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Inlet Temperature Affects Spray Drying Quality of Watermelon Powder

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Abstract

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The effect of the inlet temperature on the quality of watermelon powder after spray drying was evaluated. Inlet temperatures of the drying air of 120, 130, 140, and 150°C maintained water solubility of the watermelon powder at 96%. At 253 μM/g, the ORAC value of the watermelon powder dried at 130°C was the highest among all tested powders. The D_{50} of the watermelon powder dried at 130°C was 18.21 ± 0.22 μm with a span of 1.73 ± 0.038, which was more uniform than that of other powders. The crystallinity of the powder dried at 130°C was higher than that dried at both 120 and 150°C and showed stronger thermal stability. Moreover, watermelon powder dried at 130°C presented a similar aroma as the fresh watermelon juice when being solved. Hence, an inlet temperature of the drying air of 130°C was the optimal temperature for the production of watermelon powder.

Keywords: aroma; crystallinity; inlet temperature; ORAC; radical scavenging capacity; solubility; water activity

Watermelon (*Citrullus lanatus*) juice is an excellent source of vitamins, mineral salts, specific amino acids (e.g., arginine and citrulline), and a large variety of antioxidants (e.g., phenolics and lycopene) (Romdhane *et al.* 2017). Consumption of watermelon juice protects the chemical-induced hepatotoxicity in rats (Altaş *et al.* 2011), increases the plasma concentrations of β-carotene in humans (Edwards *et al.* 2003), and increases the antiproliferative activity on in cell lines of both liver cancer and human breast cancer (Rahmat *et al.* 2002). However, watermelon juice is sensitive to both heat and oxygen (Aguiló-Aguayo *et al.* 2010; Zhang *et al.* 2011; Feng *et al.* 2013; Liu *et al.* 2014). Spray drying is a highly appropriate technique to conserve heat- and oxygen-sensitive fruits. The technique involves atomization of the solution in a hot gas current to form a powder product in a short period. Except for maintaining the colour and aroma, the powders have great economic potential over their

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liquid counterparts such as reduced weight, reduced packaging, and extended shelf life (Tuyen *et al.* 2010; Mishra *et al.* 2014). The spray drying technique is suitable for the production of amla powder (Mishra *et al.* 2014), pear powder (Rodríguez-Hernández *et al.* 2005), black mulberry powder (Fazaeli *et al.* 2012), and tomato powder (De Sousai *et al.* 2008). The inlet temperature of the drying air is the key factor that affects the quality of the obtained powders (Rodríguez-Hernández *et al.* 2005; de Sousai *et al.* 2008; Fazaeli *et al.* 2012).

Watermelon powder has potential applications as colorant, flavouring, and ingredient in the food industry, as well as in the production of lycopene-rich capsules with functional and nutritional appeal. However, the effect of the inlet temperature on the quality of watermelon powder has not been evaluated to date. Hence, inlet temperatures varied from 120 to 150°C were applied to dry the watermelon juice. The quality of the resulting powders, including moisture content, water activity, bulk density, water solubility, repose angle, particle size, microstructure, crystallinity, antioxidant capacitates, and aroma were investigated and compared.

MATERIAL AND METHODS

Materials required. Mature watermelons (*Citrullus lanatus;* var. Jingxin No.3) were brought from a local fruit market. The fruits were round with regular stripes and weighted about 3–4 kg per piece. The flesh of the fruits was red and crisp with a soluble solid content of 11.5–13.5%. The maltodextrin with the dextrose equivalent of 15.6% was brought from Deqing Sanfu Food Co. Ltd. (China).

Preparation of watermelon juice. The watermelons were stored at 9°C for 24 hours. The cold fruits were peeled and beaten in a universal food processor (Model UMC5; Stephan Machinery Corp., USA) with a pressure of 0.01 MPa. The resultant juice was filtered by three layer gauze. The juice of 5000 ml was mixed with the maltodextrin of 300 g. The mixture was homogenized at 50 MPa (NS101L2K; GEA Niro Soavi S.p.a., Italy).

Spray drying of watermelon juice. The homogenized juice was atomized by a mini spray dryer (B-290; BUCHI Labortechnik AG, Switzerland). The inlet temperature of the drying air was set at 120, 130, 140, and 150°C when the outlet temperature was 85°C by adjusting the flow rate of the air and injection of the juice. The watermelon powder was collected and sealed in an aluminum foil bag and stored at 4°C.

Powder analysis/characterization. The moisture content of the sample was measured by heating the sample for at least 6 h at 105°C until reaching a constant weight.

The water activity was measured by a water activity meter (Aqua LAB 4TE; Decagon Devices, USA) with the standard solution of 0.25 and 0.50 as the control samples.

Bulk density was determined by gently adding 2 g of sample into an empty 10 ml graduated cylinder and holding the cylinder on a vortex vibrator for 1 minute. The ratio of mass of the powder and the volume occupied in the cylinder determined the bulk density value.

Colour was measured in the reflectance mode bya spectrophotometer (CM3700d; Konica Minolta Sensing INC., Japan) for at least 6 times at 25°C and averaged.

Water solubility was measured by dissolving the sample of 1 g (initial sample weight) in deionized water of 100 ml and magnetically stirring for 5 minutes. The mixture was centrifuged at 3000 *g* for 5 minutes. The supernatant was freeze dried. The water solubility of the sample was the ratio of the supernatant weight in the initial sample weight.

Repose angle of the sample was measured by a fixed cone bottom method. A funnel was fixed perpendicularly to the glass panel with a distance of 5 cm. The sample was added slowly in the funnel to form a cone in the glass panel. When the maximal height of the cone was reached, the angle of the cone and the glass panel was nominated as a repose angle.

ORAC value was determined according to the recently reported protocol (Ou *et al.* 2002). Fluorescein was chosen as the fluorescent probe. The final assay solution contained 0.067 μM of fluorescein, 60 mM of 2,2'-azobis-2-methyl-propanimidamide, 300 μl of sample or 7% β-cyclodextrin as a reagent blank. The fluorescence of an assay mixture was measured and recorded every minute. The trolox equivalent was calculated using a standard curve prepared with trolox, and used to compare ORAC of various samples by expressing as the μM/g.

The 2,2-diphenyl-1-picryhydrazyl radical (DPPH) scavenging activity was evaluated according to recently reported method (Yu *et al.* 2002). The final concentration was 100 μM of DPPH with the butylated hydroxytoluene as the control. The DPPH radical scavenging activity was calculated as a percentage of DPPH discolouration.

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Hydroxyl radical scavenging activity was examined based on the Fenton reaction, whereas 5,5-dimethyl-*N*-oxide pyrroline was used as the trapping agent. The reaction mixture contained 10 µl of 3 mM freshly prepared $FeSO_4$, 80 µl of 0.75 mM PBS, 15 µl of 10 mM H_2O_2, 15 µl of 1 M DMPO and 30 µl of sample or solvents for the blank.

The particle size of the sample was measured by a laser particles size analyser (Microtrac S3500; Microtrac Inc., USA). The sample was measured followed the standard operation process with alcohol as the dispersant. $D_{(number)}$ represents the value of the particle diameter at (number) % in the cumulative distribution. Thus D_{50} is also known as the median diameter or the medium value of the particle size distribution, it is the value of the particle diameter at 50% in the cumulative distribution. Span is the ratio of the difference of the D_{90} and D_{10} in D_{50}.

The microstructure of the sample was imaged using a scanning-electron microscopy (S-4800; Hitachi Co Ltd., Japan) with silver coating. The sample was fixed by a double faced adhesive tape and imaged with an acceleration voltage at 5 kV.

The X-ray diffraction of the samples was recorded on a powder X-ray diffraction (Bruker D8-Advance; Bruker AXS, Germany) using Cu-Kα radiation (λ = 1.5406 Å) with scattering angles (2θ) of 3–90° at 0.02°/step.

The aroma of the sample was compared by an electronic nose (PEN2; Airsense Analytics GmbH, Germany). The electronic nose was warmed for 30 min and flushed the testing system for 180 seconds. The sample of 1 g was dissolved in deionized water of 10 ml, and then put in the testing tube at 25°C. The electronic sensor was put into the testing tube to collect the response for 60 s with the air circulation of 300 ml/minute.

Statistical analysis. Analysis of variance was used to compare mean differences of the results. If the differences in mean existed, multiple comparisons were performed using Duncan's multiple range test. All analysis was conducted using SPSS for Window Version 19. All experiments were done in triplicates or more.

RESULTS AND DISCUSSION

Effect of inlet temperature on the properties. The moisture content of the watermelon powder decreased with increasing inlet temperature of the drying air (Table 1). The moisture contents of the powder dried at 140 and 150°C were significantly lower than those dried at 120 and 130°C, which was consistent with the results of amla powder (Mishra *et al.* 2014), pear powder (Rodríguez-Hernández *et al.* 2005), and watermelon powder (Quek *et al.* 2007).

The water activity of the watermelon powder were not significantly affected by the inlet temperature of the drying air, which was consistent with recently reported results (Quek *et al.* 2007). Remarkably, the water activity of sage powder and tamarind pulp powder decreases with increasing inlet temperature (Şahin-Nadeem *et al.* 2013; Muzaffar & Kumar 2017). This difference could result from the difference of the carrier material. Sage powder uses mixed β-cyclodextrin, arabic gum, and maltodextrin as carrier, while our watermelon powder only used maltodextrin. Moreover, the water activity of each powder remained below 0.3, which inhibited the growth of most microorganisms.

Water solubility is a further important quality aspect of powdered products as it affects the powder's functional properties in food systems (Şahin-Nadeem *et al.* 2013). The water solubility of the watermelon powder was not significantly affected by the inlet temperature of the drying air, which was consistent with the results for amla powder (Mishra

Table 1. Moisture content, water activity, solubility, and repose angle of watermelon powders

Inlet temperature (°C)	Moisture content (%)	Water activity	Solubility (%)	Bulk density (g/ml)	Repose angle (°)	DPPH scavenging capacity (%)	Hydroxyl scavenging capacity (%)	ORAC (µM/g)
120	2.09 ± 0.023^a	0.27 ± 0.0039^a	96.55 ± 0.10^a	0.46 ± 0.01^a	33.8 ± 0.52^a	19.63 ± 1.26^a	76.06 ± 3.32^a	238
130	1.98 ± 0.45^a	0.28 ± 0.0048^a	96.31 ± 1.45^a	0.45 ± 0.01^a	41.5 ± 0.84^b	20.52 ± 1.74^a	73.25 ± 2.64^a	253
140	1.78 ± 0.11^b	0.28 ± 0.0036^a	97.67 ± 0.74^a	0.47 ± 0.02^a	43.3 ± 0.96^b	30.79 ± 1.03^b	72.21 ± 3.53^a	147
150	1.43 ± 0.044^c	0.26 ± 0.0057^a	98.76 ± 1.26^a	0.43 ± 0.01^a	45.4 ± 0.90^c	21.87 ± 2.32^a	71.82 ± 4.86^a	159

Values are means ± standard deviation ($n \geq 3$). Data with different letter as superscript represent a significant difference ($P < 0.05$)

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et al. 2014), black mulberry powder (Fazaeli *et al.* 2012), pear powder (Rodríguez-Hernández *et al.* 2005), and tomato powder (De Sousai *et al.* 2008). In contrast to our results, the inlet temperature significantly increased water solubility from 59.82% to 65.22% of the tamarind pulp powder (Muzaffar & Kumar 2017). Moreover, the water solubility of all tested watermelon powders ranged around 96%, which was higher than that of the tomato powder of 17.65–26.3% (De Sousai *et al.* 2008), gac powder of 36.91–38.25 % (Kha *et al.* 2010), and pineapple powder of 81.56% (Abadio *et al.* 2004).

Bulk density and repose angle are important properties of powdered products. High bulk density is desirable to reduce shipping and packaging costs, while low bulk density (as seen in agglomerated products) influences other powder properties such as flowability and instant characteristics (Şahin-Nadeem *et al.* 2013). The bulk densities of watermelon powder remained unaffected by the inlet temperature, which was consistent with the results for sage powders (Şahin-Nadeem *et al.* 2013). The repose angle is a widely used empirical parameter for characterizing the flowability of a powder. The flowability of powders depends on both physical properties and storage conditions of powders (Rattes & Oliveira 2007). A smaller repose angle indicates better flowability. In particular, the particle size has a key influence on powder flowability. As particle size decreases, the surface area per unit mass of powder increases, thus reducing flowability. More contact surface area is available for cohesive forces and frictional forces to resist flow. Particle shape also influences powder flowability. It furthermore influences the surface contacts between particles. The powder moisture content has a significant impact on powder flowability. Increasing the moisture content leads to reduced flowability due to increased liquid bridges and capillary forces that act between the powder particles. In addition, increased moisture content can soften the powder material, and the water-soluble constituents in particular, which results in the deformation of the powder and a higher contact surface area (Kim *et al.* 2005). The repose angle of the powder dried at 120°C was significantly lower than that dried at all other temperatures. This could result from the increased particle size of the powder dried at 120°C. Remarkably, the repose angle of the powder dried at 150°C was significantly higher than that of the powder dried at 120°C although their particle sizes were similar. This difference could be explained by the higher moisture content of the powder dried at 120°C.

Effect of inlet temperature on antioxidant capacity. The ORAC value of the watermelon powder decreased when the inlet temperature increased from 120°C to 150°C (Table 1). The watermelon powder dried at 130°C showed the highest ORAC value of 253 μM/g. Similar to our results, the antioxidant content of the pear powder decreases when the inlet temperature increases from 120°C to 200°C (Rodríguez-Hernández *et al.* 2005). However, the antioxidant content of sage powder significantly increases at a higher drying temperature (Şahin-Nadeem *et al.* 2013). Moreover, the ORAC value of the powder dried at 130°C was lower than that of blueberry powder (469 μM/g), but higher than that of most fruits, such as apple of 149 μM/g, avocado of 86 μM/g, broccoli of 168 μM/g, muskmelon of 45 μM/g, carrot of 25 μM/g, and tomato of 53 μM/g (Ou *et al.* 2002). According to a survey conducted by the USDA, intake of ORAC above 6000 units per day will maintain the metabolic balance of an adult. Sub-health and elder population require to intake higher levels to maintain health (Prior & Cao 1999; Kucich 2015). The intake of watermelon powder of 20–30 g would meet the daily requirement for an adult according to the USDA standard.

The effect of inlet temperature on DPPH and hydroxyl radical scavenging capacities of watermelon powder was compared (Table 1). The watermelon powder dried at 140°C showed a stronger DPPH scav-

Table 2. Effect of inlet temperature on the colour and particle size of watermelon powders

Temperature (°C)	*L**	*a**	*b**	D_{50} (μm)	Span (μm)
120	90.56 ± 0.14^{a}	6.18 ± 0.087^{a}	12.24 ± 0.14^{a}	21.64 ± 1.22^{a}	2.76 ± 0.20^{a}
130	89.32 ± 0.06^{a}	7.47 ± 0.085^{b}	12.16 ± 0.092^{ab}	18.21 ± 0.22^{b}	1.73 ± 0.038^{c}
140	88.16 ± 0.41^{ab}	8.03 ± 0.19^{b}	13.29 ± 0.19^{ab}	13.44 ± 0.36^{c}	1.81 ± 0.044^{c}
150	86.89 ± 0.042^{b}	9.84 ± 0.081^{c}	11.84 ± 0.095^{b}	21.21 ± 0.26^{a}	2.05 ± 0.035^{b}

Values are means ± standard deviation ($n \geq 3$). Data with different letter as superscript represent a significant difference ($P < 0.05$).

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enging capacity than that dried at other temperatures. The DPPH scavenging capacity of the watermelon powder was lower than that of the normal red wine (60–70%) (Sánchez-Morenoa *et al.* 1999). This is because DPPH radical has been used to assess the ability of phenolic compounds to transfer labile H atoms to radicals (Goupy *et al.* 2003). Consequently, DPPH scavenging activity is linearly related to the phenolic content (Villano *et al.* 2007). The phenolic content of watermelon juice is typically around 50 mg/l, thus the phenolic content of the watermelon powder was around 250 mg/l after adding maltodextrin and other components, while that of wine is typically around 1000 mg/l.

Hydroxyl radicals are one of the most dangerous reactive oxygen species. Over 100 diseases have been implicated with the actions of the hydroxyl radical (Halliwell & Gutteridge 1990). The watermelon powder scavenged the hydroxyl radical at level of 70–80%. The inlet temperature showed no significant influence on the hydroxyl radical scavenging capacity of watermelon juice.

Effect of inlet temperature on the colour. The L^* of watermelon powder decreased when the inlet temperature increased, while the a^* increased (Table 2). The L^* and a^* represent the lightness and redness of the sample, respectively. Consequently, the combination of the increase of the L^* and decrease of the a^* formed the dark-red colour of the powder, which was the results of browning and caramelization of glucose and fructose in the watermelons (Souad *et al.* 2012; Zhang *et al.* 2015). Hence, a higher inlet temperature was detrimental to maintain the original colour of the watermelon powder.

Effect of inlet temperature on the particle size. The effect of inlet temperature on the particle size, span, and microstructure of watermelon powder is shown in Table 2 and Figure 1, respectively. The D_{50} of watermelon powder dried at 130 and 140°C was significantly lower than that of dried at other inlet temperatures ($P < 0.05$). In contrast to our results, the particle size of fish oil and acai powder increases with increasing inlet temperature (Tonon *et al.* 2011; Aghbashlo *et al.* 2013). Drying at a higher temperature leads to the early formation of a structure and does not allow the particles to shrink, while the particle continues to shrink when the inlet temperature is not sufficiently high to form the powder structure (Reineccius 1989). Span represents the distribution span of the powder. The sample is uniform when the span is small. The span of the powder dried at

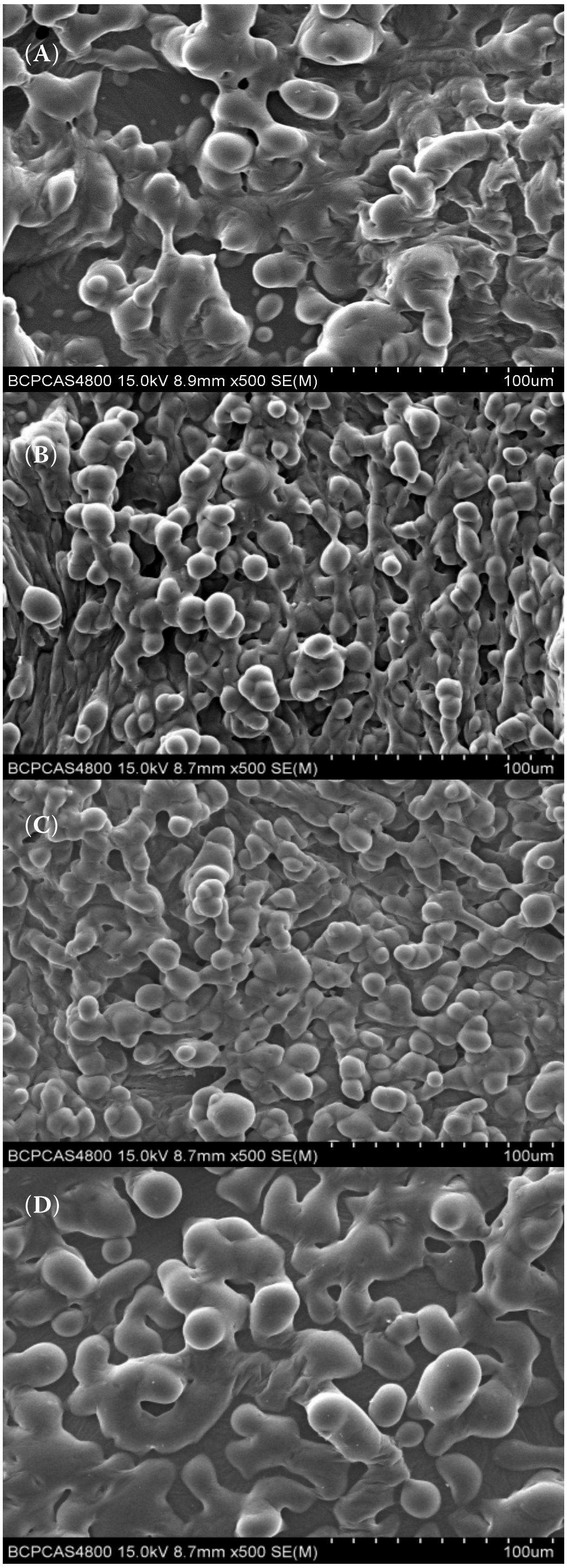

Figure 1. Microstructure profiles of watermelon powders. (**A**) 120°C, (**B**) 130°C, (**C**) 140°C, (**D**) 150°C

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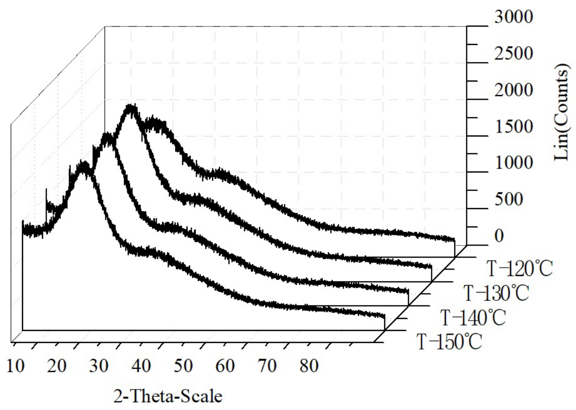

Figure 2. Effect of inlet temperature on the crystallinity of watermelon powders

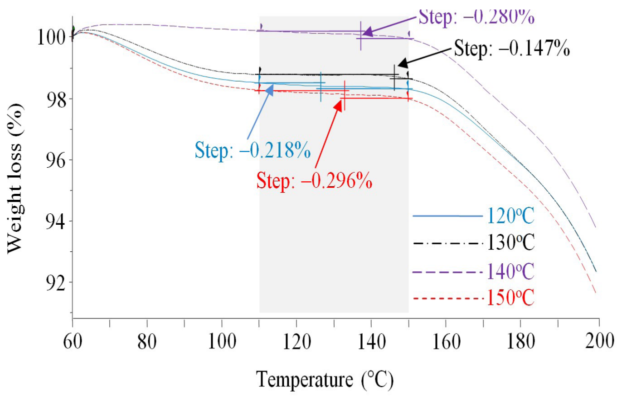

Figure 3. Effect of inlet temperature on the weight loss of watermelon powders

130 and 140°C was significantly smaller than that dried at the other temperatures. Moreover, each watermelon powder presented a smooth sphere but a different size. The inlet temperature did not affect the surface smoothness of the particles. This however contradicted the observation of the microstructure of milk powder (Nijdam & Langrish 2006), acai powder (Tonon *et al.* 2008), and tamarind pulp powder (Muzaffar & Kumar 2017). The tamarind pulp powder shows a bigger particle size at higher inlet temperatures (Muzaffar & Kumar 2017). Hence, the particle size of the watermelon powder dried at 130 and 140°C was more uniform.

Effect of inlet temperature on crystallinity and thermal stability. The effect of inlet temperature on the crystallinity of watermelon powder is shown in Figure 2. The crystallinity of the powders dried at 120, 130, 140, and 150°C were 14.3, 28.3, 26.9, and 24.9%, respectively. Watermelon powder dried at 130°C showed maximal crystallinity. Higher crystallinity would lead to higher thermal stability of the products. Consequently, the thermal stability of watermelon powders dried at different temperatures was compared via TGA analysis (Figure 3). The weight loss profile of each powder presented a plateau for temperatures of 110–150°C. On this plateau, the weight loss of the watermelon powder dried at 120, 130, 140, and 150°C reached 0.280, 0.147, 0.218, and 0.296%, respectively. The weight loss of the watermelon powder dried at 120 and 150°C was significantly higher than that of powder dried at 130 and 140°C. This phenomenon proved that the powders dried at 120 and 150°C lose weight easily. The thermal stabilities of powders dried at 130 and 140°C were higher than that dried at 120 and 150°C, which was in accordance with the crystallinity results of the watermelon powder. Therefore, the watermelon powder dried at 130 and 140°C presented stronger thermal stability due to its higher crystallinity.

Effect of inlet temperature on the aroma. The aroma of dissolved watermelon powder was compared to that of fresh watermelon juice and formulated juice via electric nose (Figure 4). The variances of the main components – 1 and 2, were 96.98 and 2.72%, respectively. The sum of the main components 1 and 2 reached 99.7%, representing the aroma of all tested samples. Remarkably, the variance of the main component 1 was 96.68 %, thus dominating the aroma of the dissolved watermelon powder. The shadow of the fresh watermelon juice in the main component 1 overlapped with that of the formulated juice and dissolved powder dried at 130–150°C. Consequently, the aroma of the dissolved watermelon powder dried at 130–150°C was similar to that of the fresh watermelon juice, while the aroma of the dissolved watermelon powder dried at 120°C was not. Hence, an inlet temperature of 130–150°C maintained the aroma of the watermelon juice.

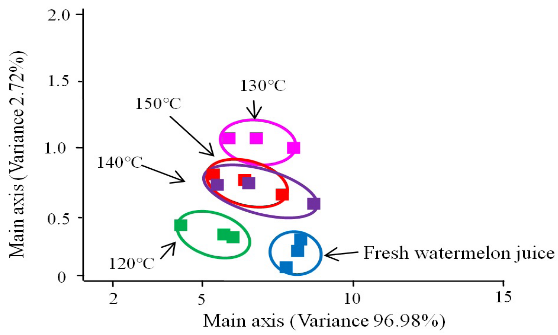

Figure 4. Effect of inlet temperature on the aroma of watermelon powders

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CONCLUSIONS

Inlet temperatures of the drying air of 120, 130, 140, and 150°C maintained water solubility of the watermelon powder at 96%, which was higher than most fruit powders. The water activity and bulk density of the watermelon powder remained unaffected by the inlet temperature. The redness of the powder was intensified with the increasing inlet temperature. Remarkably, the ORAC value of the powder dried at 130°C was the highest of 253 μM/g in all tested powders. The D_{50} of the watermelon powder dried at 130°C was 18.21 ± 0.22 μM with a span of 1.73 ± 0.038, which was more uniform than the other powders. The crystallinity of the powder dried at 130 °C was higher than that dried at 120 and 150 °C, and showed stronger thermal stability. Moreover, watermelon powder dried at 130°C presented a similar aroma as the fresh watermelon juice when being solved. Hence, the inlet temperature of the drying air of 130°C was the optimal temperature for the production of the watermelon powder.

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