Aronia Berry Fruits Processing by Spray Drying: From By-product to High Quality Functional Powder

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SUMMARY

The main aim of this study was to analyze the solid-liquid extraction followed by spray drying as a technological pathway for utilization of aronia fruit dust, which represents a by-product of filter tea factory. In the current study, ultrasound-assisted extraction was applied for production of aronia liquid feeds and maltodextrin was used as a carrier and encapsulating agent. In spray drying process, the influence of inlet temperatures and maltodextrins type and concentration on process efficiency and powders properties were observed. Obtained powders were characterized in terms of physical and chemical properties. It was determined that the powder produced using inlet temperature 140 °C, maltodextrin 19.7DE in concentration of 40 % possess the most desirable characteristics. It was recorded that the increase in maltodextrin concentration decreases the moisture content, powder hygroscopicity, and content of bioactive compounds, whereas water solubility index and particle size increase. The increase in maltodextrine dextrose equivalent affects the powder hygroscopicity and water solubility index by increasing them, while the increase of inlet temperature causes a decrease in moisture content of aronia powders.

Key words: aronia, by-product, ultrasound assisted extraction, spray drying, powder

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INTRODUCTION

In recent years, *Aronia melanocarpa* L. (also known as aronia, black chokeberry) has attracted a great interest due to its unique composition of bioactive compounds which ensures multiple health-promoting properties (1). Namely, it was found that aronia is one of the richest sources of phenolic compounds and that the content of proanthocyanidins, anthocyanins and phenolic acids in this fruit is quite high (2). Polymeric procyanidins with up to 66% of total polyphenols are the major subclass and the main contributors to the specific astringent, tart taste of aronia berry fruit and their products (3). Anthocyanins make approximately 25% of total phenols compounds present. They consist mainly of cyanidin glycosides: glucoside, galactoside, arabinoside and xyloside, with cyanidin arabinoside and galactoside accounting for 60–75% of the total anthocyanins (3). According to the study of Szopa *et al.* (4) aronia berry fruit contained significant amounts of two phenolic acids: chlorogenic and neochlorogenic acid. Antioxidant activity of aronia is significantly high (5). Positive effects of aronia on human health were the subject of numerous studies. A few of them showed favorable effects of aronia in: control and prevention of diabetes and diabetes-associated complications (6), prevention and treatment of cardiovascular diseases, treatment of colon cancer, and hepatoprotective effect (1-2, 7).

In food industry, various by-products/wastes are generated during the processing of aronia berry fruit. Aronia cake is by-product/waste generated during the production of aronia juice. After juice production, by application of adequate technological treatment, this cake is transformed into the form that is further used in the production of granola, chocolate, natural food additives, etc. For its application in fruit filter tea production, according to Ramić *et al.* (8), such cake need to be dried, milled, grounded and fractionated. After such processing, approximately 20% of totally processed cake is of particle size lower than the size of pores of filter tea bags, and as such it is discharged as by-product from filter tea factory. Since this fraction cannot be packed into filter bags it represents by-product also known as “aronia fruit dust”.

According to our previous study (8), aronia fruit dust, by-products/wastes of aronia berry fruit processing in food factory, represent a valuable source of polyphenolic constituents. In the same study it has been showed that ultrasonic-assisted extraction can be successfully applied for recovery of aronia polyphenolic constituents and for production of new functional products in a form of liquid extracts. However, to produce a more stable and “easier for manipulation” products, adequate drying technique, intended for transformation of liquid extracts into the powder forms, should be used. The production of stable powder, especially in the case of liquid extracts with high concentration of low molecular sugars, such are aronia juice and its liquid extracts, represent one of the major challenges in the powder production. Several drying techniques have been proposed for the
production of herbal and fruit powders, among them spray drying (SD) has been most widely utilized. According to Gonnissen et al. (9), continuous technological operations for production of powders, such as SD, have been preferred over batch processes due to reduced time-to-market, scale-up benefits, and improved quality of product (absence of batch to batch variations). For production of adequate powder, SD technology requires adequate adjustments of operating conditions and composition of the feed solution (mixture of liquid extract and additives). Adequate adjustments of operating conditions refers to alteration of: inlet (t₁) and outlet (t₂) temperatures, atomization flow, liquid flow rate, solids concentration in liquid feed, and concentration of added drying additives. Many problems can occur during powder production and further storage and powder utilization. One of the most frequent problem encountered in SD process is certainly the stickiness of extracts on SD unit walls (10). During storage, the stickiness issue can also occur, and this affects the products manipulation. The stickiness (cohesion and adhesion) in the process of drying is provoked by the higher content of low molecular sugars present in the liquid feed and/or by application of higher drying temperature than glass transition temperature (t₉) of the liquid feed. Low molecular sugars have low t₉, therefore, if their concentration in liquid feed is high, they can induce the decrease of Tg of the entire feed. According to Johari et al. (11), if t₉ of the feed is lower than t of the drying process (for at least 10–20 °C), the process will be unsuccessful. Since water has very low t₉ (-135 °C), it can provoke significant t₉ depression of produced powders causing a decrease of the free flowing properties, an increase of caking property, as well as manipulating problems during further use of such products. Therefore, considering the water content in powders, two main requirements should be respected: production of powders with the lowest possible content of water, and their further adequate packaging and storage in terms of adequate moisture and temperature conditions.

Liquid extracts can be successfully dried with improvements in dryer design/operation, previous addition of carriers to extracts, or both (10). Improvement of the drying operation can be achieved by analysis of main influential parameters and further adequate set-up of drying conditions. Therefore, to ensure production of quality powders from high-in-sugar liquid feeds, such as aronia liquid extracts, adequate set-up of t₁ and t₂, and addition of adequate drying carrier needs to be included. Currently, maltodextrins (MDs) are considered as one of the most suitable drying carriers in the food and pharmaceutical industries (12-14). Therefore, the main aim of this study was to analyze and to propose a new technological pathway for utilization of aronia by-products/wastes. Hereof, solid-liquid extraction followed by SD process, for recovery of bioactive compounds from aronia fruit dust and for production of new stable functional product in powder form, was applied. Drying was performed after addition of MDs, as carrier and encapsulating agents. Impacts of different MDs and their concentration, as well as impact of t₁, on
process efficiency and powder properties (chemical and physical), were observed.

MATERIALS AND METHODS

Samples and reagent
Folin-Ciocalteu reagent and gallic acid were purchased from Sigma-Aldrich Chem (Steinheim, Germany). Maltodextrin DE19.7, Maltodextrin DE13.1 and Maltodextrin DE5.9 were purchased from Brenntag (Mülheim, Germany). All other chemicals and reagents were of analytical reagent grade. Aronia fruit dust was donated by producer of herbal and fruit filter tea “Fructus” Backa Palanka, Serbia.

Preparation of liquid feed (LF)
For production of aronia liquid extract ultrasound-assisted extraction (UAE) was applied. Extraction conditions were set according to previously defined optimal conditions (temperature of 70 °C, extraction time of 80.1 min, ultrasonic power of 206.64 W and 50 % (w/w) ethanol as extraction solvent). In all experimental runs, 10.0 g of material was mixed with 50 mL of solvent in 100 mL flasks. Ultrasound-assisted extraction was performed in sonication water bath (EUP540A, Euinstruments, France) with fixed frequency at 40 kHz (10). MDs of different dextrose equivalents (19.7DE, 13.1DE and 5.9DE) in different concentrations (20, 40 and 60 %) were added to the prepared liquid extracts and mixed for 0.5 hour prior SD process. The described procedure was repeated likewise for preparation of 9 different LFs: 19.7DE with 20, 40, and 60 % of MD; 13.1DE with 20, 40, and 60 % of MD; 5.9 DE with 20, 40, and 60 % of MD.

Spray drying process
The prepared LFs were dried using laboratory Mini Spray Dryer B-290 (Buchi, Switzerland) of with following characteristics: evaporation rate 1.0 L/h for water solutions, maximal inlet temperature ($t_i$) of 230 °C, heating capacity of 230 W, maximal air flow of 35m³/h, nozzle diameter of 0.7 mm. LFs were dried at three different $t_i$: 120, 140 and 160 °C, while outlet temperature ($t_o$) was kept constant at 80±5 °C. The feeding rate was constant at 10 mL/min. The obtained powder was separated from air by a cyclone. The production yield of SD process was determined according to the mass of total solids measured in the feed and the mass of dry powder obtained at the end of the process in powder receptacle. Yield was expressed as a percentage of the mass of final product compared to the total amount of the spray dried materials.

Powder characterization
Moisture content
The moisture analyzer with halogen heating (MA 35, Sartorius) was used to determine moisture content at set temperature of 105 °C (Laboratory oven, Series 11446, Sutjeska, Belgrade, Serbia). Moisture content analyses of all samples were performed immediately after the SD process. All experiments were performed in three replicates.

Hygroscopicity
Hygroscopicity of aronia powders was analyzed according to procedure described previously by Vladic et al. (15). Powder sample, approximately 1 g, was placed in an airtight container at 25 °C or in desiccator filled with NaCl saturated solution (relative humidity 70 %). Increase in mass, caused by absorbed water, was measured after 48 h. Hygroscopicity was expressed as a gram of absorbed water per 100 g of powder. All experiments were performed in three replicates.

Water solubility index (WSI) and water absorption index (WAI)
The WSI index is used as a measure of degradation for powder constituents. The low WSI represents a minor degradation of constituents, defining a low number of soluble molecules present in the powder. WAI is an indicator of the powder ability to absorb water. WAI depends on the availability of hydrophilic groups, responsible for binding water molecules, and on the gel-forming capacity of macromolecules (16). The low WAI indicates better stability during the storage.
The WSI and WAI were determined according to the procedure previously described by Phoungchandan and Sertwasana (17). 1.25 g of powder and 15 mL of water were vigorously mixed in a 50 mL centrifuge tube. Afterwards, the mixture was incubated in a water bath at 30 °C for 30 min, and centrifuged for 15 min at 3000 × g (Tehnica LC-320, Železniki, Slovenia). The supernatant was collected in a pre-weighed Petri dish and the residue was weighed after being oven dried at 105 °C overnight. The amount of solids in the dried supernatant was calculated as a percentage of the total dry solids in the original 1.25 g of sample, and is an indicator of WSI. The WAI was calculated as the mass of solid pellets remaining after centrifugation divided by the mass of the original dry sample. All experiments were performed in three replicates.

Wettability
For analysis of product/powder wettability the OCA Contact Angle System (Dataphysics OCA 20, Dataphysics Inc., GmbH, Germany) was applied. In analysis, by a Specac hydraulic press (Specac Inc., USA), 0.15 g of obtained aronia powder was compressed under the pressure of 1 ton. The wetting angles were determined after 4.3 μL of distilled water was dropped onto the surface of the
pressings. Using the circle fitting method of the OCA System the change in the wetting angle was registered, from 1 to 25 s (a minimum of 5 parallel numbers).

Two liquids with known polar ($\gamma^p$) and dispersion ($\gamma^d$) components were used for the measurement. In our study these were: bidistilled water ($\gamma^p = 50.2$ mN m$^{-1}$, $\gamma^d = 22.6$ mN m$^{-1}$) and diiodomethane ($\gamma^p = 1.8$ mN m$^{-1}$, $\gamma^d = 49$ mN m$^{-1}$). According to Wu and Brzozowski (18), the solid surface free energy, as the sum of the polar ($\bar{\alpha}^p$) and non-polar ($\bar{\alpha}^d$) components, was calculated by following equation (Eq. (1)):

$$\gamma_s = \frac{4(\gamma_s^d \gamma_p^d)}{\gamma_s^d + \gamma_p^d} + \frac{4(\gamma_s^p \gamma_p^p)}{\gamma_s^p + \gamma_p^p}$$

where $\Theta$ is the contact angle, $\gamma_s$ is the solid surface free energy and $\gamma$ is the liquid surface tension.

The percentage polarity can be calculated from the $\gamma^p$ and $\gamma$ values: $(\gamma^p/\gamma)*100$ (18).

Analysis of powder morphology - Scanning electron microscopy

The morphology of the obtained aronia powders particles was analyzed similar as in our previous study (15) using Scanning Electron Microscopy (SEM) (Hitachi S4700, Hitachi Scientific Ltd., Tokyo, Japan). To induce electric conductivity on the surface of the samples sputter coating apparatus (Bio-Rad SC 502, VG Microtech, Uckfield, UK) was applied. The air pressure was 1.3-13.0 mPa.

Analysis of powder particle size - microscopic measurement

For measurement of powders particle size LEICA Image Processing and Analysis System (LEICA Q500MC, LEICA Cambridge Ltd., England), as optical microscope was used. Dry powder was dispersed on the slide and 40-100 x magnifications were applied. The particles were described by their length and width. Description was made based on the measurement of 350 particles per sample.

Differential scanning calorimetry (DSC)

The DSC measurements were performed similar as in our previous study (17) using Mettler Toledo DSC 821e thermal analysis system, with STARe thermal analysis program V6.0 (Mettler Inc., Schwerzenbach, Switzerland) (19). In analysis 2–5 mg of powder was analyzed in the temperature range from 25 to 300 °C. Applied heating rate was 5 °C min$^{-1}$, while argon, at flow rate of 10 L/h, used as a gas carrier.
X-ray powder diffraction (XRPD)

XRPD spectra were recorded similar as in our previous study (15) using BRUKER D8 Advance X-ray diffractometer system (Bruker AXS GmbH, Karlsruhe, Germany) with Cu Kα1 radiation (λ = 1.5406 Å) over the interval 5-30°/2. The applied analysis conditions were as follows: target, Cu; filter, Ni; voltage, 40 kV; current, 40 mA; time constant, 0.1 s; angular step 0.010. In the determination of the degree of crystallinity, the total area of the three peaks with largest intensity was examined, after smoothing and background removal.

Content of bioactive compounds

Analysis of total phenols (TP) content

In obtained aronia powders content of TP was determined by standard Folin–Ciocalteu procedure (20). TP was expressed as a mg of gallic acid equivalent per g of obtained powder (mg GAE/g). All experiments were performed in three replicates.

Analysis of monomeric anthocyanins (MA) content

In obtained aronia powders content of MA in aronia was estimated using VIS-spectrophotometer by the pH differential method reported by Abu Bakar et al. (21) with minor modifications (22). Two buffer systems, potassium chloride buffer, pH 1.0 (0.0025 M) and sodium acetate buffer, pH 4.5 (0.4 M), were used. Briefly, 400 μL of sample (diluted liquid extract) was added in 3.6 mL of corresponding buffer solutions and absorbance was measured against a blank probe at 510 and 700 nm.

Absorbance (A) was calculated as:

\[ A = (A_{510} - A_{700})pH_{1.0} - (A_{510} - A_{700})pH_{4.5} \]  

Anthocyanin concentration in the extract was calculated and expressed as cyanidin-3-glycoside equivalent (C3G):

\[ MA = \frac{(A \times MW \times DF \times 1000)}{M_a} \]  

where is difference in absorbance, is a molecular weight for cyanidin-3-glucoside (449.2 g/mol), is the dilution factor of the samples and is the molar absorptivity of cyanidin-3-glucoside (26.900 M/cm). Results were expressed as mg of cyanidin-3-glucoside equivalents per g of obtained powder (mg C3G/g). All experiments were performed in three replicates.
Statistical analysis

The experiments were carried out in triplicate and the results were expressed as mean ± standard deviation and considered significantly different when \( p \leq 0.05 \). One-way ANOVA was conducted to test the individual factors influence on observed property and Tukey’s HSD post hoc test was used for differences between the mean values detection (STATISTICA v. 8) (23).

RESULTS AND DISCUSSION

This study explored the possibility of aronia fruit dust to be utilized through application of solid-liquid extraction followed by SD technique. Research was focused on the influence of two dominant process parameters in SD: drying and encapsulating agents (type and concentration) and \( t_i \) (at constant \( t_o \)). The effect of drying agents was investigated in experiments where three different MDs (5.9DE, 13.1DE and 19.7DE) in three different concentrations (20, 40 and 60 %) were applied in SD of aronia liquid extract (\( t_i \) and \( t_o \) were set at 120/80 ºC) (Table 1). Effects of \( t_i \) on the process efficiency, properties and quality of obtained powders were studied at three different temperatures: 120, 140 and 160 ºC, with MD 19.7DE (\( t_o \) was 80 ºC). This MD was chosen as drying agent due to fact that it is the most common MD in the food industry. Results of this part of research are presented in Table 2. During the investigation, 15 different powders were prepared.

Process efficiency and visual appearance of obtained aronia powders

In the majority of investigated samples recovery was greater than 50 %, while in a few of them its drying efficiency was even up to 75 %. According to Bhandari et al. (24), SD process recovery greater than 50 % in the cyclone is regarded as the criteria for efficient drying in laboratory dryers. Also, phenomenon such as stickiness did not occur, while wall depositions were present in several cases but in the form of thin and non-significant deposits. Therefore, regarding the above mentioned criteria, all investigated drying and powder producing processes can be considered as efficient. Visual appearance is a very important criteria concerning powders application. This is especially the case with aronia powders used as natural colorants. Quek et al. (25) reported that in the case of watermelon powder, if the added MD was higher than 10 %, powders lost their attractive red-orange color. This was not the case in obtained aronia powders. Visual appearances of all 15 powders were adequate. Even when 60 % of MD was added, they retained attractive red-purple color.
Moisture content of obtained aronia powders

Moisture content is one of the most important criteria for evaluation of powder quality. If it is inadequate it will provoke decrease of powder stability, in terms of microbiological status and loss of physical properties. Criteria for moisture content in powders for application in functional food and pharmaceutical industries are provided in numerous official documentation. According to the United States Pharmacopeia (26), moisture content values lower than 5 % (m/m) are considered as adequate for pharmaceutical powders, including SD extracts/powders.

In present study all obtained and analyzed powders showed moisture content lower than 5 %, except in the case of powders obtained by MD 19.7DE in concentration of 20 and 40 %, at t of 120 °C. In these two cases moisture content was above 5 %, more precisely 5.72 % and 5.18 %, respectively. For all other powders, moisture content ranged from 4.08 to 4.71 %. As moisture content of obtained aronia powders (except the aforementioned two samples) was below 5 %, and in line with requirements of USP from 2007, they can be considered as adequate for use as pharmaceutical powders. This low moisture content will ensure prolonged shelf life of powders due to low occurrence of microbiological contamination. Also it will enable prolonged stability of powders’ physical properties (flowability, caking, hygroscopicity, etc.), which is of importance while handling and utilization on industrial level.

Decrease of moisture content with an increase of MD concentration and t, was noticed in all investigated cases. This observation was in accordance with the observation of Abadio et al. (27) and Quek et al. (25). According to Abadio et al. (27), the water content of the feed has an effect on the final moisture content of produced powder in SD system. Addition of MD to the feed prior to the SD process increases the total solid content and reduces the amount of water available for evaporation (28). According to Quek et al. (25), this means that powders with lower moisture content could be obtained by increasing the percentages of MD added. Shrestha et al. explained the decrease of moisture content with the increase of MD by the fact that MD has the capability to interfere with sugars in the fruit powder, which is highly hygroscopic in terms of absorbing the humidity from its surroundings (29). Therefore, higher moisture content in the case of the aforesaid two powders (obtained by MD 19.7DE in concentration of 20 and 40 %, at t of 120 °C) is probably provoked by a specific chemical structure of MD 19.7DE, in terms of much higher water binding capabilities in comparison to powders prepared by MDs of lower DE. This is in accordance with Goula and Adamopoulos (30) who studied the effect of different type of MD addition (MD 6DE, 12DE and 21DE) on the properties of tomato powder. Their results showed that the higher MD dextrose equivalent causes higher moisture content in the powder. This was explained by the chemical structure of high DE MDs, which has numerous ramifications with hydrophilic groups, and thus can easily bind water
molecules from the ambient air during powder handling after SD. This phenomenon has been overcome by increasing $t$ for powders obtained by addition of 20 and 40 % of MD 19.7DE but at higher inlet temperatures (140 and 160 °C).

Hygroscopicity of obtained aronia powders

The hygroscopicity can be defined as the capacity of powder to absorb the moisture from the ambient. According to Nadeau and Puiggali (31), in the case of powder for pharmaceutical and/or food applications, hygroscopicity has been related to the porosity of the powder or to the amorphous glassy state of sugars present in the foods (32). According to Phisut (28), the differences in water adsorption can be explained by the chemical structure of the applied drying agent. Namely, the phenomenon of water adsorption by carbohydrate can be attributed to formation of links between the hydrogen in water molecules and the hydroxyl groups in the amorphous regions of the substrate as well as in the surface crystalline regions.

Hygroscopicities of aronia powders obtained using following MDs: 5.9DE, 13.1DE and 19.7DE, in concentrations 20, 40 and 60 %, ranged from 12.44 to 15.02 % (Table 1). Hygroscopicity was measured after 48 hours. According to the obtained results, the lowest hygroscopicity was in powders obtained using MD with the lowest DE (5.9). With an increase of DE in MD, the increase of hygroscopicity is observed. This difference in water absorption rate can be explained by the chemical structure of the agent and higher binding of water molecules considering number of hydrophilic groups. The observed behavior is in accordance with Tonon’s et al. (33) observation that polymerization of MD influences the hygroscopicity of powder. According to these authors, particles of acai powder produced by using MD 10DE showed lower moisture adsorption rate than samples produced using MD 20DE. As MD 5.9DE used in this study was less hydrolyzed than MDs 13.1DE and 19.7DE, it possesses lower number of hydrophilic groups which results in lower interaction with the hydrogen present in water molecules, and therefore, lower adsorption of water. According to the results present in Table 1, the hygroscopicity of obtained aronia powders decreases with the increase of MD concentration, and the lowest one was observed for powder obtained using MD DE5.9 in concentration of 60 %. This observation is in accordance with Phisut’s statement that high concentration of MD reduces the hygroscopicity of powders obtained using SD (28). Thus, with an increase of MD concentration and decrease of DE, properties of aronia powders related to water absorption can be improved, i.e. hygroscopicity can be reduced. Although the decrease of hygroscopicity with an increase of MD concentration and decrease of DE has been noticed, the difference in hygroscopicity between each sample was not so high, app. between 0.04 and 2.1 %.
Inlet temperature affects the hygroscopicity of powders produced by SD process. According to the results present in Table 2, at the same $t_i$ for all three investigated temperatures, the decrease of aronia powder hygroscopicity with the increase of MD concentration has been noticed. On the other side, with the increase of $t_i$ in the case of aronia powders obtained by addition of 20 and 40 % of MD 19.7DE, the hygroscopicity increases. This is in accordance with the study of Tonon et al. (34) who noticed that acai powders produced at higher $t_i$ were more hygroscopic. According to Phisut (28), this is related to the water concentration gradient between the product and the surrounding air, which is greater for the less moist powder. In the powders obtained by addition of MD 19.7DE in concentration of 60 %, there was no significant change of hygroscopicity with increase of $t_i$.

**WSI and WAI**

According to Hogekamp and Schubert (35), ideal powder behavior implies that the powder should wet quickly and thoroughly, sink rather than float, and disperse/dissolve without lumps. These properties of powders can be closely described using calculation of water solubility index (WSI) and water absorption index (WAI). Solubility of powders impacts their further application. Thus, powders of high WSI can be used directly as instant food products, while those of lower WSI need to be modified. It is preferable for powders that WAI is the lowest possible, conversely to WSI. WAI depends on the availability of hydrophilic groups and the gel formation capacity of the macromolecules (36).

The WSI of aronia powders ranged from 47.07 to 67.09 % (Table 1 and Table 2). An increase of WSI with an increase of MD concentration, for all three MDs types, was noticed (Table 1, Table 2). This observation is in accordance with observations of two research groups (19, 26) which explored powders production of ginger and orange juice. Aronia powders obtained using MD 19.7DE showed a higher WSI in comparison to powders obtained using MD 5.9DE and MD 13.1DE. More precisely, the increase of WSI with the increase of DE for all observed MDs concentrations was noticed (Table 1). This observation can be explained by the chemical structure of each investigated MD. Since MDs with lower DE are less hydrolyzed than MDs with a higher DE, they possess a lower number of hydrophilic groups for building the links with surrounding molecules of water. According to obtained results, it can be concluded that WSI of aronia powders can be improved by applying MDs with a higher DE and with adding of MDs in higher concentration. The desired WSI was observed in aronia powder obtained by addition of MD 19.7DE in concentration of 60 %, while the second best were WSI of powders obtained by addition of 40 % MD 19.7DE at $t_i$ of 120 °C, and by addition of MD 19.7DE in concentration of 60 % at $t_i$ 140 and 160 °C. Thus, these four powders would be the best choice as ingredients in instant food products. According to the results from Table 2, $t_i$ did not show significant effect on the WSI. The similar observation was reported by Sousa et al. (37) during the investigation.
on SD of tomato powders. WAI of aronia powders ranged from 0.28 to 0.41 g/g dry powder (Table 1 and Table 2). Decrease of WAI with increase of MD concentration was noticed, and this was in accordance with the study of Grabowski et al. (38), who noticed that adding MD reduced the water-holding capacity of the sweet potato powders. Likewise, the decrease of WAI with an increase of DE in applied MD was observed for all three investigated MDs concentrations.

**Wettability**

Wettability of powder is an important property involved in many practical problems such as: characterizing the dispersibility of a powder in liquid, evaluating the bioavailability of a medicine, selecting a liquid binder to granulate the powder, etc. (39). According to Buckton (40), improving the wettability of a drug leads to less agglomeration of drug particles in contact with the liquid. Thus, the dissolution rate of the drug powder is increased because the surface area, definitely wetted by the solvent, is greater. During the wettability measurements, it was detected that produced aronia powders had nearly the same contact angles (Θ) with polar and nonpolar solvents (Table 3 and Table 4). The angle of contact with water ranged from 37.20 to 54.43°, while the angle of contact with diiodomethane ranged from 29.82 to 40.12°. This means that produced aronia powders can be well dissolved in both media, hydrophilic and lipophilic. Since in both cases contact angles were between 0 and 90°, wettability of aronia powders can be characterized as high. No significant impact of $T_i$ on the wettability was noticed.

Based on the further results of surface free energy ($\gamma$) and the polarity (%) (Table 3 and Table 4), produced aronia powders can be considered as powders with hydrophilic character and of high polarity, which is of importance while considering their possible final application.

**Aronia powder particle size, morphology and structure**

Tables 3 and 4 summarize the average particle size of obtained powders determined by optical microscopy. The applied SD procedure provided the micronized particles in all cases. Lengths of powders particles ranged from 5.04 to 12.32 μm, while widths ranged from 3.98 to 10.02 μm. Results from Table 4 showed that there was a difference between the sizes of particles in powders obtained by different MDs. The application of MD with the highest DE (MD 19.7DE) resulted in the production of powders with the highest values of both length and width. Also, the increase of particles length and width with the increase of MDs concentration was noticed. Generally, higher applied $t_i$ (140 and 160 °C) resulted in the production of particles with lower values of length and width if compared to those obtained at $t_i$ of 120 °C (Table 4), but there was no clear dependence between the decrease of particle size and the increase of inlet temperature.
Using SEM, morphologies of MDs and obtained powders were examined. Fig. 1 shows the morphologies of applied MDs (5.9DE, 13.1DE and 19.7DE). The surfaces of applied MDs were very similar, rugged surfaces containing big pores. The average size of MDs was as follows: MD 5.9DE 142±6.89 µm, MD 13.1DE 165±5.43 µm and MD 19.7DE 243±10.46 µm.

SEM analysis of powders obtained at $t_i$ of 120 ºC showed that the particles of aronia powders had mainly roundish shape and micronized size. The higher MD concentration resulted in a slightly greater particle size. In all powders, beside very small spherical (2-3 µm) particles, greater (10-20 µm) porous particles were also found. In the Fig. 2, morphologies of aronia powders obtained at $t_i$ of 120 ºC by addition of different types of MDs in concentration of 40 %, are presented. This figure interprets how the morphology changes when a different type of MD is used. SEM analysis showed that the increase of $t_i$ can help to decrease the size of powder particles. This analysis also showed that increase of $t_i$ can affect the production of more monodisperse distribution. Fig. 3 shows how the morphology and size of particles in powder produced by addition of MD 19.7 DE in concentration of 40 % was changed by the increase of $t_i$.

In the Fig. 4, the chemical structure of applied MDs was presented. According to the thermo-analytical measurements, the DSC curves indicated the water loss of the samples close to 100 ºC and a very small endothermic peak at 172 ºC for MD 13.1. Furthermore, it means that sharp endotherm was not present on the curves, therefore the crystalline structure of active agents and matrix former did not build up. On the XRPD patterns characteristic peaks could not be seen, therefore their amorphous state was verified by this method. Spray drying is an integration procedure to get micronized final solid products. All analyzed powders showed amorphous character. Amorphous form of drug has the order of liquid or melts state of matter and lack the long range order of solid crystalline state. So, no energy is required to break the bonds during the dissolution process (drug release) such in case of crystalline state. As a general, bioavailability is defined as rate and extent of drug absorption. The rapid release of drug leads to higher absorption rates. Therefore we can predict faster dissolution and improved efficacy.

Analysis of health beneficial constituents of aronia powder
Horszwald et al. (41) reported that SD technique preserves higher levels of TP compounds when compared to other drying techniques. In this study the content of TP in aronia powders obtained by SD ranged from 178.51 to 325.06 mg GAE/g of dry powder (Table 1 and 2). As such, it was significantly higher comparing to the TP reported in scientific literature for other aronia products. Thus, it was approximately from 2 to 10 folds higher in comparison to the content reported by Oszmianski and Wojdylo (3) in aronia dried fruits (78.5 mg/g), aronia pomace (105.8 mg/g), and aronia juice (37.2
mg/g). It was observed that the increase of MD concentration decreases TP for all MDs types and for all applied t. This is logical because total content of active material was decreased. Thus, the increase of MD 5.9DE from 20 to 60 % decreases the TP for 35.65 %, while the increase of concentration of MD 13.1DE and MD 19.7DE from 20 to 60 % decreases TP for 22.68 and 13.14 %, respectively. In the case of powders obtained by adding MD in concentration of 20 and 40 %, there was no significant difference between TP in powders produced by different type of MDs DE. However, in the case of higher concentration of MD (60 %) this difference was significant and it seems to be decreased with the increase of DE. According to the results from Table 2, there was a significant difference between powders obtained at different t. The lowest TP was measured in powders obtained at t of 120 °C. The highest TP was measured in aronia powders produced at 140 °C. In comparison to TP at 140 °C, TP at t of 160 °C decreased, probably due to degradation of thermo-sensitive phenolics.

Aronia powders showed high MA ranging from 26.91 to 38.39 mg C3G/g of powder (Table 1 and 2). The results were mostly in accordance with results reported by Horszwald et al. (41). In general, MA makes app. 11-15 % of TP in produced aronia powders. As in the case of TP, MA decreases with increase of MD concentration in powders. There was no significant difference in MA content in powders obtained using different types of MDs. Therefore, it can be concluded that the type of MD DE does not affect the content of MA in aronia powders. There was no significant difference in content of MA in powders obtained at different t using addition of 20 and 40 % of MD. Conversely, in the case of powders obtained by addition of 60 % of MD, the difference was significant. The highest content of MA was detected in aronia powder obtained using MD 19.7DE in concentrations of 20 % at t of 140 °C, while the lowest was determined in powder obtained at 160 °C using MD 19.7DE in concentration of 60 %.

CONCLUSIONS

This study was performed in order to investigate the possibility of aronia fruit dust utilization and recycling through application of UAE followed by the SD process. The results showed that in this way processed aronia fruit dust resulted in the production of amorphous powders, of adequate moisture content, with micronized roundish particles and very high concentration of bioactive compounds, particularly TPs. Obtained powders were of fast dissolution rate, high WSI and low WAI, thus these properties could enable utilization of powders as future constituents of fast dissolving products, such as instant tea, instant juices, effervesences, etc. Comprehensive analyses of all investigated powders and their characteristics (physical, structural and chemical) indicate that the powder produced at t of 140 °C using MD 19.7DE in concentration of 40 % is the one with the most appropriate properties.
The study also investigated the influence of main spray drying parameters (MD type and concentration, and $t_i$) on process and powder properties (chemical and structural). It was observed that an increase of MD concentration decreases the moisture content, powder hygroscopicity, and content of bioactives, while WSI and particle size are increased. Increase of MDs dextrose equivalent increases the powder hygroscopicity and WSI, while the increase of $t_i$ induces a decrease of moisture content in aronia powders.

ACKNOWLEDGMENTS

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   https://doi.org/10.1177/1082013203034938


   https://doi.org/10.1111/j.1750-3841.2006.00036.x

   https://doi.org/10.1016/j.jcis.2010.02.051

   https://doi.org/10.1016/0032-5910(90)80090-L

   https://doi.org/10.1016/j.foodchem.2013.05.103
Table 1. Impact of MD type and concentration on physical and chemical properties of aronia powders obtained at $t/t_{0}$ of 120/80 °C

<table>
<thead>
<tr>
<th>Type of MD</th>
<th>w (MD)/%</th>
<th>Process efficiency/ %</th>
<th>Moisture content/%</th>
<th>Hygroscopicity/%</th>
<th>WAI/(g/g dry powder)</th>
<th>WSI/%</th>
<th>w (TP as GAE)/(mg/g)</th>
<th>w (TMA as C3G)/(mg/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5.9</td>
<td>20</td>
<td>64.00</td>
<td>4.52±0.11$^{bc}$</td>
<td>13.86±0.1$^{a-d}$</td>
<td>0.41±0.01$^{a}$</td>
<td>47.07±0.7$^{a}$</td>
<td>277.05±0.4$^{ab}$</td>
<td>32.08±0.7$^{bc}$</td>
</tr>
<tr>
<td>5.9</td>
<td>40</td>
<td>72.8</td>
<td>4.58±0.15$^{bc}$</td>
<td>13.14±0.6$^{bcd}$</td>
<td>0.40±0.01$^{ab}$</td>
<td>49.68±0.2$^{e}$</td>
<td>238.31±0.5$^{d}$</td>
<td>29.77±0.5$^{d}$</td>
</tr>
<tr>
<td>5.9</td>
<td>60</td>
<td>74.3</td>
<td>4.08±0.04$^{c}$</td>
<td>12.44±1.1$^{d}$</td>
<td>0.38±0.05$^{ab}$</td>
<td>54.91±1.0$^{e}$</td>
<td>178.51±0.9$^{h}$</td>
<td>27.64±0.1$^{e}$</td>
</tr>
<tr>
<td>13.1</td>
<td>20</td>
<td>67.63</td>
<td>4.24±0.08$^{bc}$</td>
<td>14.98±0.5$^{a}$</td>
<td>0.37±0.08$^{ab}$</td>
<td>55.56±0.2$^{e}$</td>
<td>278.18±1.3$^{a}$</td>
<td>33.13±0.1$^{b}$</td>
</tr>
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<td>4.68±0.25$^{bc}$</td>
<td>13.97±0.0$^{a-d}$</td>
<td>0.34±0.01$^{a}$</td>
<td>57.57±0.2$^{d}$</td>
<td>261.95±0.3$^{e}$</td>
<td>28.28±0.6$^{e}$</td>
</tr>
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<td>0.32±0.05$^{ab}$</td>
<td>59.99±0.9$^{c}$</td>
<td>215.01±0.1$^{f}$</td>
<td>27.85±0.9$^{e}$</td>
</tr>
<tr>
<td>19.7</td>
<td>20</td>
<td>52.25</td>
<td>5.72±0.50$^{a}$</td>
<td>15.02±1.1$^{a}$</td>
<td>0.33±0.04$^{ab}$</td>
<td>59.99±0.7$^{c}$</td>
<td>275.65±0.3$^{b}$</td>
<td>35.11±0.7$^{a}$</td>
</tr>
<tr>
<td>19.7</td>
<td>40</td>
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<td>0.30±0.00$^{ab}$</td>
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<td>247.86±0.5$^{d}$</td>
<td>31.19±0.1$^{cd}$</td>
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<td>14.32±0.7$^{ab}$</td>
<td>0.28±0.06$^{bc}$</td>
<td>67.09±0.7$^{a}$</td>
<td>218.38±0.2$^{f}$</td>
<td>27.47±0.5$^{e}$</td>
</tr>
</tbody>
</table>

Values are shown as means ± standard deviation (n = 3). Different letters within columns indicate a significant difference, based on Tukey's HSD test at p < 0.05.

Table 2. Impact of inlet temperature ($t$) and MD concentration on physical and chemical properties of aronia powder produced with MD 19.7 DE ($t_{0}$ was constant)

<table>
<thead>
<tr>
<th>$t/^{\circ}$C, $t_{0}/^{\circ}$C</th>
<th>w (MD)/%</th>
<th>Process efficiency/ %</th>
<th>Moisture content/%</th>
<th>Hygroscopicity/%</th>
<th>WAI/(g/g dry powder)</th>
<th>WSI/%</th>
<th>w (TP as GAE)/(mg/g)</th>
<th>w (TMA as C3G)/(mg/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>120/80</td>
<td>20</td>
<td>52.25</td>
<td>5.72±0.50$^{a}$</td>
<td>15.02±0.7$^{b}$</td>
<td>0.33±0.01$^{a}$</td>
<td>59.99±1.4$^{cd}$</td>
<td>275.65±1.8$^{d}$</td>
<td>35.11±0.8$^{bc}$</td>
</tr>
<tr>
<td>120/80</td>
<td>40</td>
<td>59.00</td>
<td>5.18±0.13$^{ab}$</td>
<td>14.69±0.1$^{b}$</td>
<td>0.30±0.01$^{bc}$</td>
<td>63.25±0.9$^{b}$</td>
<td>247.86±2.0$^{d}$</td>
<td>31.19±0.2$^{de}$</td>
</tr>
<tr>
<td>120/80</td>
<td>60</td>
<td>61.84</td>
<td>4.71±0.05$^{bc}$</td>
<td>14.32±0.9$^{b}$</td>
<td>0.28±0.00$^{c}$</td>
<td>67.09±0.6$^{a}$</td>
<td>218.38±1.0$^{f}$</td>
<td>27.47±2.1$^{g}$</td>
</tr>
<tr>
<td>140/80</td>
<td>20</td>
<td>65.37</td>
<td>4.62±0.06$^{bc}$</td>
<td>17.51±1.0$^{a}$</td>
<td>0.35±0.02$^{a}$</td>
<td>56.34±0.2$^{e}$</td>
<td>325.06±1.0$^{a}$</td>
<td>38.39±0.1$^{a}$</td>
</tr>
<tr>
<td>140/80</td>
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<td>71.02</td>
<td>4.58±0.15$^{bc}$</td>
<td>15.26±0.9$^{b}$</td>
<td>0.30±0.01$^{bc}$</td>
<td>62.23±1.6$^{bc}$</td>
<td>280.70±0.5$^{c}$</td>
<td>32.60±0.5$^{cd}$</td>
</tr>
<tr>
<td>140/80</td>
<td>60</td>
<td>72.68</td>
<td>4.57±0.33$^{bc}$</td>
<td>14.50±0.5$^{b}$</td>
<td>0.30±0.03$^{bc}$</td>
<td>65.76±0.1$^{a}$</td>
<td>239.43±0.3$^{g}$</td>
<td>29.72±0.4$^{d}$</td>
</tr>
<tr>
<td>160/80</td>
<td>20</td>
<td>58.65</td>
<td>4.33±0.22$^{bc}$</td>
<td>18.21±0.9$^{a}$</td>
<td>0.32±0.02$^{bc}$</td>
<td>59.79±0.5$^{d}$</td>
<td>311.02±0.7$^{b}$</td>
<td>37.20±0.4$^{ab}$</td>
</tr>
<tr>
<td>160/80</td>
<td>40</td>
<td>54.69</td>
<td>4.52±0.14$^{bc}$</td>
<td>16.20±0.2$^{ab}$</td>
<td>0.32±0.02$^{bc}$</td>
<td>62.20±0.3$^{c}$</td>
<td>252.63±0.0$^{a}$</td>
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<tr>
<td>160/80</td>
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<td>14.91±0.7$^{b}$</td>
<td>0.30±0.01$^{bc}$</td>
<td>65.68±0.8$^{a}$</td>
<td>226.24±0.1$^{h}$</td>
<td>26.91±1.3$^{g}$</td>
</tr>
</tbody>
</table>

Values are shown as means ± standard deviation (n = 3). Different letters within columns indicate a significant difference, based on Tukey's HSD test at p < 0.05.
Table 3. Influence of MD type and MD concentration on wettability, polarity and particle size of the aronia powders obtained at t/t₀ 120/80 °C

<table>
<thead>
<tr>
<th>Type of MD</th>
<th>w (MD)/%</th>
<th>Θwater/°</th>
<th>Θdiiodomethan/°</th>
<th>γ/(mN m⁻¹)</th>
<th>Polarity/%</th>
<th>Length/μm</th>
<th>Width/μm</th>
</tr>
</thead>
<tbody>
<tr>
<td>5.9</td>
<td>20</td>
<td>45.34</td>
<td>29.82</td>
<td>65.46</td>
<td>40.13</td>
<td>6.45±1.17ᵇ</td>
<td>4.47±2.02ᶜ</td>
</tr>
<tr>
<td>5.9</td>
<td>40</td>
<td>44.13</td>
<td>30.60</td>
<td>65.30</td>
<td>38.85</td>
<td>7.12±2.49ᵇ</td>
<td>5.89±1.21ᶜ</td>
</tr>
<tr>
<td>5.9</td>
<td>60</td>
<td>43.27</td>
<td>32.45</td>
<td>64.78</td>
<td>37.91</td>
<td>7.89±2.01ᵃᵇ</td>
<td>6.01±1.57ᵇᶜ</td>
</tr>
<tr>
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</tr>
<tr>
<td>13.1</td>
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<td>37.63</td>
<td>39.50</td>
<td>65.97</td>
<td>45.20</td>
<td>8.13±1.56ᵃᵇ</td>
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<td>40.12</td>
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<td>45.98</td>
<td>9.53±1.13ᵃᵇ</td>
<td>7.55±2.01ᵃᵇᶜ</td>
</tr>
<tr>
<td>19.7</td>
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<td>40.29</td>
<td>37.90</td>
<td>64.40</td>
<td>42.87</td>
<td>9.98±1.26ᵃᵇ</td>
<td>7.63±0.86ᵇᵃ</td>
</tr>
<tr>
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<td>41.70</td>
<td>38.21</td>
<td>64.33</td>
<td>42.90</td>
<td>11.72±1.21ᵃᵇ</td>
<td>9.75±0.67ᵃᵇ</td>
</tr>
<tr>
<td>19.7</td>
<td>60</td>
<td>42.98</td>
<td>39.72</td>
<td>65.87</td>
<td>43.78</td>
<td>12.32±1.09ᵃᵇ</td>
<td>10.02±0.98ᵃᵇ</td>
</tr>
</tbody>
</table>

Values are shown as means ± standard deviation (n = 3). Different letters within columns indicate a significant difference, based on Tukey’s HSD test at p < 0.05.

Table 4. Impact of t and MD concentration on wettability, polarity and particle size of aronia powders produced with MD 19.7 DE

<table>
<thead>
<tr>
<th>t/°C, t₀/°C</th>
<th>w (MD)/%</th>
<th>Θwater/°</th>
<th>Θdiiodomethan/°</th>
<th>γ/(mN m⁻¹)</th>
<th>Polarity/%</th>
<th>Length/μm</th>
<th>Width/μm</th>
</tr>
</thead>
<tbody>
<tr>
<td>120/80</td>
<td>20</td>
<td>40.29</td>
<td>37.90</td>
<td>64.40</td>
<td>42.87</td>
<td>9.98±1.26ᵃᵇ</td>
<td>7.63±0.86ᵇᵃ</td>
</tr>
<tr>
<td>120/80</td>
<td>40</td>
<td>41.70</td>
<td>38.21</td>
<td>64.33</td>
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<td>11.72±1.21ᵃᵇ</td>
<td>9.75±0.67ᵇᵃ</td>
</tr>
<tr>
<td>120/80</td>
<td>60</td>
<td>42.98</td>
<td>39.72</td>
<td>65.87</td>
<td>45.98</td>
<td>12.32±1.09ᵃᵇ</td>
<td>10.02±0.98ᵇᵃ</td>
</tr>
<tr>
<td>140/80</td>
<td>20</td>
<td>39.21</td>
<td>41.13</td>
<td>65.97</td>
<td>45.20</td>
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<td>4.12±0.10ᵇ</td>
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<td>39.87</td>
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<td>65.15</td>
<td>42.53</td>
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<tr>
<td>160/80</td>
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<td>43.45</td>
<td>7.20±0.40ᵇᶜ</td>
<td>3.99±2.13ᵇ</td>
</tr>
</tbody>
</table>

Values are shown as means ± standard deviation (n = 3). Different letters within columns indicate a significant difference, based on Tukey’s HSD test at p < 0.05.
Fig. 1. Morphology of MDs (MD 19.7DE, MD 13.1DE, MD 5.9DE)
Fig. 2. Effect of MD type on the structure of powders (A - powder obtained by addition of MD 5.9DE; B - powder obtained by addition of MD 13.1DE; C - powder obtained by addition of MD 19.7DE)
Fig. 3. Effect of $t$ on the structure of powders (A - powder obtained at 120 °C; B - powder obtained at 140 °C; C - powder obtained at 160 °C)

Fig. 4. Structure of MDs used for aronia powder production