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Effect of spray drying conditions on the physicochemical properties of barberry *(Berberis vulgaris)* extract powder

[1*]Sharifi, A., [2]Niakousari, M., [3]Maskooki, A. and [4]Mortazavi, S. A.

[1]*Department of Food Science and Engineering, Faculty of Industrial and Mechanical Engineering, Qazvin Branch, Islamic Azad University, Qazvin, Iran*
[2]*Department of Food Science and Technology and Faculty of Advanced Technologies, Shiraz University, Shiraz, Iran*
[3]*Department of Food Processing, Research Institute of Food Science and Technology (RIFST), Iran*
[4]*Department of Food Science and Technology, Sabzevar Branch, Islamic Azad University, Sabzevar, Iran*

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Abstract

Seedless barberry is one of the most popular native fruits in Iran. Conventionally, barberry fruit is dried and consumed as ornamental additive in traditional food. Barberry fruit juice contains high anthocyanins, therefore its spray dried powder can be used as good sources of anthocyanins. Maltodextrin(MD)-Arabic gum(AG) mixture, as carrier in various ratios, were added to the extract to prepare spray dried powder. The drying inlet air temperatures were 160°C and 180°C with air flow rate set to 50 m^3/h. The anthocyanins content, product recovery, color, moisture content, dissolution, water activity, and density of spray-dried barberry powder were measured. The results revealed that the product recovery was about 78% when the air temperature set to 160^{o}C and the barberry extract was supplemented with 75:25 ratio of MD:AG. The optimum amount of anthocyanins (390.46 mg/100g) in powder was achieved when the inlet air temperature was 160^{o}C and equal proportions of MD:AG added to the feed. Powder loose and tapped bulk density increased from 0.179 to 0.310 and 0.380 to 0.570 g/cm^3 respectively, as level of MD in the ratio increased from 7.5 to 15 g/100 ml. SEM micrographs of the powder indicate an increasing trend in particle size as result of increase of concentration of MD as drying aid. The data obtained in the present work may be used to prepare spray dried barberry extract in a commercial scale.

Introduction

Seedless barberry is grown exclusively in Iran as a commercial plant and generally consumed in traditional food and medicine. Furthermore, it may be grown in order to produce natural color, because its fruits contain great amounts of anthocyanins, phenolic compounds (Maskooki *et al*., 1993). It has been proved that the anthocyanins and other phenolic compounds possess the positive effects on health because of their antioxidant properties. Moreover, the anthocyanin compounds have been known as natural colors and can be used foodstuff (Giusti *et al*., 1999).

Barberry is a seasonal fruit and it is not available all year round. The full-ripened fruits with high red color are available in Iran from October to late November. The taste of barberry fruit juice is very sour due to its high amounts of organic acids and it cannot be consumed on its own and it is usually mixed with other fruit juice prior to consumption or it is used in the food mixtures as additive. One of the promising products of barberry extract is spray dried powder in order to produce natural antioxidant powder. Fruit juice powders have many benefits and economic potentials over their liquid counterparts such as reduced volume or weight, reduced packaging, easier handling and transportation, and much longer shelf life. Besides, their physical state provides a stable, natural and easily disables ingredients, which generally finds usage in many foods and pharmaceutical products such as flavoring and coloring agents (Shrestha *et al*., 2007).

Spray drying technique is a conventional method which extensively used to produce dried food and food ingredients in many industries and is economical method for preservation of natural compounds individually or encapsulated by coating material (Ersus and Yurdagel, 2006; Bakowska-Barczaka and Kolodziejczyk, 2011). Maltodextrin is a water soluble powder which not only can act as drying aid but also protects encapsulated ingredient from oxidation, it has low viscosity at high solids

*Corresponding author.
Email: *akramsharifi76@gmail.com*

ratio and is available in different molecular weights which provides different wall densities around the sensitive materials (Quek *et al*., 2007).

Many researchers have worked on drying process parameters for various fruits, vegetables, and other hygroscopic powders. Ersus and Yurdagel (2006) produced spray dried anthocyanin extracts by maltodextrin as a carrier from black carrot. Their results showed that adding maltodextrin (Glucodry 210, DE=20–21) as a wall material gave the highest anthocyanins content powder at the end of drying process. There are many researches on production of watermelon, orange, apple juice and various concentrated fruit juice powders by spray drying with maltodextrin and Arabic gum as carrier (Gupta, 1978; Bahandari *et al*., 1993; Quek *et al*., 2007). Tonon and Brabet (2010) evaluated anthocyanins stability and antioxidant activity of powdered Acai juice (Euterpe oleracea Mart.) over 120 days. The results demonstrated that anthocyanins degraded in the spray-dried Acai juice which produced with different carrier agents (maltodextrin, Arabic gum and tapioca starch). The objectives of this study were to prepare extract powder from seedless barberry by spray drying and to investigate the influence of conditions and ratios of MD:AG on powder physicochemical characteristics.

Materials and Methods

Preparation of barberry extract

Seedless barberries *(Berberis vulgaris)* were harvested from a barberry orchards in Ghaen city a town in south Khorasan province, Iran. The samples were cleaned, leaves, stems and all artifact materials were removed and immediately stored in a cold storage room at 4±1°C and 85±3 RH for further experiments. The extraction process was carried out using a reflux system set up in a water bath. 200g of barberry were used for each experiment. The fruit samples were first squeezed by means of a Black & Decker grinder (Model no. JBG60, USA) then is placed into a flask with 500 ml of distilled water. The flask was placed in a water bath (Model LAUDA E200, Germany) which was maintained at 60±1°C, for 2 hours. The flask was then separated from the condenser and stored in the dark for 2 hours. The barberry extract was filtered in a vacuum Buchner funnel using filter paper Wattman 40 (Maskooki *et al.,* 1993). The filtered extracts were concentrated at a low temperature (<50°C) using a vacuum rotary evaporator (BUCHI- water bath B-480, Flawil, Switzerland) to obtain a constant mass concentration of total soluble solids in concentrate (TSS) of about 14±0.2% (digital refractometer-Atago-RX-5000a). The concentrated extracts were stored in air tight dark glass bottles and kept refrigerated (4°C) for further treatments. The pH values of concentrated extracts and concentrations were measured by a pH meter (Metrohm 780, Switzerland). Vacuum oven (Lab Tech, South Korea) was used to evaluate the dry matter content of the extract and concentrate (AOAC, 1984). Pycnometeric method was used to determinate the density of samples.

Spray drying

Spray drying of fruit juice with high content of low molecular sugars and organic acids to obtain powder is fairly difficult due. This issue was described by turn results in low values of glass transition temperature (Chegeni *et al*., 2004). To ease the drying process, it is customary to add some carriers to the juice or concentrate. Various amounts of maltodextrin (dextrose equivalent 12; Tg of about 150°C) and Arabic gum(AG) (Sigma-Aldrich Corp., St. Louis, Mo., U.S.A.) were mixed thoroughly at 1000 rpm for 30 min (Silverson L4R mixer, Gemini B.V, Netherland) with barberry juice concentrate (TSS=14±0.2%) prior to spray drying. From now on, the concentrates containing various ratios of MD:AG are called "feed". The feed TSS varies between 26.84 – 27.14 %. The feed was preheated to a temperature of about 45°C prior to spray drying. Table 1 summarizes various experimental conditions in terms of inlet air temperature, ratio of MD to AG in the feed and final concentration of feed. Throughout this work, the atomizer pressure (compressed air flow), feed rate, feed temperature, and airflow rate were kept constant at 5±0.1 bar, 1.75±0.05 g/min or 500±20 L/h, 45±0.5°C and 50m^3/h, respectively. The powders were collected from the bottom of the cyclone and stored in air tight dark glass bottle at 20°C until further analysis.

Table 1. Samples prepared for spray drying

Samples	Inlet Temp(°C)	MD (g per 100 ml)	AG (g per 100 ml)	Final Brix
sample 1	180	15	0	26.72
sample 2	180	7.5	7.5	26.58
sample 3	180	11.25	3.75	26.77
sample 4	180	0	15	26.54
sample 5	180	3.75	11.25	27.14
sample 6	160	15	0	26.72
sample 7	160	7.5	7.5	26.60
sample 8	160	11.25	3.75	26.83
sample 9	160	0	15	26.48
sample 10	160	3.75	11.25	26.68

Moisture content

1 g of powder from each run was carefully weighed and left in a vacuum oven (40 L Lab Tech, Korea) at 75°C and 25 mmHg to reach a constant weight. The samples were then taken out of the oven and placed in a desiccator, and re-weighed to evaluate the moisture content. (Egan *et al*., 1981; AOAC, 1984).

Determination of water activity

The water activity (a_w) values of powders were determined using water activity meter at 20°C (Testo, 400, Germany).

Solubility

1 g of powder was dissolved in 100 ml water. The solution was centrifuged for 10 minutes at 4654 g. 25 ml of supernatant was removed and dried in the oven at 105°C for 5 hours. The percentage of solubility was calculated according to AOAC (1990).

Bulk density

Loose bulk density (ρ_{bl}) of powders was evaluated by pouring a specified weight of sample (M) into a 500 ml graduated cylinder (V_1). The cylinder with the same sample was tapped 20 times on a smooth, soft surface from a height of 10 cm and the volume of the sample was evaluated (V_2). Loose bulk density (ρ_{bl}) and tapped bulk density (ρ_{bt}) was calculated by the following equations (Goula and Adamopoulos, 2005).

$$\rho_{bl}=M/V_1 \qquad (1)$$

$$\rho_{bt}=M/V_2 \qquad (2)$$

Powder recovery

Product yield was determined by dividing the weight of the powder collected in the product collector at the bottom of the cyclone into the TSS of feed (varied between 26.84–27.14 depending on the sample). The exhaust air may carry some dried powder out of the drying chamber and some solids may stick in the dryer.

Anthocyanins content

The amount of anthocyanins was calculated by the following equation (Fuleki and Francis, 1968). The UV visible absorbance at pH=1 and 4.5 and the wavelength of 510 nm (the main barberry pigment is cyanidin-3-glycosid which indicate maximum absorption at this wavelength) was measured by a spectrophotometer (UV-Visible, Shimadzu).

$$C\ mg/l = \Delta A/\ \varepsilon\ L \times M \times D \qquad (3)$$

Where L is the length of the unit cell per cm, ε is the molar absorption of the pure pigment (molar absorption of cyanidin-3-Glycosid 29.600 l/mol.cm), C is concentration in mg/l, D is the dilution factor, ΔA is the difference between two absorption readings at pH=1 and pH=4.5 and M is molecular mass of the main anthocyanins of barberry (445 g /mol).

Determination of color

The indicators related to the color of the samples were determined by color analyzer (1002p Lutron model, India). Each sample powder were sprinkled on a black, opaque, with a completely smooth and uniform surface, randomly. The pixel value of each sample was shown by color indices (HSL and RGB). The color indices R (red), G (green) and B (blue) have been reported in the range of 0 to 1023. H (hue) index for the different samples varies in the range 0-360, that is describing a set of colors such as red, green, yellow, etc. S (saturation) and L (luminance) do not indicate a specific color. S index shows purity and color saturation and L shows brightness of the colors in two colors, black and white. In the darker sample amount of L is less (Gonzalez and woods, 2002; Ghazvini *et al*., 2009).

Scanning electron microscopy (SEM)

Surface micrographs of dried powder were obtained through scanning electron microscope (SEM) using a Stereoscan S360 Oxford model operated at 15 kV. Simple inspection of the particles morphology was carried out when different level of drying aids were used. In order to increase the ability of the specimens to conduct electricity and emit secondary electrons the sample surfaces were previously covered with a very thin gold layer by Fison sputter coater system.

Statistical analyses

All experiments were conducted in triplicate and an analysis of variance was performed. The least significant difference at $p< 0.05$ was calculated using the Duncan Multiple Range Test on SAS software version 9.1.

Results and Discussion

Physicochemical properties of Barberry extract

Every 100 g of Barberry extract had a total soluble solid of 9.50±0.2 g/100 g and total solids of 10.81±0.04 g/100 g. Its pH and specific weight measured at 20°C was 2.875±0.12 and 1.044 g/cm³, respectively. The initial content of anthocyanins in

Table 2. Mean data from the physical characteristics of powders produced

Samples	Water activity	Moisture %	Solubility %	Loose bulk density g/ml	Tapped bulk density g/ml	Product yield (%)
sample 1	0.1850^{b}	2.79^{e}	87.20^{f}	0.179^{i}	0.380^{g}	52.46^{g}
sample 2	0.1610^{d}	2.23^{g}	96.40^{b}	0.216^{e}	0.470^{e}	68.73^{f}
sample 3	0.1260^{h}	1.98^{i}	88^{ef}	0.209^{f}	0.490^{d}	50.95^{h}
sample 4	0.1840^{b}	3.28^{b}	93.80^{c}	0.202^{g}	0.420^{f}	69.56^{e}
sample 5	0.1415^{f}	2.42^{f}	91^{d}	0.226^{d}	0.470^{e}	42.78^{i}
sample 6	0.1210^{i}	1.80^{j}	96.80^{ab}	0.252^{c}	0.480^{de}	70.32^{d}
sample 7	0.1550^{e}	2.90^{d}	87.40^{f}	0.310^{a}	0.550^{b}	74.09^{c}
sample 8	0.12210^{a}	3.65^{a}	88.40^{e}	0.182^{h}	0.390^{g}	77.65^{a}
sample 9	0.1330^{g}	2.20^{h}	91.20^{d}	0.272^{b}	0.570^{a}	76.87^{b}
sample 10	0.1810^{c}	3.20^{c}	97.40^{a}	0.252^{c}	0.520^{c}	50.05^{h}

Mean values followed by different superscript letters in a column differ significantly (P < 0.05).

the extract used to serve as an indicator for nutrient retention during the spray drying process, was 108.34 mg/l.

Physical characteristics of barberry extract powder

Spray drying is a method which strongly depends on the feed properties and adjusting of drying conditions. The combination of these effects influences the product parameters, including powder temperature, moisture content, particle size, and powder recovery (yield). Optimization of process carried out by a trial-and-error approach is outdated, costly, and time consuming. Masters (1991) indicated that some initial conditions could be found in the application database for equivalent or similar products. The use of existing empirical data such as those proposed by Goula and Adamopoulos (2005) which used in this study is an alternative way to establish the experimental conditions.

The analysis of physical characteristics of powders including water activity, moisture content, solubility, loose and tapped bulk density, and powder recovery were shown in table 2. Water activity (a_w) is an important factor which could give a good indication of the powder shelf life. It has been well known that most of food with a_w less than 0.6 are stable toward microorganisms and biochemical reactions. In this study the a_w for all spray dried powders were between 0.121-0.221, respectively. There was no significant difference between sample 1 and 4 in terms of water activity, and sample 8 had the highest value of a_w (P>0.05). These results are in good agreement with findings of spray dried water melon powders (Quek *et al.*, 2007).

The ratio of MD:AG had no significant effect on the moisture content of the samples (P>0.05). Sample 8 has the maximum moisture which was 3.65%. The highest amount of moisture content was observed in the samples which were dried at air inlet temperature of 160°C. Besides inlet air temperature, the outlet air temperature has an important effect on the powder properties. By decreasing inlet air temperature, the outlet temperature is decreased and because of that the moisture content of powder is increased. The rate of heat transfer to the particle is greater because of higher inlet temperature; and providing greater driving force for moisture evaporation. Consequently, powders with reduced moisture content are formed. The moisture content will have an influence on the keeping quality of the powder (Goula *et al.*, 2004). In this study, the outlet air temperature of the spray dryer varied from 80 to 90˚C.

The percentage of dissolved powder in water was determined by solubility test. Sample 10 had the most amount of solubility which was prepared at 160◦C with the ratio of MD to AG (75:25), but increasing MD and AG or increasing the temperature had no significant effect on the powder solubility (P>0.05). The sample 1 had the minimum solubility which was prepared at 180◦C and just used MD as the carrier. At higher inlet temperature, a hard surface layer might be formed over the powder particles. This could prevent to release of water molecules from diffusing through the particle (Chegeni and Ghobadian, 2005).

Loose and tapped bulk density of the powders is one of the most important parameters which are measured. This is important because of the terms of transportation, warehousing and packaging (Athanasia *et al.*, 2004). The highest value of tapped bulk density was found for sample 9 (0.570 g/cm^3) which had a significant difference (P<0.05) with other samples. The result of the tapped bulk density

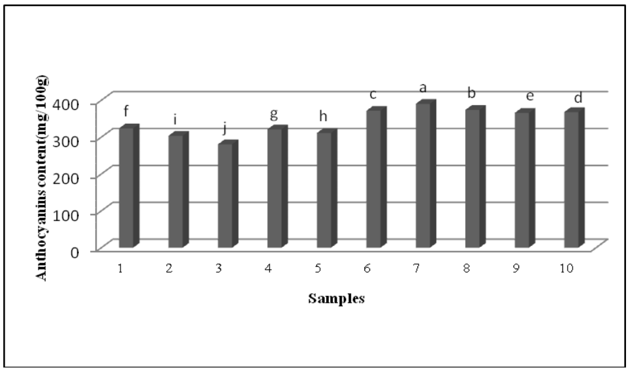

Figure 1. The amount of anthocyanins found in produced powder of 10 samples (1: T=180ºC, MD:AG 100:0, 2: T=180ºC, MD:AG 50:50, 3: T=180ºC, MD:AG 75:25, 4: T=180ºC, MD:AG 0:100, 5: T=180ºC, MD:AG 25:75, 6: T=160ºC, MD:AG 100:0, 7: T=160ºC, MD:AG 50:50, 8: T=160ºC, MD:AG75:25, 9: T=160ºC, MD:AG 0:100, 10: T=160ºC, MD:AG 25:75)

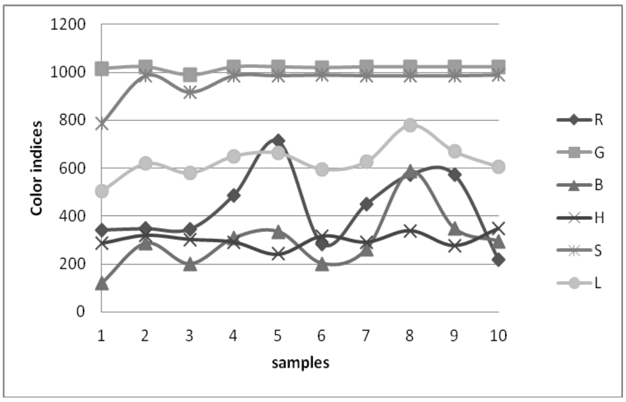

Figure 2. Measured indicators to determination quality and quantity of color samples

of samples 7, 9 and 10 has shown that increasing percentage of AG in MD/AG ratio leads to increase in the tapped bulk density values of samples. The same trend was observed for loose bulk density values. This may be justified by the smaller molecular size of AG than MD and hence obtaining finer particles when the amount of this carrier increased in the ratio of MD/AG.

Powder recovery

Product yield is defined as the ratio of the powder mass recovered from the drying chamber to the mass of TSS in the feed. By referring to table 2, maximum and minimum of the yield of produced powder were corresponded to samples 8 (77.65%) and 5 (42%), respectively. In this study, the temperature had a negative effect on products yield. Fazaeli *et al*. (2012) reported that inlet air temperature had a positive effect on process yield of black mulberry juice powder to 160˚C, which can be attributed to the greater efficiency of heat and mass transfer processes, but Papadakis *et al.* (2006) showed that by increasing the inlet air temperature, the process yield of raisin juice decreased. This phenomenon is due to stickiness problems and it means that drying temperature is above their glass transition temperatures.

The types of the carrier material had no significant effect on the increasing or decreasing of produced powder yield (P>0.05) (table 2). An additive such as MD and AG have been used to induce physical changes in the product, consequently reducing stickiness and wall deposition in spray drying. The addition of these carriers into the feed improves powder recovery (Truong *et al.,* 2005; Shrestha *et al.*, 2007). Other researchers such as Chopda and Barrett (2001) also considered 50% powder recovery in the drying of guava fruit juice to be a successful drying process.

Determination of anthocyanins

Maximum and minimum of the anthocyanins content were found in samples 3 and 7 respectively (Figure 1). The anthocynin contents were reduced with increasing temperature, however, the ratio of the carrier material had no significant effect on the content of anthocyanins (P>0.05). Ersus and Yurdagel (2007) made similar conclusions in the production of encapsulated anthocyanins from extracted juice of black carrot. Tonon and Brabet (2010) investigated the degradation of anthocyanins during storage of the encapsulated powder of Acai (*Euterpe oleracea* Mart.) with different covering materials. They concluded that degradation of anthocyanins followed first-degree equation, rising temperature had the negative effect on the amount of anthocyanins content of the powder, but the kind of coverings had no significant effect on the destruction or preservation of anthocyanins. Negative effect of temperature on the amount of anthocyanins has been proved by some researchers. Higher degradation rate of anthocyanins in the higher temperature could be due to the presence of sugar and protein in the products structure, which ultimately led to the Millard reaction during production or storage (Daravingas and Cain, 1965).

Determination of color

The quality and quantity of color samples were measured that shown in Figure 2. The following figure data and the result of ANOVA table showed that samples produced at 180°C had a less R (red) index. There was decreased in red color when inlet temperature was increased. Change the ratio of carrier agent has no significant effect on R index (P>0.05) samples of 2, 4, 5, 7, 8, 9 and 10 also there were not significant differences about the G index that represents the green. The lowest amount of this

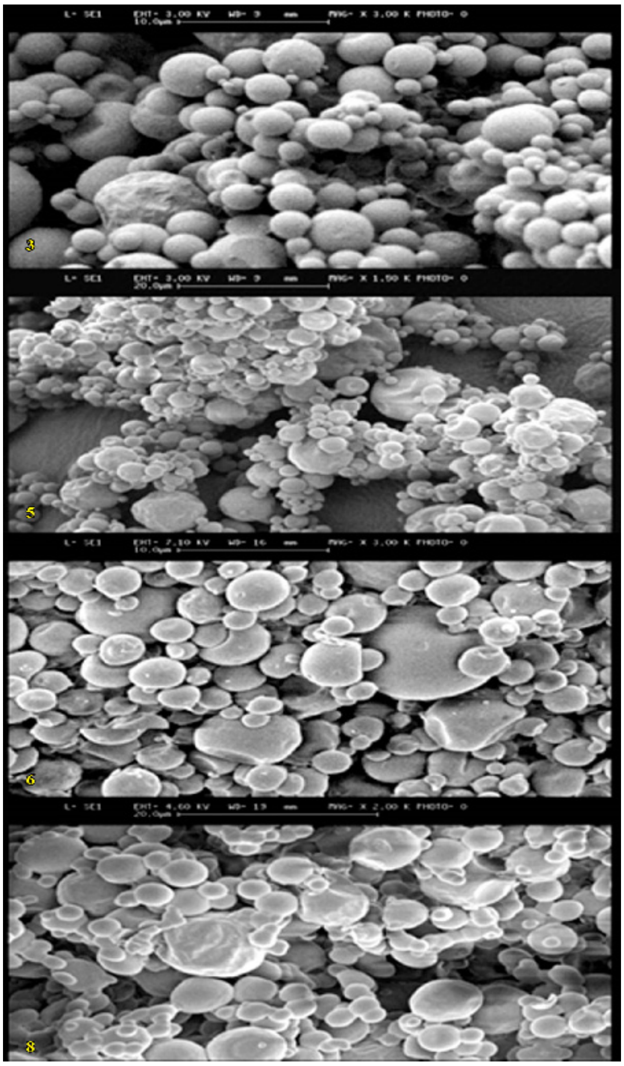

Figure 3. The scanning electron micrographs for barberry extract powder, while different level of MD:AG were used as the drying aid (samples 3,5,6,and 8)

index (G) was observed in sample 1. Maximum and minimum of B (blue) index was seen in 8 and 1 sample, respectively. The result about Hue index showed that the purity and color saturation in samples produced at 160°C is much more than other samples and the result of last index L (luminance) revealed that if the ratio of MD as a carrier agent is greater, this index has increased more. Similar observation was reported in the spray drying of watermelon juice by Quek and others (2007). Other studies also indicate the significant influence of air temperature on variation of color in carrot powder (Chen *et al*., 1995) and tomato products (Shi *et al*., 1999).

Particle size and microstructure

SEM micrographs of particles which were spray dried at 160 and 180°C air inlet temperature with different levels of MD and AG showed that the particle size of powders ranged from 10 to 20 micron approximately (figure 3). All of the samples containing more MD looked like smooth spheres. Largest and smallest particles diameter belong to samples of 1 and 9, respectively. The increasing of air inlet temperature had no significant effect on particles size ($P>0.05$) but different ratio of the carrier material include MD and AG had significant effect on particles size ($P<0.05$). Particles that coated with AG were smaller in size.

Tonon and Brabet (2010) found a wide range of particle size from Acai. Diameters of particles were between 0.1 to 41 microns. Their observations showed particles with Arabic gum had less diameters than maltodextrin and tapioca starch. Maltodextrin with higher DE due to higher degree of hydrolysis and shorter chains produced smaller particles.

Conclusion

A considerable portion of seasonal fresh barberry production in the eastern part of Iran is lost because of the lack of appropriate drying technology. In this study, extract of seedless barberry was successfully dried by spray dryer. The barberry extracted samples were dried using a mixture of MD:AG of various ratios, as carriers. The largest amount of anthocyanins were observed in samples which were produced when the inlet temperature reached at 160°C with same ratio amount of MD and AG (390.46 mg/100g). The anthocyanins content was increased when inlet/outlet temperature decreased. The best product recovery (77.65%) was related to samples which were dried at 160°C and 75:25 ratio of MD and AG. SEM micrographs showed the particle size of powders ranged from 10 to 20 micron. Overall observation of the SEM images indicates that the particle size was increased as the MD concentration increased. The experimental results are promising for scaling up the spray drying of barberry extract for commercial purposes.

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