Effects of Spray-Drying Temperatures and Ratios of Gum Arabic to Microcrystalline Cellulose on Antioxidant and Physical Properties of Mulberry Juice Powder

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Abstract: This study aimed to investigate the influences of inlet air temperatures (120 °C, 130 °C, 140 °C and 150 °C, 160 °C) and the ratios of gum Arabic to microcrystalline cellulose on the quality of mulberry juice powder produced by the spray drying process. Determination of moisture content, bulk density, solubility, total phenolic content (TPC), total anthocyanin content (TAC) and antioxidant capacity (AC) of the obtained powders was performed. The study on temperatures showed that all parameters studied were significantly affected by the shift in temperatures. The powder dried at 160 °C had the highest TPC (38.32 ± 0.36 mg GAE/g DW) and water solubility index (78.70 ± 0.75%); however, it had the lowest moisture content (3.90 ± 0.14%) when compared with the samples dried at other temperatures. Meanwhile, at 140 °C, powder samples with the highest TAC (4.690 ± 0.18 mg cyd-3-glu/g DW) and highest AC (648.09 ± 22.98 μmol TE/g DW), respectively, were obtained. Increasing microcrystalline cellulose and decreasing gum Arabic contents in the carrier mixtures showed significant decreases in moisture content, bulk density, solubility, TPC, TAC and AC.

Keywords: mulberry; spray drying; antioxidant capacity; inlet temperatures; microcrystalline cellulose

1. Introduction

Mulberry is a species of flowering plants in the genus Morus (family Moraceae). The plant has been distributed throughout the world, but is mostly cultivated in warm-temperature regions and the subtropics of Asia, Africa, North America and southern Europe [1]. In some Asian countries such as China and Vietnam, while mulberry leaves are used as a food source for silkworms, the mulberry fruit has been used as traditional folk medicine for the treatment of fever, anemia, sore throat, hypertension, liver or kidney damage, anti-inflammatory effects, anti-aging agents [2–4]. There has been great interest in determining the promising health benefits of mulberry that are primarily due to its polyphenol content.

Several studies have proven the presence of many phenolic compounds in mulberries, such as carotenoids, flavonoids [5] and anthocyanins. According to a study done by Suh et al. [6], mulberry, especially black mulberry, is a concentrated source of anthocyanins—water-soluble pigments that are responsible for the red, blue and purple color of fruit and vegetables. They represent a class of phenolic compounds with potential anti-oxidative effects. Various studies on red-colored juices such as pomegranates, different berries and grapes have proved their beneficial effects on human health, as they are high in anthocyanin content and antioxidant activity [7]. The correlation between anthocyanins and the reduced risk of coronary heart disease, stroke, certain types of cancers and

aging has also been published in epidemiological studies [8,9]. The bioactive compounds, especially anthocyanin, and functional activities of black mulberry have recently been of interest.

Though mulberry is a promising functional food for humans, it is only available in season. Fresh mulberry is also high in moisture content and sensitive to the surrounding environment (i.e., temperature and light), which shortens its shelf life. To utilize the benefits of mulberry at its best, reducing its moisture content would be a promising approach for preserving mulberry. Dehydration not only enhances the shelf-life of mulberry, but also makes it available throughout the year, providing a variety of mulberry products that can be applied extensively in the food industry, such as colorants, flavorants, and antioxidant supplements.

Spray drying has been proven to be one of the most effective drying methods, especially in the food industry, for its high yield, lower price and the reduction of thermal exposure time, which reduces thermal damage of final products. A variety of spray-dried fruit juice has been studied, leading to successful applications in colorant production from fruits and vegetables such as berries, pomegranate, Gac fruit, purple sweet potato, black carrot [10–16]. However, spray-drying of fruit juice can be associated with some problems such as stickiness, hygroscopicity and solubility due to the presence of low molecular weight sugars and acids, which results in a low glass transition temperature (T_g) [17]. Carrier agents are used in spray-drying as drying aids to increase the overall glass transition temperature (T_g), product yield, overcome stickiness, and agglutination, and also to protect heat-sensitive compounds (i.e., pigments, antioxidants) against unfavorable ambient conditions, and reduce the volatility of flavors and aromas by microencapsulation.

Polymers and gums such as maltodextrins, gum Arabic, gelatin, starches, pectin, methyl cellulose and their combinations are common carrier agents used in the food industry [18,19]. Maltodextrins are widely used in spray drying, as they are high in molecular weight and glass transition temperature, (T_g), soluble in cold water with low viscosity, and prevent stickiness and wall deposition. However, spray drying with the aid of maltodextrins alone is not efficient, particularly for the preservation of sensitive compounds, as compared to other carriers, due to their low film-forming ability [20]. Gum Arabic is said to have strong film forming ability, but a low glass transition temperature. The combination of maltodextrins and gum Arabic was proved to significantly improve the final quality of spray-dried powder. However, maltodextrin still has an amorphous nature; therefore, when exposed to relatively high humidity, spray-dried powders containing maltodextrin have the likelihood of becoming sticky and caking. Incorporation of crystalline carbohydrates in spray-dried fruit juice powders such as microcrystalline cellulose (MCC) is said to produce a partially crystalline surface, increasing their crystallinity and thus enhancing stability and reducing stickiness [21].

Inlet air temperature, known as drying temperature, is also one of the most important factors affecting the physical properties, as well as the nutritional quality, of spray-dried powder. It was found that at a constant feed flow rate, the higher the inlet air temperature, the faster the drying rate, leading to changes in the moisture content, particle size, bulk density, flowability and solubility of final products. The decrease in moisture was observed in different spray-dried powders, including Gac fruit [22], watermelon juice [23], tomato juice [24], acai juice [14,15] and pineapple juice [25]. Solubility is also an important quality property of powdered products, and can directly influence the reconstitution behavior of the spray-dried powder. The solubility of cagaita powders increased with rising of spray drying temperature from 120 °C to 160 °C [26].

The levels of antioxidants in processed juice might be lower compared to fresh ones due to maceration, heating and various separation steps, which result in oxidation, thermal degradation, and leaching of antioxidants in plant tissues [27]. Despite the damage to phytochemicals during spray-drying, recent studies have shown that the antioxidant levels of some processed food, such as tomato [28] and sour cherry [29], are maintained by the high recovery of the compounds during processing, or might even be enhanced by the appearance of new compounds, which have a higher antioxidant capacity [30]. However, very little information on the utilization of microcrystalline cellulose in spray-dried mulberry powder has been published.

Based on these reasons, the present study aimed to investigate the effects of spray-drying temperature and the ratio of Gum Arabic to microcrystalline cellulose on the physiochemical and antioxidant properties of mulberry juice powder.

2. Materials and Methods

2.1. Materials

Fresh mature black mulberries (*Morus nigra*) were collected from Lam Dong province, Vietnam and transported to the International University laboratory. The fruits were homogeneously and carefully sorted in term of ripeness and wholesomeness and stored immediately in the freezer at $-20\,^\circ$C prior to the experiments.

2.2. Sample Preparation

Frozen mulberries were thawed overnight at 4 $^\circ$C before mashing for 60 s using a blender (Panasonic MX-GM1011GRA, Berhad, Malaysia). Mulberry must was then incubated with Pectinex Ultra SP-L (0.2% *v/w*) (Novozymes, Bagsvaerd, Denmark), at 50 $^\circ$C for 120 min [31]. After the treatment, mulberry juice was obtained by filtration with filter papers with size pore of 20–25 μm. To determine the total solid content of mulberry juice, dry 5 mL juice at 80 $^\circ$C for 24 h using an oven (WiseVen, Wisd Laboratory Instruments, Gangwon-do, Korea). Carrier solutions were separately prepared by dispersing Maltodextrin (MD), Gum Arabic (GA) and Microcrystalline Cellulose (MCC) (Merck KGaA, Darmstadt, Germany) into 50 $^\circ$C distilled water following the ratios in Table 1. The carrier solutions were mixed with mulberry juice to make feed solutions of 30% (*w/w*) carrier agents in dry basis prior to spray drying.

2.3. Spray-Drying Conditions for Mulberry Juice

The feed solutions containing carrier agents (MD, GA and MCC) were spray-dried using a Lab Plant SD 06 spray-dryer (Lab Plant Ltd., Keison, Chelmsford, UK) at 8 rpm. At the end of the process, spray-dried mulberry powder was collected and transferred into dark vials with caps enclosed. The vials were sealed and stored in the freezer at $-20\,^\circ$C prior to analysis.

2.4. Experimental Methods

2.4.1. Study on Effects of Temperatures

To investigate the influences of different temperatures on the quality of spray-dried mulberry juice powder, the mulberry juice was mixed with maltodextrin and gum Arabic at the ratio of 7:3 (*w/w*) [32] and then spray-dried with the constant feed flow rate of 8 rpm at different temperatures (120 $^\circ$C, 130 $^\circ$C, 140 $^\circ$C and 150 $^\circ$C, 160 $^\circ$C), as described by Fazaeli et al. [11] with slight modification.

2.4.2. Study on Effects of Gum Arabic/Microcrystalline Cellulose Ratios

To investigate the influences of the carrier ratios on the final quality of spray-dried mulberry powder, the mulberry juice was mixed at different ratios of gum Arabic to microcrystalline cellulose with maltodextrin concentration was fixed at 21% (Table 1). Then, the mixtures were spray-dried at 8 rpm and 140 $^\circ$C, chosen from the previous experiment.

Table 1. Concentrations of carrier agents used the mixture.

% Maltodextrin	% Gum Arabic	% Microcrystalline Cellulose	Overall Concentration (%, *w/w*)
21	9	0	30
21	8.5	0.5	30
21	8	1	30
21	7.5	1.5	30
21	7	2	30

2.5. Analytical Methods

2.5.1. Determination of Moisture Content

The moisture content of spray-dried mulberry powder was determined based on AOAC 2002 [33]. In detail, 0.1 g of each mulberry powder sample was weighed and placed in an air oven at 70 °C until a constant weight.

$$\text{Moisture content }(\%) = \frac{initial\ weight - final\ weight}{weight\ of\ sample} \times 100 \tag{1}$$

2.5.2. Determination of Bulk Density

Bulk density (g/mL) is defined as the total mass of powder divided by the volume it occupies. It was determined using the method of Barbosa-Cánovas et al. [34] with slight modification. Gently, 0.5 g mulberry powder was poured into a 5 mL graduated cylinder. The sample was then tapped repeatedly 10 times at a vertical distance of 10 mm, and the volume of the weighed sample was recorded. The measurement was done in ambient temperature and in triplicates.

$$\text{Bulk density }(\text{g/mL}) = \frac{weight\ of\ sample\ at\ recorded\ volume\ (g)}{volume\ of\ sample\ (mL)} \tag{2}$$

2.5.3. Determination of Water Solubility Index (WSI)

Water solubility index of mulberry powder was determined using the procedure described by Anderson et al. [35] with modifications. Briefly, 0.1 g of each sample was dispersed in 1 mL distilled water at ambient temperature and stirred in a vortex for a minute. After that, the solution was placed in a water bath of 37 °C for 30 min, and then centrifuged at 9500 rpm for 10 min (Z326K, Wehingen, Germany). The supernatant was placed in aluminum foil and dried in the oven at 105 °C until it reached constant weight. The WSI was calculated based on weight difference, and was expressed as percentage.

$$\text{Water solubility index }(\%) = \frac{Dried\ supernatant\ weight}{Initial\ sample\ weight} \times 100 \tag{3}$$

2.5.4. Extraction of Total Phenolic Content

The extraction procedure was modified from a method of Vinson et al. [36]. Briefly, 0.1 g of each spray-dried sample was weighed into a 50 mL falcon, followed by the addition of 10 mL of extraction solvent, included 1.2 M HCl in 50% Methanol (*v/v*) (Merck, Darmstadt, Germany). The mixtures of sample and solvent were incubated for 30 min at ambient temperature using the shaking incubator at 200 rpm. The extracts were centrifuge at 10,000× *g* at 4 °C for 10 min. All samples were extracted twice. The supernatants were collected and transferred into dark vials with caps and stored at −20 °C prior to analysis.

2.5.5. Extraction of Total Anthocyanin Content (TAC)

The procedure for extraction of TAC followed the method of Liu et al. [37] with modifications. In detail, spray-dried powder samples (0.1 g) were transferred to 15 mL-falcon with caps, and then mixed with solvent containing 0.1% HCl in 60% Ethanol (*v/v*) (Merck, Darmstadt, Germany) (pH ~ 1) to give a liquid:solid ratio of 15.7:1 (*v/w*). The mixtures were incubated in a shaking incubator at 65 °C for 114 min, and then centrifuged at 5000 rpm for 10 min at 25 °C. The supernatants were collected and stored in dark vials with caps enclosed at 4 °C prior to analysis.

2.5.6. Determination of Total Phenolic Content (TPC)

Total phenolic content was determined using Folin-Ciocalteu assay as described by Singleton et al. [38] with some modifications. In detail, 0.5 mL aliquot of diluted extract was mixed with 2.5 mL of Folin-Ciocalteu reagent (0.2 N) (Sigma-Aldrich, Steinheim, Germany) and incubated for 8 min, then 2 mL of Na_2CO_3 7.5% (Merck, Darmstadt, Germany) was added. The absorbance was measured at 760 nm using a UV-Visible spectrophotometer (Genesys 10 S UV-Vis, Madison, WI, USA) after 30 min of incubation at ambient temperature (25 °C). Gallic acid (0–50 µg/mL) (Sigma-Aldrich, Steinheim, Germany) was used to conduct standard curves. The obtained results are expressed as milligram of Gallic acid equivalent per g dry weight (mg GAE/g DW).

2.5.7. DPPH Free Radical Scavenging Assay (AC)

To determine to antioxidant capacity of mulberry powder, a measurement of the DPPH (1,1-diphenyl-2-picrylhydrazyl) (Sigma-Aldrich, Steinheim, Germany) radical-scavenging potential reported by De Souza et al. [39] was carried out. Firstly, DPPH was prepared by dissolving 3.94 mg of DPPH in 100 mL of methanol to make 0.1 mM methanolic DPPH solution. Then, 2 mL of 0.1 mM methanolic DPPH solution was added to 2 mL of diluted extract, followed by incubation in the dark for 30 min. The absorbance was then measured using UV-Visible spectrophotometer (Genesys 10SS UV-Vis, Madison, WI, USA) at the wavelength of 517 nm. A standard curve was conducted by Trolox (6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid) (Sigma-Aldrich, St. Louis, MO, USA). The results achieved were displayed as µmol Trolox equivalent (TE)/g dry weight (DW).

2.5.8. Determination of Total Anthocyanin Content (TAC)

Total anthocyanin content was determined by the pH differential method [3] which involved the measurement of absorbance at pH 1 and pH 4.5, using KCl (0.025 M) and CH_3COONa (0.4 M), respectively, as buffers. The absorbance was then measured at 510 nm (γ_{max}) and 700 nm using UV-Visible spectrophotometer (Genesys 10SS UV-Vis, Madison, WI, USA) against a blank with distilled water. Absorbance (A) is calculated using equation:

$$A = (A_{510} - A_{700})_{pH1.0} - (A_{510} - A_{700})_{pH4.5} \tag{4}$$

The total anthocyanin content (TAC) is calculated following the equation below:

$$AC = \frac{(A \times M_w)}{\varepsilon \times 1} \times D_f \times 1000 \tag{5}$$

In which:

- M_w: relative molecular mass of cyanidin-3-glucoside (cyd-3-glu) (449.38 g/mol)
- ε: molar absorptivity of cyanidin-3-glucoside (cyd-3-glu) (26,900 L/mol·cm)
- D_f: dilution factor of the extracts
- AC: anthocyanin content of mulberry powder is expressed as mg cyaniding-3-glucoside (cyd-3-glu) equivalent/g DW.

2.6. Statistical Analysis

One-way ANOVA was performed using SPSS version 20 (IBM) (Armonk, NY, USA) to determine significant differences between the variables being investigated. Significant differences were accepted at $p < 0.05$. All the experiments were done in three replicates and data is reported as mean ± standard deviation.

3. Results and Discussion

3.1. Effects of Spray-Drying Temperatures on Physiochemical Properties of Instant Mulberry Powder

3.1.1. Moisture Content

Table 2 shows the significant influence of different inlet temperatures on the moisture content of mulberry powder. For spray-drying in general, increasing inlet temperatures leads to greater loss of water in the powder. It is verified, with the help of Table 2, that the moisture content of mulberry powders spray-dried at increasing inlet air temperatures significantly dropped from 5.15% to 3.90%. It can be concluded that the higher the inlet air temperature, the higher the rate of heat transfer to the particles, providing greater driving force for water evaporation, thus forming powders with low moisture content [15]. It was also reported by Kha et al. [22] that the moisture content of Gac fruit microspheres decreased remarkably, from 5.29% to 3.88%, with an increase in temperature from 120 °C to 200 °C. A similar observation was obtained in the different fruit juice powders of which moisture contents had a declining tendency with the increase of temperatures such as 2.78% to 1.47% in watermelon juice [23], 12.41% to 2.91% in tomato juice [24], 2.89% to 0.64% in acai juice [14,15].

Table 2. Effects of spray-drying temperatures on physiochemical and antioxidant properties of instant mulberry juice powder.

Inlet Temperatures (°C)	Moisture Content (%)	Bulk Density (g/mL)	Water Solubility Index (%)	Total Phenolic Content (mg GAE/g DW)	Total Anthocyanin Content (mg cyd-3-glu)/g DW)	Antioxidant Capacity (μmol TE/g DW)
120	5.15 ± 0.13 [a]	0.49 ± 0.02 [a]	65.81 ± 0.7 [°C]	36.99 ± 1.07 [ab]	3.61 ± 0.11 [bc]	587.10 ± 3.08 [b]
130	4.78 ± 0.16 [b]	0.46 ± 0.02 [ab]	70.05 ± 0.91 [b]	35.90 ± 1.22 [abc]	3.44 ± 0.07 [c]	642.93 ± 5.92 [a]
140	4.54 ± 0.10 [b]	0.45 ± 0.02 [ab]	71.58 ± 0.56 [b]	35.69 ± 1.14 [bc]	4.69 ± 0.18 [a]	648.09 ± 22.98 [a]
150	4.06 ± 0.18 [c]	0.42 ± 0.01 [b]	77.23 ± 0.62 [a]	34.35 ± 0.98 [c]	3.80 ± 0.20 [b]	583.66 ± 3.27 [b]
160	3.90 ± 0.14 [c]	0.41 ± 0.03 [b]	78.70 ± 0.75 [a]	38.32 ± 0.36 [a]	3.53 ± 0.14 [bc]	593.63 ± 20.91 [b]

Note: The values followed by different lowercase letters (a–c) within a column are significantly different at $p \leq 0.05$.

3.1.2. Bulk Density

It can be seen from Table 2 that bulk density of mulberry powder varied from 0.49 g/mL to 0.41 g/mL as the inlet air temperatures went up from 120 °C to 160 °C. Mulberry powder particles showed a decreasing tendency in tapped bulk density when temperatures increased. In detail, the powder sample with the highest bulk density value of 0.49 g/mL was spray-dried at the lowest temperature, 120 °C. As the temperature reached 160 °C, the lowest value for bulk density was obtained, 0.41 g/mL. As explained by Walton [40] and Chegini et al. [41], with the increase in temperature, the water evaporation rate accelerates, forming large particles with the tendency of being hollow or porous, leading to the decrease in bulk density. Moreover, temperature also has an influence on the bulk density of spray-dried powders with respect to the amount of moisture they carry. Chegini et al. [41] reported that powder with higher moisture content tends to exhibit higher bulking weight. The relationship between bulk density and inlet air temperatures in this present study has been recorded as negatively correlated, and this is in agreement with previous studies. Fazaeli et al. [11] reported that bulk density values of mulberry powder varied from 0.35 to 0.55 g/mL as the temperatures increased from 110 °C to 150 °C. Also, in another study [22], it was found that the bulk density of spray-dried Gac powder dropped considerably from 0.78 to 0.66 g/mL with an increase in temperature from 120 °C to 200 °C. It is desirable for products to obtain high bulk density values, as they require less volume in containers. Moreover, high bulk density products tend to occupy more space, and thus there is less space among the particles, preventing oxidation during storage [38].

3.1.3. Water Solubility Index

In this current study, inlet air temperatures showed a significant influence on the WSI of the mulberry powder (Table 2). The WSI of encapsulated mulberry powder ranged from 65.81 to 78.70%, exhibiting an upward trend as the temperatures increased. The highest WSI value was recorded as 78.70% at 160 °C, and the lowest WSI was 65.81% when spray-dried at 120 °C. It was reported in [11] that there was an inverse relation between bulk density and solubility of spray-dried powders, meaning that as the inlet air temperatures rise, product has lower bulking weight, and its solubility tends to decrease. It can be concluded that under high temperatures, the drying rate is accelerated, implying that fewer shrinkage droplets, and more porous and bigger particles with fragmented structures are formed [40]. Large particles may sink to the bottom, whereas smaller particles tend to float on the surface of water, resulting in uneven reconstitution. The obtained results are consistent with previous studies on the spray-drying of fruit juice. In a study on spray-dried tomato powder, Sousa et al. [42] reported that the WSI of tomato powder slightly increased from 17.65% to 26.3% while another study also showed a rise in WSI values from 36.91% to 38.25% in Gac powder [22].

3.1.4. Total Phenolic Content

The total phenolic content of mulberry powder at various spray-drying temperatures is illustrated in Table 2. The results reveal that the drying temperatures did have some influences on TPC of mulberry powder. It is clear from the data that as the inlet air temperatures rose from 120 °C to 150 °C, TPC reduced from 36.99 mg GAE/g DW to 34.35 mg GAE/g DW (Table 2). In general, phenolic compounds have been reported to be highly susceptible to temperature [43,44]; thus, thermal processing could result in thermal, oxidative degradation and significant loss of natural antioxidants [45]. Similar results were observed in spray-dried amla juice powder [46], spray-dried blackberry powder [47]. However, it is interesting to note that there was a reverse trend in the amount of total phenolic compounds above the temperature of 160 °C. When the inlet air temperature reached 160 °C, the TPC significantly ($p < 0.05$) increased from 34.35 mg GAE/g DW to 38.32 mg GAE/g DW (Table 2). The result obtained from the current study is in agreement with Madrau et al. [48], Ahmed et al. [49] and Chong et al. [50] in their work on the spray drying of apricot juice, purple sweet potato pulp, and sapodilla powder, respectively. It was explained by Demarchi et al. [51] that the higher the temperatures, the faster the drying process, and thus a shorter heat exposure time and less heat degradation for heat-sensitive compounds. Moreover, the increase in TPC with the increase of temperature could be due to the polymerization and releasing polyphenols from bound form while the temperatures were elevated up to 200 °C [46].

3.1.5. Total Anthocyanin Content

The content of anthocyanin in spray-dried mulberry powder is illustrated in Table 2. The results show that the inlet air temperatures had no significant influence on TAC of the powders spray-dried at 120 °C, 130 °C, 150 °C and 160 °C. However, it can be clearly observed that TAC of mulberry powder reached the highest value of 4.69 mg/g DW at 140 °C, followed by a remarkable decline to 3.80 mg/g DW and 3.53 mg/g DW at 150 °C and 160 °C, respectively. It was reported that by increasing inlet air temperatures, the evaporation process was accelerated; a smooth and more stable wall material was formed, acting as a matrix which protected anthocyanin core from thermal processing. However, as the temperatures rose too high, TAC was significantly decreased due to its thermos sensitivity. The negative relation between TAC and inlet air temperatures has been proved by some researchers. It was reported by Tonon et al. [14,15] that inlet air temperatures significantly affected TAC of acai powder. A similar observation was reported by Ersus et al. [52] during the microencapsulation of anthocyanins extracted from black carrots using three different inlet air temperatures (160 °C, 180 °C, and 200 °C). Powders spray-dried at high temperatures tend to obtain low bulk density with large particle size, containing more space among the particles, which makes it susceptible to oxidation [53].

3.1.6. DPPH Free Radical Scavenging Activity

The free radical scavenging activity of spray-dried mulberry powder was determined by DPPH assay, and the results are shown in Table 2, with values ranging from 583.66 ± 3.27 to 648.09 ± 22.98 µmol TE/g DW. As can be seen from the Table, there is a significant increase in term of DPPH scavenging activity when the temperatures rose from 120 °C to 140 °C. This could be due to a significant increasing of TAC (Table 2). However, the activity was significantly reduced from 140 °C to 150 °C (Table 2). Similar observations from various studies have been reported such as spray-dried Gac fruit powder [22], spray-dried amla juice powder [46], and spray-dried blackberry [54]. The possible explanation for this phenomenon could be due to the exposure of thermally sensitive compounds to high heat, adversely affecting the structure of phenolic compounds, causing structural breakage, forming different compounds, and thus reducing the antioxidant activity [46].

3.2. Effects of the Ratios of Gum Arabic to Microcrystalline Cellulose on the Quality of Mulberry Powder

3.2.1. Moisture Content

Table 3 presents the moisture content (%) of spray-dried mulberry powder at various ratios of GA to MCC. As seen, the addition of MCC in the feed solutions showed a considerable effect on the moisture content of mulberry powder. Reducing GA concentrations from 9 to 7% and increasing MCC concentrations from 0% to 2% resulted in significant reductions of moisture contents ($p < 0.05$) in the final products, from 4.54–3.20%. The lowest moisture content was observed for the formulation containing 7.5% GA and 1.5% MCC. However, adding more MCC (2%) showed no significant difference in moisture as compared to 1.5%. The phenomenon could be explained by the transformation of particle configuration with the presence of MCC. The introduction of MCC to the carrier formulation produced finer powder with semi-crystalline structure and little to no space among powder particles [55].

Table 3. Effects of microcrystalline cellulose concentrations on physiochemical properties of instant mulberry powder.

% GA (w/w)	% MCC (w/w)	Moisture Content (%)	Bulk Density (g/mL)	Water Solubility Index (%)	Total Phenolic Content (mg GAE/g DW)	Toal Anthocyanin Content (mg cyd-3-glu/g DW)	Antioxidant Capacity (µmol TE/g DW)
9	0	4.54 ± 0.1 [a]	0.49 ± 0.02 [a]	71.58 ± 0.56 [a]	35.69 ± 1.14 [a]	4.68 ± 0.05 [a]	504.89 ± 1.61 [e]
8.5	0.5	3.85 ± 0.07 [b]	0.40 ± 0.02 [b]	68.65 ± 0.29 [b]	35.28 ± 1.05 [a]	3.41 ± 0.04 [b]	609.64 ± 1.33 [a]
8.0	1	3.42 ± 0.07 [c]	0.35 ± 0.01 [bc]	66.68 ± 0.58 [b]	35.83 ± 1.14 [a]	3.56 ± 0.07 [b]	586.47 ± 3.41 [c]
7.5	1.5	3.12 ± 0.06 [d]	0.32 ± 0.02 [c]	62.78 ± 0.53 [c]	32.42 ± 0.77 [b]	3.30 ± 0.07 [bc]	591.73 ± 2.94 [b]
7	2	3.20 ± 0.05 [d]	0.31 ± 0.02 [c]	60.07 ± 0.26 [c]	31.36 ± 0.53 [b]	3.19 ± 0.05 [c]	554.02 ± 2.88 [d]

Note: The values followed by different lowercase letters (a–c) within a column are significantly different at $p \leq 0.05$.

3.2.2. Bulk Density

Bulk density presents the mass of the solid particles and moisture divided by the volume they occupy. As shown in Table 3, there is an adverse relationship between bulk density and the addition of MCC as bulk density had a tendency of reduction, from 0.45 g/mL to 0.31 g/mL, while MCC concentration increased from 0% to 2% (*w/w*). It has been reported in previous studies that using cellulose as an assisting carrier during spray-drying resulted in remarkable effects on bulk density of the final products due to the change in configuration of particles. According to Cano-Chauca et al. [21], it could be confirmed that the addition of cellulose as carrier aid was essential to the induction of crystallization; thus, branched, finer, semi-crystalline structured powder with lower bulk density was produced. However, the addition of more MCC (2%) showed no considerable shift in term of bulk density. This might be due to the formation of more packed powder in the measurement cylinder as cellulose occupied the spaces between the branches, and thus less internal space [56].

3.2.3. Water Solubility Index

Table 3 shows the WSI of spray-dried mulberry powder. When treated with different ratios of GA to MCC, the WSI of mulberry powder significantly decreased from 71.58% to 60.07%. The highest WSI value was obtained with no MCC added; meanwhile, the lowest value for WSI was obtained at the highest concentration of MCC (2%). The former powder sample was only spray-dried with the aid of MD and GA, which are both known for their amorphous nature, thus resulting in high WSI. Some reported literature indicated that the increase in solubility is in relation to a high degree of amorphous surfaces. Generally, amorphous forms are said to have higher thermodynamic quantities, thus they require less energy to dissolve, resulting in higher solubility. The more MCC incorporated in the carrier solutions, the lower the solubility of the final products as the microstructure of the powder was altered. Spray-dried powders with the presence of cellulose tend to have more crystalline, packed structure; therefore; lower solubility. As the MCC concentration decreased, more GA was added, and higher WSI values were obtained. Gum Arabic, together with Maltodextrin, is known for its great ramifications for hydrophilic groups, resulting in higher water adsorption, and thus higher in WSI [55]. Similar results were observed in spray-dried mango powder [6] and pomegranate juice powder [56].

3.2.4. Total Phenolic Content

There was an insignificant difference in total phenol contents between samples mixed with 0, 0.5% and 1% MCC (Table 3). It might be concluded that as MCC was added, the polar carboxyl group could be absorbed on to the surfaces of droplets and acted as a protective coat from the degradation of phenolic compounds. However, when the concentration of MCC increased from 1% to 2%, TPC significantly dropped from 35.83 mg GAE/g DP to 31.36 mg GAE/g DP. Generally, it was reported that the increase in MCC could lead to the decrease in TPC due to the high polymerization ability of MCC, thus resulting in the changes in structure and configuration, producing microencapsulated particles that were lacking in coverage and were more prone to being oxidized [55].

3.2.5. Total Anthocyanin Content

As anthocyanins are thermosensitive pigments, the retention of anthocyanins has been reported to be significantly affected by temperatures; therefore, to investigate the ratios of GA to MCC on TAC, a constant spray-drying temperature (140 °C) was applied for all experiments. The variation of TAC, therefore, was related to the changing ratios of GA to MCC. Overall, the interaction between investigated carriers and TAC was found to be significant ($p < 0.05$). As the concentration of MCC increased from 0.5% to 2% and GA decreased from 9% to 7%, TACs decreased from 355.95 mg/100 g to 319.72 mg/100 g powder. According to Yousefi et al. [55], high cellulose concentrations were found to be unsuitable to encapsulate anthocyanins in spray-dried fruit juice due to the structural changes in microstructure of particles. As stated above, with the addition of more MCC, the microencapsulated particles become more scattered, with many dents on the surface, making them more prone to oxidation. At lower cellulose concentrations, more GA was added in order to make a 30% (*w/w*) carrier solution. GA has been reported for its emulsifying capacity, thus forming a protective layer around the anthocyanin core. Therefore, the higher the GA percentage added, the higher the TAC that remained.

3.2.6. DPPH Free Radical Scavenging Activity of the Mulberry Powder

The ability to scavenge free radicals of mulberry powder spray-dried with the different ratios is illustrated in Table 3. The ability of the powder was found to significantly decrease ($p < 0.05$) with increasing the MCC concentration from 1.0 to 2.0%. The highest value for the DPPH scavenging activity was obtained with 0.5% MCC added. This phenomenon could be explained based on the fact that when MD and GA with high solubility were mixed with stock solution and spray-dried, it tended to form a matrix that could entrap bioactive compounds, thus providing complete encapsulation [14]. As more cellulose was added, there were significant decreases of antioxidant capacity, from 609.64

± 1.33 to 554.02 ± 2.88 μmol TE/g DW. The reduction of the scavenging activity during the drying process might be due to the oxidative reactions as TPC and anthocyanins were more prone to oxidation at higher concentrations of cellulose due to the lack of protective layers. In addition, due to the insoluble property of MCC, the mulberry powder produced contained the extract and MCC particles separately; hence, there was less protection on bioactive compounds.

4. Conclusions

This study was performed at different inlet air temperatures and different ratios of GA to MCC for producing the instant mulberry juice powder. In general, the increase in temperatures led to the decrease in moisture content, bulk density, TPC, TAC, and DPPH scavenging activity; while there was an improvement in water solubility index of spray-dried powder. It was observed in the present study that thermal processing resulted in increasing of TPC. Mulberry powder spray-dried at 160 °C retained the highest total phenolic compounds; however, the retention of anthocyanins and antioxidant activity were significantly lower than the powder spray-dried at 140 °C. As the juice was spray-dried at constant inlet air temperature and feed flow rate, the increased addition of MCC and reduction of GA were recorded to have significant decreases of moisture content, bulk density, water solubility and antioxidant properties of the mulberry juice powder.

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