, at 50°C and 2.73% in the case of that achieved by lyophilisation, at -55°C), protein (6.81% in the case of powder achieved by dehydration with hot air, at 50°C and 7.03% in the case of that achieved by lyophilisation, at -55°C), and the powder achieved from *Aronia melanocarpa* pomace (III) has the highest content of total sugar (15.52% in the case if powder achieved by dehydration with hot air, at 50°C and 15.80% in the case of that achieved by lyophilisation, at - 55°C) and total fiber (57.60% in the case of powder achieved by dehydration with hot air, at 50°C and 58.67% in the case of that achieved by lyophilisation, at -55°C). For *Aronia melanocarpa* powder, achieved by dehydration with hot air, at 60°C and grinding of *Aronia melanocarpa* pomace, resulted after the juice obtaining, Reiβner et al. (2018), have obtained similar values for the principal physicf-chemical indicators: moisture = 2.72%; lipids = 3.61% s.u.; protein = 5.97% s.u.; ash= 1.92%s.u.; total fiber = 59.50% s.u. Also, Sidor and Michalowska (2019), report for the powder achieved from *Aronia melanocarpa* pomace a protein content of 10.67%, lipid content of 5.15%, ash content of 1.95%.

Also, powders obtained from *Aronia melanocarpa* pomace, are noted for their content in pectic substances (6.23 – 7.75%), expressed as calcium pectate (Table 2).

Content in pectic substances of the powders achieved from *Aronia melanocarpa* pomace, obtained in this study, are in accordance with that reported by Sidor and Michalowska (7.52%).

The consumption of pectins generates an increase in beneficial microbial populations in the gastrointestinal tract, increasing production levels of short chain fatty acids and gases such as methane, carbon dioxide and hydrogen, positively affecting health (Blaut, 2002; David et al., 2014). Also, researches in the field have shown a relationship between pectins consumption and maintenance of normal blood cholesterol levels or a reduction of post-prandial glycaemic responses (Ferretti et al., 2014).

Table 2. Content in pectic substances of powders achieved from *Aronia melanocarpa* pomace

| Functional ingredient | Pectic substances (% calcium pectate) |
|---|---|
| **Dehydration with hot air at 50°C** | |
| Sample powder I | 7.10±0.18 |
| Sample powder II | 6.23±0.16 |
| Sample powder III | 7.50±0.19 |
| **Lyophilization at -55°C** | |
| Sample powder I | 7.28±0.18 |
| Sample powder III | 7.75±0.19 |

Powders achieved from *Aronia melanocarpa* pomace, are noted for their mineral content (K, Ca, Mg, Fe, Zn). Figures 5 and 6 show the mineral content of the powder achieved from *Aronia melanocarpa* pomace by dehydration, at 50°C.

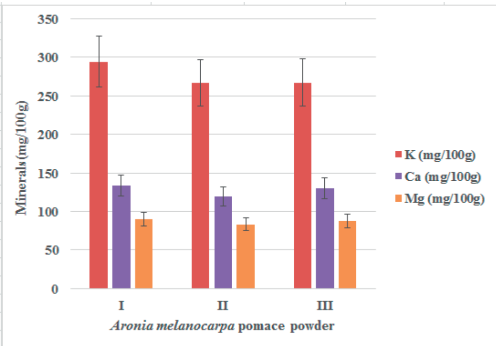


Figure 5. Mineral content (Na, K, Ca and Mg) of the powders achieved from *Aronia melanocarpa* pomace (dehydration at 50°C)

Powder obtained from *Aronia melanocarpa* pomace, the sample I, has the highest potassium content (294.04 mg/100 g). Calcium content of the powders achieved from *Aronia melanocarpa* pomace, varied in the range 119.94-133.29 mg/100 g, the maximum value being recorded in the case of powder achieved from the fruits sample III (organic culture). Magnesium content of the powders taken into study represents about 67-69.6% of the calcium content of them.

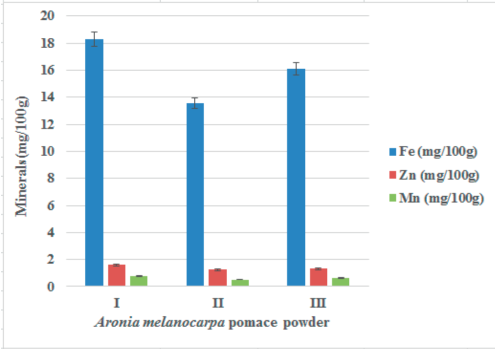


Figure 6. Mineral content (Fe, Zn and Cu) of the powders achieved from *Aronia melanocarpa* pomace (dehydration at 50°C)

Iron content of the powders achieved from *Aronia melanocarpa* pomace, varied in the range 13.55-18.30 mg/100 g, the maximum value being recorded in the case of powder achievde from *Aronia melanocarpa* pomace, sample I. Zinc content of the powders in this experimental study, varied in a narrow range: 1.24-1.59 mg/100 g (maximum value was also recorded for powder achieved from *Aronia melanocarpa* pomace, sample I. The lowest values were recorded for the manganese content (0.52-0.77 mg/100 g).

Content in K, Mg, Fe and Zn of powders achieved from *Aronia melanocarpa* pomace, by dehydration with hot air, at 50°C, is comparable to that reported by Sidor and Michalowska (2019), as being obtained by Pieszka et al. (2015), in the case of *Aronia melanocarpa* pomace, resulted after juice obtaining (K: 278 mg/100 g; Mg: 88 mg/100 g; Fe: 19.7 mg/100 g; Zn: 1.57 mg/100 g). In contrast, the Ca and Mn content of the powders taken into study is lower, compared to that obtained by Pieszka et al. (2015), in the case of *Aronia melanocarpa* pomace, resulted after the juice obtaining (Ca: 275 mg/100 g; Mn: 3.15 mg/100 g).

Between the mineral content of the powders achieved by dehydration at 50°C and, respectively, lyophilisation at - 55°C (in the case of *Aronia melanocarpa* I and III fruit samples), there are very small differences, caused by the difference of sample moisture (Table 3).

Table 3. Mineral content of the powders achieved from *Aronia melanocarpa* pomace (lyophilisation at -55°C)

| Minerals | Powders achieved from *Aronia melanocarpa* pomace | |
|---|---|---|
| | Sample powder I | Sample powder IIII |
| K (mg/100g) | 303.35 ±34.28 | 287.23±32.46 |
| Ca (mg/100g) | 136.95±14.12 | 131.56±13.56 |
| Mg (mg/100g) | 92.10±9.21 | 88.79±8.88 |
| Fe (mg/100g) | 18.80±0.56 | 16.33±0.49 |
| Zn (mg/100g) | 1.63±0.09 | 1.34±0.08 |
| Mn (mg/100g) | 0.79±0.02 | 0.64±0.02 |

*Bioactive compounds content*

Powders achieved from *Aronia melanocarpa* pomace are sources of bioactive compounds: total polyphenols, vitamin C and vitamin E (α-tocoferol) (Table 4).

Table 4. Bioactive compounds content of the powders achieved from *Aronia melanocarpa* pomace

| Functional ingredient | Total polyphenols (mg GAE/100g) | Vitamin C (mg/100g) | Vitamin E (mg/100g) |
|---|---|---|---|
| Dehydration with hot air at 50°C | | | |
| Sample powder I | 6105.55±152.64 | 12.44±0.41 | 1.27±0.07 |
| Sample powder II | 5025.65±125.64 | 9.05±0.30 | 1.12±0.06 |
| Sample powder III | 6585.28±164.63 | 14.25±0.47 | 1.40±0.08 |
| Lyophilization at -55°C | | | |
| Sample powder I | 8566.85±214.17 | 20.53±0.68 | 2.16±0.12 |
| Sample powder III | 9692.32±234.63 | 26.08±0.86 | 2.45±0.13 |

Total polyphenol content of the powders achieved from *Aronia melanocarpa* pomace varied in the range 5025.65-9692.32 mg GAE/100 g, and vitamin C content in the range 9.05-26.08 mg/100 g. It is worth noting that the powders achieved from *Aronia melanocarpa* pomace, by lyophilisation, have a higher content of polyphenols, respectively, vitamin C, compared to those achieved by dehydration at 50°C (1.4 times, respectively, 1.47 times in the case of total polyphenols; 1.65 times, respectively, 1.83 times, in the case of vitamin C). Also, in the case of these powders, the content of vitamin E (α-tocopherol) varied in the range 1.12-2.45 mg/100 g (the minimum value was recorded in the case of powder achieved from *Aronia melanocarpa* pomace, by dehydration, at 50°C, and the maximum one in the case of that obtained by lyophilisation, at -55°C).

*Antioxidant capacity*

Due to their content in antioxidants (total polyphenols, vitamin C, vitamin E etc.), powders achieved from *Aronia melanocarpa* pomace, have antioxidant capacity (Figure 7).

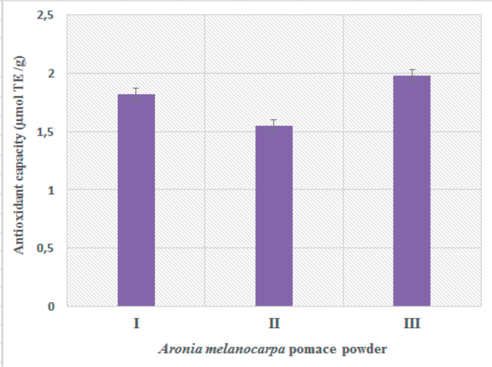


Figure 7. Antioxidant capacity of the powders achieved from *Aronia melanocarpa* pomace

In the case of the powders achieved from *Aronia melanocarpa* pomace, by dehydration, at 50°C, the antioxidant capacity varied in the range 1.55-1.98 µmol Trolox Equivalents/g (Figure 7). Powders achieved from *Aronia melanocarpa* pomace, by lyophilisation, at -55°C, recorded higher values of antioxidant capacity: 2.60 µmol Trolox Equivalents/g (sample I) and, respectively, 2.95 µmol Trolox Equivalents/g (sample III).

*Microbiological analysis*

Results of the microbiological analysis of the powders achieved from *Aronia melanocarpa* pomace are presented in the Table 5.

Table 5. Microbiological analysis of powders achieved from *Aronia melanocarpa* pomace

| Functional ingredient | Yeast and mold (CFU/g) | *Enterobacteriaceae* (CFU/g) | *Escherichia coli* (CFU/g) | *Salmonella* (in 25 g) | Water activity (Aw) |
|---|---|---|---|---|---|
| Dehydration with hot air at 50°C | | | | | |
| Sample powder I | < 10 | < 10 | < 10 | absent | 0.305 |
| Sample powder II | < 10 | < 10 | < 10 | absent | 0.315 |
| Sample powder III | < 10 | < 10 | < 10 | absent | 0.300 |
| Lyophilization at -55°C | | | | | |
| Sample powder I | < 10 | < 10 | < 10 | absent | 0.285 |
| Sample powder III | < 10 | < 10 | < 10 | absent | 0.290 |

Microbiological analysis shown that the achieved powders are in the frame of the provisions of the legislation into force. These powders show low values of water activity (0.285-0.315), which give them microbiological stability.

## CONCLUSIONS

Powders achieved from *Aronia melanocarpa* pomace are important sources of minerals (K, Fe, Mg, Ca and Zn), dietary fibres and bioactive compounds.

The powders achieved in this study, are noted for their content in polyphenols (5025.65-9692.32 mg GAE/100 g) vitamin C (9.05-26.08 mg/100 g) and vitamin E (1.12-2.45 mg/100 g). Also these powders have antioxidant capacity (1.55-2.95 μmol Trolox Equivalents/g), being beneficial in a healthy diet for prevention of diseases caused by free radicals.

On the other hand, powders achieved from *Aronia melanocarpa* pomace are characterized by high dietary fibre (55.42-58.67%) and pectic substances (6.23-7.75%), content being important sources to increase the fibre and pectic substances content of foods (bakery products, pastry products, etc.). Increase of the fibre content in case of the sweet flour products is very important because it reduces their glycemic impact on the human body, thus preventing the development of diabetes mellitus and obesity. Also, dietary fibre have an important role in promoting feeling of satiety.

Powders achieved from *Aronia melanocarpa* pomace can be regarded as functional ingredients and can be used to fortify food products (bakery and pastry products, especially) in order to increase the nutritional value and their antioxidant capacity.

## ACKNOWLEDGEMENTS

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