

Enhancing the stability of anthocyanins: Effects of encapsulation and drying in black grape juice powder

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Abstract

Plant-based beverages are one of the fastest-growing sectors in the food and beverage industry. However, the short shelf-life and lower stability of its liquid form required conversion into a stable instant juice powder, with enhanced shelf life. Microencapsulation via spray-drying protects sensitive bioactive compounds, such as anthocyanins, from degradation. This study evaluated the impact of different encapsulating agents (EAs) and inlet temperatures on the stability of anthocyanins and the physicochemical properties of spray-dried black grape juice powder. Maltodextrin (MD) and gum Arabic (GA) were used as EAs. Black grape juice was spray-dried at an inlet temperature of 160°C using different concentrations of MD (25, 30, 35, 40, 45, and 50%, w/v). The MD concentrations of 40% and 50% yielded the highest powder recovery (50.06–50.38%). The concentration of EA (40%, w/v) and inlet temperature (160°C) were selected to compare the efficacy of MD and the combination of MD and GA (MD+GA) at a ratio of 8:1. A higher total monomeric anthocyanin content (TMAC) (8.69 mg/100 g dry matter [DM]) and lower moisture content (~1.681%) were observed in MD+GA. Spray-drying at an inlet temperature of 180°C significantly ($p < 0.05$) enhanced TMAC and antioxidant activity and produced the smoothest particle surfaces, compared to 150°C, 160°C, and 170°C. These findings offer valuable insights for the food industry into optimizing encapsulation strategies and processing conditions to enhance stability and physicochemical properties of anthocyanins in black grape juice powder.

Keywords: gum Arabic; maltodextrin; microencapsulation; natural colorants; spray-dried powder

Introduction

In this advanced technological era, demand for health-promoting food and beverages, especially juice, is increasing in the food industry. Grapes (*Vitis vinifera*) are one of the largest harvested crops globally. According to the US Department of Agriculture, Foreign Agricultural Service (2025), 27.9 million metric tons of grapes were produced from 2023 to 2024. A high concentration of antioxidants, fibers, sugars, acids, vitamins,

and minerals from various parts of grape extracts (skin, seeds, and juice) is responsible for their antioxidant, anti-inflammatory, and cholesterol-reducing properties, contributing to improved heart health (Margean *et al.*, 2020).

Anthocyanins are the members of the phenolic compounds group known as flavonoids. This water-soluble pigment is commonly found in fruits and vegetables, exhibiting blue, red, or purple colors. The molecular structure of anthocyanin is characterized by a typical C6-C3-C6

configuration, composed of two aromatic rings, where one forms a benzopyrylium (flavylium) core and the other a phenolic ring, which is interconnected by a three-carbon chain. This configuration underlies the basic structural motif of flavonoids (Vergel-Alfonso *et al.*, 2025). These pigments are particularly unstable and prone to degradation because of environmental factors, such as light, temperature, pH, humidity, oxygen, and enzymes (Ijod *et al.*, 2024; Nawawi *et al.*, 2023). Various methods of anthocyanins stabilization were used to preserve their integrity, including micro- and nano-encapsulation, film, and copigmentation (Estrella-Osuna *et al.*, 2022; Figueroa-Enríquez *et al.*, 2024; Ijod *et al.*, 2025). Furthermore, anthocyanin-rich fruit juice, such as grape juice, is unstable, especially in liquid form. Its high moisture negatively affects its shelf life and makes difficult its handling. As a result, development of powdered fruit juices in the beverage industry represents a remarkable measure of introducing a product with high stability and economic benefits (Srivastava *et al.*, 2022).

Microencapsulation is an excellent process to reduce the vulnerability of fruits' bioactive components to various environmental conditions and improve the quality of powdered fruit juices. It is commonly recognized to protect bioactive compounds using various encapsulating agents (EAs) as a protective wall material (Laureanti *et al.*, 2023). Spray-drying is widely used to transform liquid into powder form by atomizing the liquid in a hot drying chamber at specific parameters. The production of spray-dried powder involves various steps, such as preparation, homogenization, atomization, dispersion, and liquid dehydration (Ijod *et al.*, 2024). Owing to their versatility and efficiency, spray-dried powders are highly relevant in various sectors, such as pharmaceutical, cosmetics, nutraceutical, and the food industry. In addition, the reproducibility and scalability of spray-drying make it popular, compared to freeze-drying (Catalkaya *et al.*, 2022). Nowadays, microencapsulation is widely applied in the food industry to preserve and prevent the degradation of heat-sensitive compounds, such as anthocyanins (Ijod *et al.*, 2024). Various studies have reported the efficiency of spray-drying for protecting and producing excellent spray-dried powder qualities (shape and size). For instance, Catalkaya *et al.* (2022) reported that using maltodextrin (MD) and gum Arabic (GA) at a MD–GA ratio of 15:5 to encapsulate black chokeberry using inlet temperature (150°C) and outlet temperature (90°C) produced the highest encapsulation efficiency, better protection of antioxidant activity, and uniform and spherical powder size, all suitable for food application.

However, improper selection of inlet temperature can negatively influence the stability and integrity of anthocyanins, especially with insufficient EAs

(Nguyen *et al.*, 2021). MD is a widely used EA and exhibits excellent solubility and moderate hygroscopicity, and can increase glass transition (T_g) temperature (Adetoro *et al.*, 2020), making it suitable for producing stable anthocyanin-based powder. MD is a colorless, odorless, and highly soluble natural polysaccharide that is usually produced from the starch of corn, wheat, or potato (Sharif *et al.*, 2020). GA, on the other hand, is a complex material made up of polysaccharides and glycoproteins from the acacia tree, which has excellent emulsifying, stabilizing, and film-forming properties, making it ideal for protecting anthocyanins during spray-drying (Catalkaya *et al.*, 2022; Nguyen *et al.*, 2022). Both MD and GA are food-grade components safe for consumption. Various studies have explored the microencapsulation of grape juice to preserve anthocyanin and antioxidant activity (Almeida *et al.*, 2024; Karadag *et al.*, 2024; Nascimento *et al.*, 2023). For instance, Almeida *et al.* (2024) used a combination of rice bran protein hydrolysates (RBPH) and MD to encapsulate grape juice using an inlet temperature of 140°C. The authors revealed that the combination of RBPH and MD better preserves anthocyanins and antioxidant activity, reduces particle size, and improves powder recovery, compared to the unencapsulated sample. Meanwhile, Karadag *et al.* (2024) studied the effect of combining grape pectin (GP) and MD by using different ratios of MD–GP (e.g., 100:0, 90:10, 85:15, 75:25, and 60:40 w/w) to encapsulate grape juice. The study revealed that combining 25% of GP in MD leads to a better powder yield and increases the preservation of anthocyanins. These findings suggest superior synergistic effect between two EAs, compared to a single EA, to preserve bioactive compounds in a better manner in grape juice.

However, limited studies have reported the effect of combining MD and GA (MD+GA) for the microencapsulation of grape juice. Hence, MD+GA as an EA is a promising approach to produce a stable and instant black grape juice powder. Therefore, this study aimed to investigate the effects of (i) MD and MD+GA, and (ii) different inlet temperatures on the anthocyanins' stability and physicochemical properties of black grape juice powder.

Materials and Methods

Materials

The following chemicals were obtained from Sigma-Aldrich, Merck (St. Louis, MO, USA): potassium chloride buffer (0.025 M), sodium acetate buffer (0.4 M), 4,6-tripryridyls-triazine (TPTZ), gallic acid (GA, 98%), Trolox, Folin–Ciocalteu reagent, 2,2-diphenyl-2-picrylhydrazyl (DPPH), and iron (III) chloride hexahydrate. Black grapes were

purchased from a supermarket in Putrajaya, Malaysia. EA, including MD with dextrose equivalent 10 (DE 10) and GA were acquired from R&M Chemicals, UK.

Extraction of black grape juice

Black grapes were removed from stems and soaked in tap water for 10 min, followed by a thorough rinsing under flowing water. Black grape juice was extracted by grinding in a blender (Panasonic, Japan) for 2 min. The crunched pulp was then passed through a 20-mesh sieve to separate fiber from juice and centrifuged in a centrifuge (Kubota, Japan) at 2,500 rpm for 20 min to further separate juice from pulp.

Determination of MD concentration as encapsulating agent

The spray-drying process was executed in duplicate (n=2) using different concentrations of MD in grape juice (25, 30, 35, 40, 45, and 50%, w/v). First, MD was added to grape juice and stirred using a stirrer until it was fully dissolved (Favorit, Malaysia). A mini spray dryer (Büchi Labortechnik AG, Switzerland) was used with an aspirator rate (100%), pump power (10%), and inlet temperature (160°C), following the process of Fang and Bhandari (2012) with slight modifications. Distilled water was sprayed for 5 min before and after the feed was passed through to clean the tube and nozzle.

Comparison between MD and MD+GA

Concentration of MD with the highest powder yield was compared with that of MD+GA at a ratio of 8:1 (w/v). The selection of 8:1 (w/v) ratio was based on preliminary studies, where MD+GA at a ratio of 8:1 (w/v) showed significantly higher solubility and improved drying operation. MD+GA was added to grape juice and stirred with a stirrer until it was fully dissolved. The parameters of spray-drying (aspirator rate, pump power, and inlet temperature) and cleaning protocol were fixed as mentioned in the previous section.

Determination of inlet temperature

Encapsulating agent, which yielded powder with the highest levels of total monomeric anthocyanin content (TMAC), total flavonoid content (TFC), total phenolic content (TPC), and antioxidant activities, was used to determine the most effective inlet temperature (150, 160, 170, and 180°C). The powder yield and physicochemical

properties of spray-dried powder were used to determine the most efficient inlet temperatures. The parameters for spray-drying (aspirator rate and pump power) and cleaning protocol were fixed following the previous section. Spray-drying using different inlet temperatures was performed in duplicate (n=2). Figure 1 shows the flow chart of spray-drying of grape juice.

Analysis of black grape juice

pH and total soluble solids (TSS)

The acidity of black grape juice was measured using a pH meter (Toledo, Spain). The TSS of black grape juice was obtained by using a digital refractometer (Hanna Instrument, Woonsocket, Romania). Measurements were performed in triplicate.

Analysis of black grape juice powder

Process yield

The process yield of spray-dried powder was calculated according to the method described by Fang and Bhandari (2012) by using the following equation:

$$\text{Process yield (\%)} = \frac{\text{Weight of spray dried powder (g)}}{\text{Weight of feed solution (g)}} \times 100\% \quad (1)$$

Moisture content

The moisture content of black grape juice powder was measured using a moisture analyzer (Ohaus, USA). All measurements were performed in triplicate.

Bulk density

The powder's bulk density was measured using a 5-mL measuring cylinder. Grape powder (0.5 g) was poured into a measuring cylinder, which was then tapped for 20 times. Bulk density was calculated by using the following equation:

$$\rho = \frac{m}{V} \left(\frac{\text{g}}{\text{mL}} \right), \quad (2)$$

where m is the weight of black grape powder and V is the apparent volume of the measuring cylinder (Chang *et al.*, 2020). Measurements were performed in triplicate.

Solubility

The solubility of spray-dried grape powder was determined by dissolving 0.5 g of powder in 15 mL of distilled water by stirring at a constant speed of 160 rpm with a magnetic stirrer. The time was recorded until

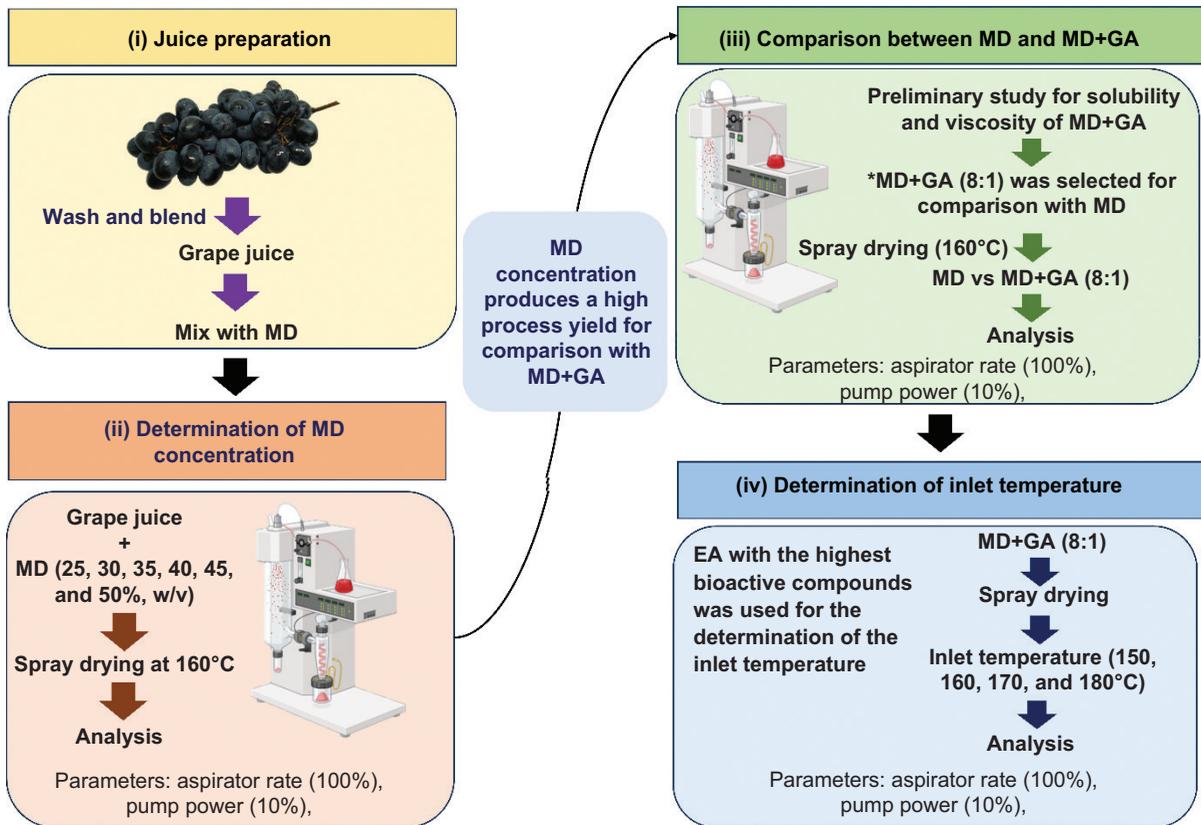


Figure 1. Flow chart of spray-drying operation for production of spray-dried grape juice powder.

the powder was fully dissolved. The solubility was calculated according to the following equation (Chang *et al.*, 2020):

$$\text{Solubility} \left(\frac{\text{s}}{\text{g}} \right) = \frac{\text{Time required to dissolve the powder (s)}}{\text{Weight of powder (g)}} \quad (3)$$

Hygroscopicity

The hygroscopicity of grape powder was determined using the method described by Sarabandi *et al.* (2019) with slight modification. Saturated sodium chloride (NaCl) was placed in a desiccator to maintain a relative humidity (RH) of 54%. In all, 1-g samples were weighed in a petri dish, and all samples were placed inside a desiccator and left for a week. Hygroscopicity was determined initially on a wet basis. For standardization and better comparison, values were recalculated and expressed on a dry matter (DM) basis using the following equation:

$$\text{Hygroscopicity (dry basis)} = \frac{\text{Hygroscopicity (wet basis)}}{\text{Dry matter fraction}} \times 100, \quad (4)$$

where the dry matter fraction was derived from the moisture content expressed on a dry basis.

Color measurements

Dry powder's color was measured using a chromameter (Konica Minolta, USA). The color reading was expressed as L^* (lightness or darkness), a^* (redness or greenness), and b^* (yellowness or blueness). The hue and chroma of spray-dried grape juice powder were calculated using the following equation:

$$\text{Hue} (h^\circ) = \tan^{-1} \left(\frac{b^*}{a^*} \right), \quad (5)$$

$$\text{Chroma} = \sqrt{(a^*)^2 + (b^*)^2}. \quad (6)$$

Total phenolic content

The TPC was determined according to the Folin–Ciocalteu method as described by Azman *et al.* (2020). The absorbance was measured at 765 nm using a spectrophotometer (Biomate; Thermo Scientific, UK). The standard curves of gallic acid were plotted between 0.0 mg/mL and 1.0 mg/mL. Measurements were performed in triplicate and the

results were expressed in mg gallic acid equivalent (GAE)/100 g DM of samples.

Total flavonoid content

The TFC was determined following the method described by Mat Ramlan *et al.* (2021). The absorbance was measured at 450 nm by using a spectrophotometer. The standard curves of quercetin were plotted between 0.00078 mg/mL and 0.1 mg/mL. Measurements were performed in triplicate and the results were expressed in mg quercetin equivalent (QE)/100 g DM of samples.

Total monomeric anthocyanin content

The TMAC of black grape powder was determined according to the method described by Lee *et al.* (2005). The absorbance was measured at 520 nm and 700 nm using a spectrophotometer for pH 1.0 and 4.5 solutions. Measurements were performed in triplicate. The results were expressed in mg/100 g DM of samples. The concentration of TMAC was calculated using the following equation:

$$\text{Cyanocyanin} \left(\frac{\text{mg}}{\text{L}} \right) = \frac{A \times MW \times DF \times 10^3}{\epsilon \times 1}, \quad (7)$$

where A is the absorbance, MW is the molecular weight of cyanidin-3-*O*-glucoside, DF is the dilution factor, and $\epsilon = 26,900$ molar. The anthocyanin content was then calculated using the following equation:

$$\text{Anthocyanin content} \left(\frac{\text{mg}}{100\text{g}} \right) = \frac{\text{Cyanocyanin} \left(\frac{\text{mg}}{\text{L}} \right) \times \text{extract (L)}}{\text{sample (g)} \times 100}. \quad (8)$$

Radical scavenging activity

The DPPH radical scavenging activity of black grape juice powder was determined according to the method described by Ezzat *et al.* (2020). DPPH reagent, 0.15 mM, was prepared in methanol. The absorbance was measured at 517 nm by using a spectrophotometer. Standard curves of Trolox equilibrium were plotted at 0–2,000 $\mu\text{mol/L}$. Measurements were performed in triplicate, and the results were expressed in μmol Trolox equivalent (TE)/100 g DM of samples.

Reducing power

The ferric-reducing antioxidant power of black grape juice powder was determined according to the method described by Nawawi *et al.* (2023). The absorbance was measured at 593 nm by using a spectrophotometer. Standard curves of Trolox equilibrium were plotted at 250–1,500 $\mu\text{mol/L}$. Measurements were performed

in triplicate, and the results were expressed in μmol TE/100 g DM of samples.

Morphology of powder

The morphology of black grape juice powder was investigated using a scanning electron microscope (SEM) (JEM-2100F, JEOL, Osaka, Japan). An ample quantity of powder was affixed to SEM stubs, coated with platinum, and subjected to SEM for image analysis. The observations were conducted at an accelerating voltage of 5.0 kV with 500 \times , 1,000 \times , and 2,000 \times magnification.

Statistical analysis

All data were expressed as mean \pm standard deviation (SD). The software for statistical analysis was Minitab version 21 (Minitab Inc, USA). One-way analysis of variance (ANOVA) was performed to assess significant differences among treatments, followed by Tukey's HSD test for *post hoc* multiple comparisons ($p < 0.05$). Pearson's correlation was used to analyze relationship between the data.

Result and Discussion

Physicochemical properties of black grape juice with different MD concentrations

pH and TSS

The pH values of black grape juice with different concentrations of MD ranged from 4.23–4.62 (Table 1). The pH level of black grape juice without MD was notably lower compared to that with MD ($p < 0.05$). MD is a polysaccharide that is either neutral or slightly acidic and does not have a significant impact on the system's acidity. Adding MD to black grape juice may dilute acidic components, leading to a slight increase in pH (Paiva *et al.*, 2023).

Table 1. pH value and total soluble solids (TSS) of black grape juice with different concentrations of MD.

Concentration of MD in black grape juice (% w/v)	pH	TSS ($^{\circ}$ Brix)
0	4.23 \pm 0.02 ^d	21.25 \pm 0.07 ^g
25	4.40 \pm 0.01 ^c	37.20 \pm 0.14 ^f
30	4.57 \pm 0.01 ^{a,b}	39.45 \pm 0.07 ^e
35	4.54 \pm 0.01 ^b	41.25 \pm 0.07 ^d
40	4.62 \pm 0.01 ^a	42.85 \pm 0.07 ^c
45	4.40 \pm 0.01 ^c	44.70 \pm 0.14 ^b
50	4.39 \pm 0.02 ^c	48.50 \pm 0.14 ^a

Notes: Each value represents mean \pm standard deviation ($n = 3$). Values with the same superscript alphabet in each column are not significantly different ($p > 0.05$).

The TSS content of grape juice increased significantly after adding 25% MD ($p < 0.05$). This increase can be attributed to the hygroscopic nature of MD, which absorbs moisture and adds to the solid content in juice, thereby elevating the TSS measurement (Adetoro *et al.*, 2020). There is a positive correlation between pH and TSS ($r = 0.573$, $p < 0.05$), indicating that the acidity of grape juice affects TSS value.

Spray-dried black grape juice powder with different MD concentrations

Process yield, feed flow rate, and outlet temperature

Incorporating 40% and 50% MD concentration resulted in a significantly higher process yield compared to other concentrations ($p < 0.05$) (Table 2). However, 40% concentration was selected due to the lower amount of MD used. The higher initial solid content was linked to increased recovery under the tested conditions. However, owing to the yield calculation methodology limitations, we acknowledge that this trend may not be solely attributable to the feed solid concentration. According to Fang and Bhandari (2012), higher concentrations of MD lead to increased powder recovery because of the elevation of solid content in the feed. Owing to limitations, the current study only uses °Brix for the estimation of the soluble solids in the powder, and this does not directly reflect the total DM in juice (soluble and insoluble solids). Therefore, a proper evaluation using the gravimetric method to improve the accuracy should be explored in the future. The process yield increases with higher concentration of MD; however, a decrease was observed at 45% MD, compared to that of 40% MD concentration ($p < 0.05$). High viscosity at 45% MD concentration may have disrupted atomization. This creates irregular particle sizes, and some may be deposited on the wall or lost in the cyclone. However, at 50% MD concentration, owing to the higher solids content, the formation of droplets and drying kinetics improved, which explains improvement in the process yield at 50% MD. A similar condition was reported by Nguyen *et al.* (2021), who found that the spray-drying of roselle anthocyanins resulted in poor particle formation at a certain concentration of EA, which influenced the process yield, and possibly have occurred in the current study.

During the drying operation, the feed flow rate of black grape juice was purposely adjusted to ensure optimal and consistent atomization because of difference in viscosity and TSS in grape juice solution with MD. The feed flow rate, which ranged from 3.52 g/min to 3.89 g/min, was used to obtain an optimal drying operation for different concentrations of MD. A lower concentration of MD is expected to result in a less viscous feed than with a higher MD concentration. Lower viscosity facilitates atomization, producing finer droplets that dry more

Table 2. Physical analyses of spray-dried black grape juice powder with different MD concentrations.

MD (% w/v)	Process yield (%)	Feed flow rate (g/min)	Outlet temperature (°C)	Moisture content (% dry basis)	Hygroscopicity (% dry basis)	Bulk density (g/mL)	Solubility (s/g)	L*	a*	b*	Hue (h°)	Chroma
25	34.61 ± 0.35 ^d	3.52 ± 0.06 ^b	97–109	2.40 ± 0.28 ^a	28.10 ± 1.93 ^{a,b}	0.23 ± 0.01 ^c	45.18 ± 6.82 ^d	73.01 ± 0.27 ⁱ	13.63 ± 0.03 ^a	0.25 ± 0.03 ^f	1.05 ± 0.12 ^e	13.63 ± 0.03 ^a
30	37.96 ± 0.23 ^c	3.82 ± 0.08 ^a	97–108	2.04 ± 0.06 ^{a,b}	32.21 ± 1.27 ^a	0.24 ± 0.02 ^{b,c}	61.30 ± 1.84 ^d	75.23 ± 0.03 ^e	12.25 ± 0.04 ^b	0.55 ± 0.02 ^e	2.57 ± 0.09 ^d	12.26 ± 0.04 ^b
35	38.48 ± 0.11 ^c	3.78 ± 0.04 ^a	95–103	1.84 ± 0.07 ^{a-c}	30.58 ± 3.16 ^a	0.29 ± 0.01 ^{b,c}	98.10 ± 1.84 ^c	79.79 ± 0.24 ^d	11.47 ± 0.04 ^c	1.06 ± 0.01 ^b	5.28 ± 0.04 ^b	11.52 ± 0.04 ^c
40	50.06 ± 0.08 ^a	3.70 ± 0.02 ^{a,b}	93–103	1.55 ± 0.07 ^{b-d}	20.28 ± 2.32 ^b	0.31 ± 0.03 ^{b,c}	90.75 ± 6.72 ^c	80.71 ± 0.10 ^c	10.70 ± 0.07 ^d	0.94 ± 0.05 ^c	5.04 ± 0.23 ^b	10.74 ± 0.08 ^d
45	48.18 ± 0.06 ^b	3.85 ± 0.06 ^a	89–106	1.40 ± 0.01 ^{c,d}	26.88 ± 2.35 ^{a,b}	0.32 ± 0.01 ^b	132.20 ± 9.62 ^b	83.66 ± 0.37 ^a	9.20 ± 0.01 ^f	1.43 ± 0.03 ^a	8.81 ± 0.15 ^a	9.31 ± 0.01 ^f
50	50.38 ± 0.18 ^a	3.89 ± 0.06 ^a	94–104	1.15 ± 0.21 ^d	32.21 ± 2.18 ^a	0.40 ± 0.03 ^a	189.80 ± 9.90 ^a	82.07 ± 0.04 ^b	10.18 ± 0.05 ^e	0.73 ± 0.00 ^d	4.10 ± 0.02 ^c	10.21 ± 0.05 ^e

Notes: Each value represents mean ± standard deviation (n = 3). Values with the same superscript alphabet in each row are not significantly different ($p > 0.05$).

rapidly. However, a slower feed rate is required to ensure adequate drying. A previous study on spray-dried apple powder conducted by Qadri *et al.* (2022) suggested that increasing the feed flow rate can decrease powder yield because of insufficient moisture removal, which restricts porosity and reduces powder dispersibility. If the feed rate is too high, then rapid atomization of a less viscous solution increases the risk of incomplete drying.

The outlet temperature of spray-drying ranged from 89°C to 109°C. The outlet temperature in spray-drying significantly influences the final product, affecting moisture content, particle size and morphology, and product quality attributes, such as color, flavor, nutrient content, powder flow ability, and solubility. Solubility is a crucial factor for balancing drying efficiency, product quality, and energy consumption, with higher temperatures speeding up drying and requiring less energy but potentially degrading sensitive compounds. Lower temperatures preserve these compounds but possibly require more energy and time (Abdullah *et al.*, 2022).

Physical analysis of spray-dried black grape juice powder using different MD concentrations

The moisture content of grape powders showed a decreasing trend with increase in MD concentration (Table 2). The moisture content was found to be lowest when a 50% concentration of MD was used ($p < 0.05$). Siccama *et al.* (2021) reported a correlation between the increase in MD concentration and the reduction of moisture content in spray-dried asparagus concentrate. According to Abdullah *et al.* (2022), the moisture content of spray-dried products must be lower than 5% to ensure powder stability during storage.

As the concentration of MD in grape powder increases, more moisture is absorbed and bound by MD molecules, thereby reducing free moisture content in the overall powder (Valenzuela and Aguilera, 2015). In addition, MD might enhance drying efficiency during the spray-drying process. It forms a protective layer around grape particles, facilitating better heat transfer and more efficient water removal, which result in lower moisture content in the final product (Mishra *et al.*, 2014). Fundamentally, the incorporation of MD for increasing concentrations leads to a more efficient encapsulation and binding of moisture, thereby reducing the overall moisture content of grape powders.

In this study, the hygroscopicity of black grape juice powder ranged from 19.96% to 31.85%. The hygroscopicity of powder from 40% MD exhibited a significantly lower value than 30%, 35%, and 50% MD ($p < 0.05$). At 40% MD, there may be an optimal balance between MD and other components in the powder. This balance can make a structure or composition less likely to absorb environmental moisture. In contrast, 30%, 35%, and 50% MD concentrations might not provide this optimal balance, resulting in

a higher hygroscopicity. However, no consistent trends were observed in the hygroscopicity of powder incorporated with different concentrations of MD. This result contradicts the study of spray-dried roselle anthocyanin conducted by Nguyen *et al.* (2021), who concluded that increasing roselle–MD ratio (1:50, 1:60, 1:70, 1:80, 1:90, and 1:100) led to a lower hygroscopicity. Notably, the hygroscopicity of spray-dried powders can be influenced by various factors, such as the type and concentration of EA, drying temperature, and feed flow rate. Therefore, determining the hygroscopicity characteristic of anthocyanin-rich powder is important for understanding suitable storage, handling, and stability of spray-dried products.

As shown in Table 2, the bulk density of spray-dried black grape juice powder showed an increasing trend with increasing MD concentrations. 50% MD recorded significantly high bulk density (0.40 ± 0.03 g/mL) among other MD concentrations ($p < 0.05$). As MD concentration increased, change was observed in the characteristics of the particles formed during spray-drying. Higher MD concentrations can lead to the formation of denser, spherical particles, which pack more efficiently, reducing the volume of the air trapped between particles and thus increasing the bulk density (Saha *et al.*, 2019). Additionally, higher MD may lead to lower free moisture content, leading to less agglomeration and denser powder packing.

Similar trends were observed in the findings of spray-dried ‘cempedak’ by Pui *et al.* (2021). The authors concluded that a higher concentration of MD led to an increase in bulk density. Typically, higher bulk density in powder is preferred as it reduces air spaces between particles and improves the overall storage stability (Parvez *et al.*, 2022). Therefore, increase in bulk density with higher MD concentrations in spray-dried powders is likely a result of changes in particle characteristics, particle size, moisture content, feed viscosity, and the molecular properties of MD, all of which contribute to denser powder packing.

Solubility refers to the ability of the powder to dissolve in a solvent, typically water. It is a critical property, especially for food, pharmaceutical, and various industrial applications (Jafari *et al.*, 2017). The current study used the time-based solubility method, modified from Chang *et al.* (2020), which measured the dissolution rate, rather than the total solubility capacity. The solubility of the spray-dried grape powder ranged from 45.18 s/g to 189.80 s/g. Increasing concentrations of MD resulted in decreased solubility, whereas powder encapsulated with 25% and 30% MD has the highest solubility, while 50% MD has the lowest solubility ($p < 0.05$). These findings contradict previous findings of Nguyen *et al.* (2021) on spray-dried roselle powder, where the encapsulation of roselle anthocyanins using roselle anthocyanins–MD ratios of 1:50, 1:60, 1:70, 1:80, 1:90, and 1:100 (w/w)

showed no significant changes in powder solubility. However, directly comparing the current findings on solubility might not be fully comparable because of the different method used by Nguyen *et al.* (2021). Improvement in solubility correlates directly with increase in MD concentration because MD exhibits a rapid dissolution rate attributed to its inherent water solubility.

As reported by Caliskan and Dirim (2016), the solubility of spray-dried powders generally increases with the addition of MD, which is commonly used in spray-drying formulations because of its positive impact on solubility. Notably, however, the effect of MD on solubility is influenced by various factors, such as its concentration, molecular weight, and the specific formulation of powder. In the current study, extremely high concentrations of MD might lead to a saturation point where additional MD may not significantly increase solubility and could affect other properties such as viscosity and texture (Siccama *et al.*, 2021). Chang *et al.* (2020) reported that the solubility of the spray-dried papaya puree powder ranges from 48.17 s/g to 65.72 s/g. The authors also observed that 10% MD concentration showed the lowest solubility, and increasing the MD concentration improved solubility. However, no significant difference in solubility was observed between 20% and 40% MD concentration, which contradicts the current study, where lower solubility was observed at higher MD concentration.

A strong negative correlation was observed between moisture content and process yield ($r = -0.935$), bulk

density ($r = -0.946$), and solubility ($r = -0.926$) at $p < 0.05$. A higher yield produced powder with lower moisture content because of efficient drying. Lower moisture content resulted in higher bulk density and a longer time to solubilize black grape juice powder. This is due to formation of agglomerates with a high average density, creating saturated internal spaces and increasing the bulk density (Pitt *et al.*, 2018). Moreover, lower moisture content led to lower solubility, confirming the reduction of conducive conditions for solute dissolution, resulting in lower solubility. Additionally, a strong positive correlation between bulk density and process yield ($r = 0.830$, $p < 0.05$) suggests that a higher yield indicates higher spray-drying efficiency, resulting in a powder with saturated pore spaces, thereby increasing bulk density.

Color measurements of spray-dried black grape juice powder with different MD concentrations

As shown in Table 2, the lightness (L^*) ranged from 73.01 to 83.66, redness (a^*) from 9.20 to 13.63, yellowness (b^*) from 0.25 to 1.43, hue (h°) from 1.05 to 8.81, and chroma from 9.31 to 13.63. MD concentration significantly affected spray-dried powder's L^* , a^* , and b^* values ($p < 0.05$). Lower L^* values indicate an increase in the darkness of powder. As depicted in Figure 2A, there was a significant decrease in the darkness of grape juice powder ($p < 0.05$), which was supported by the observed increase in L^* values. These results were in line with the study on spray-dried barberry juice conducted by Nadali *et al.* (2021), where increasing MD concentration increased the lightness of barberry juice powder and led

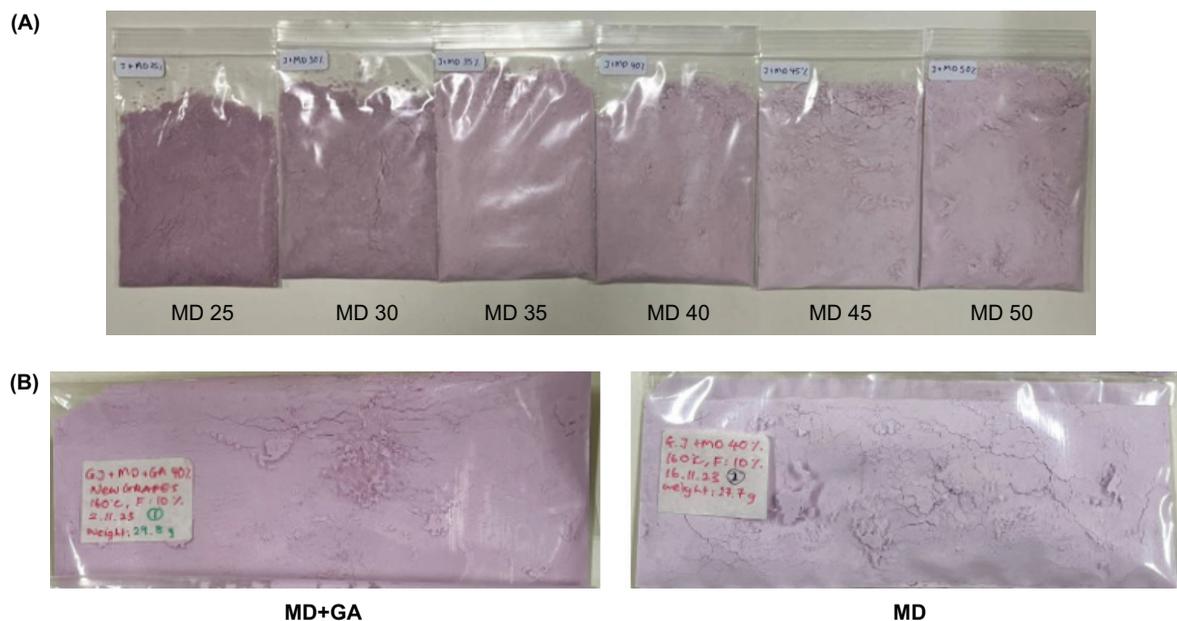


Figure 2. (A) Color of black grape juice powder spray-dried using different concentrations (%) of maltodextrin (MD), and (b) color of black grape juice powder spray-dried using MD and MD+GA. MD: maltodextrin; and GA: gum Arabic.

to a reduction in a^* and b^* values, suggesting that the white color of MD powder was possibly contributing to the increase in L^* value of spray-dried powder.

Increasing MD concentration dilutes the original material, reducing its color intensity. This dilution effect decreased the redness of spray-dried powder. In addition, MD itself may have a slight yellowish tint, and as its concentration increases, it can add to the overall yellowness of the mixture, hence increasing the b^* value. MD might increase the opacity or cloudiness of solution, which can also influence color perception. Overall, the decrease in a^* values and increase in b^* values with increasing MD concentrations are primarily due to the dilution of the original material's color intensity, inherent MD color properties, and changes in the optical properties of the mixture.

The low hue angles in all spray-dried powders were within the range of 0° – 90° , indicating the reddish color tone of powder. This indicates that the color of grape juice was retained even at higher MD concentrations, despite significant changes in the hue value across the samples. However, as reflected by the chroma value, the intensity significantly decreased when a higher MD concentration was used. The highest chroma was observed at 25% MD, indicating a higher color intensity in spray-dried grape juice powder. The intensity decreased with increase in the concentration of MD, suggesting that the white color of MD led to a faded or dull color of spray-dried powder. The lowest chroma was observed at 45% MD, possibly because of various reasons, such as encapsulation inefficiency, poor mixing, or the T_g effect. The nonlinear behavior of chroma at 45% MD was probably due to incomplete encapsulation, leading to the exposure of grape juice to oxidation and degradation. Furthermore, as MD concentration increased, the viscosity increased, resulting in uneven mixing or distribution during drying. This can also influence T_g effects that promote stickiness and caramelization, accelerating the color loss of grape juice.

A strong positive correlation between L^* and process yield ($r = 0.860$), bulk density ($r = 0.830$), solubility ($r = 0.820$), and b^* ($r = 0.871$) at $p < 0.05$ indicated that increasing the concentration of MD resulted in a higher L^* , process yield, bulk density, and b^* value, suggesting a better encapsulation of spray-dried black grape juice powder. However, it led to lower solubility of powder. In addition, a strong negative correlation was observed between L^* and moisture content ($r = -0.931$) and a^* ($r = -0.977$) at $p < 0.05$, indicating that higher MD concentration led to less hygroscopicity and efficient spray-drying. Meanwhile, lower a^* values were due to the dilution of black grape juice powder with increasing MD concentration.

Comparison between MD and MD+GA

Process yield, feed flow rate, and outlet temperature

Based on Table 2, 40% MD concentration was determined as the most efficient EA concentration, resulting in a higher process yield. Hence, 40% MD (w/v) was used for comparison between MD and MD+GA using a ratio of 8:1 (w/v) at an inlet temperature of 160°C . Table 3 describes the process yield, feed flow rate, and outlet temperature of the spray-drying process between MD and MD+GA. No significant difference was observed in the yield produced using MD and MD+GA as an EA ($p \geq 0.05$). This finding is contrary to the previous study conducted by Sarabandi *et al.* (2019), who reported that eggplant peel powder produced by the addition of 10% (w/v) MD resulted in a higher yield, compared to 10% (w/v) GA. According to Silva *et al.* (2019), this was due to GA increasing the viscosity of juice, which led to clumped and coarse powder.

The mixture's interaction between MD and GA may not significantly impact the drying behavior. Both components might retain their drying characteristics, resulting in the similar overall process efficiency (Mishra *et al.*, 2014). In addition, according to Mohsin *et al.* (2021), MD and GA can complement each other's functional properties, as MD provides the bulk and body to the mixture. In contrast, GA acts as an emulsifier or stabilizer. Their combination might not significantly alter the overall behavior of the mixture, compared to MD alone.

In this study, the outlet temperature of spray-drying ranged from 97°C to 107°C . Temperatures within this range are typically sufficient to effectively evaporate moisture from the product without requiring excessive energy, thus balancing between efficient drying and energy use. Temperatures between 80°C and 110°C are often low enough to prevent thermal degradation of sensitive components during spray-drying processes involving PRODUCTS rich in vitamins, flavors, and colors. This is particularly important in food and pharmaceutical applications.

Physical analysis of spray-dried black grape juice powder using MD and MD+GA as EA

Table 3 shows the physical analyses of spray-dried black grape juice powder produced from different EAs. The combination of MD+GA resulted in a significantly lower moisture content powder than MD alone ($p < 0.05$). Mohsin *et al.* (2022) reported similar findings in spray-dried kombucha. The authors noted that adding 10% GA produced powder with low moisture content. However, contradictory results in a spray-dried apple powder were reported by Qadri *et al.* (2022). In this case, the authors observed that MD+GA did not

Table 3. Physical analysis of spray-dried black grape juice powder using MD and MD+GA as an EA.

Type of EA	Process yield (%)	Feed flow rate (g/min)	Outlet temperature (°C)	Moisture content (% dry basis)	Hygroscopicity (% dry basis)	Bulk density (g/mL)	Solubility (s/g)	L*	a*	b*	Hue (h°)	Chroma
MD	50.06 ± 0.08 ^a	3.70 ± 0.28 ^a	97–104	1.93 ± 0.06 ^a	17.28 ± 0.43 ^b	0.31 ± 0.03 ^a	140.60 ± 1.41 ^b	82.62 ± 0.21 ^a	11.38 ± 0.01 ^b	-2.54 ± 0.04 ^a	-12.58 ± 0.14 ^b	11.66 ± 0.00 ^b
MD + GA	50.40 ± 1.27 ^a	4.06 ± 0.08 ^a	92–107	1.68 ± 0.01 ^b	20.13 ± 0.49 ^a	0.37 ± 0.01 ^a	149.40 ± 0.85 ^a	78.61 ± 0.06 ^b	14.69 ± 0.52 ^a	-2.46 ± 0.23 ^a	-9.52 ± 0.85 ^a	14.90 ± 0.34 ^a

Notes: Each value represents mean ± standard deviation (n = 3). Values with the same superscript alphabet in each row are not significantly different ($p > 0.05$). MD: maltodextrin; GA: gum Arabic; EA: encapsulating agent.

significantly influence the moisture content of spray-dried powder, suggesting that different effects might be observed even using the same EA, indicating that sample types, preparation, and different equipment influence spray-dried powder.

In this study, adding GA resulted in significantly higher hygroscopicity ($19.80 \pm 0.49\%$; $p < 0.05$). In most cases, low hygroscopicity is considered better because it reduces the tendency to absorb moisture from its surroundings. High hygroscopicity can lead to clumping, degradation, or altered properties in some materials. Previous studies on spray-dried eggplant peel extract (Sarabandi *et al.*, 2019) and roselle anthocyanins (Nguyen *et al.*, 2022) reported different findings, where adding GA reduced the hygroscopicity of powder. Differences in hygroscopic values resulted from the characteristics of powders and their capacity to draw in moisture from the surrounding air. GA is recognized for its hygroscopic characteristic, indicating its ability to absorb and hold environmental moisture (Qadri *et al.*, 2022).

There were no significant differences in the bulk density of spray-dried powder between MD and MD+GA ($p \geq 0.05$; Table 3). A study conducted by Kalušević *et al.* (2017) on soybean coat extract demonstrated no significant difference in bulk density between those encapsulated with MD or GA. Usually, increasing MD can lead to higher bulk density because of higher glucose polymers (Pui *et al.*, 2021). Meanwhile, GA has a lower bulk density, resulting in a lower bulk density of spray-dried powder. Nevertheless, the insignificant difference observed is possibly due to small GA–MD ratio (1:8); a higher MD ratio retains its high bulk density properties; and despite GA having a high bulk density, its small ratio likely explains the lack of significance in the bulk density of grape powder.

The solubility of black grape juice powder ranged from 140.60 s/g to 149.40 s/g. Powder encapsulated with MD only showed a significantly better solubility (140.60 s/g; $p < 0.05$). Nguyen *et al.* (2021) reported that MD exhibits commendable water solubility and maintains low viscosity even at high concentrations. Meanwhile, GA is characterized by a branched arrangement of simple sugars (galactose, glucuronic acid, arabinose, and rhamnose) and a small fraction of covalently bonded protein. GA is often linked with increasing the viscosity of feed solution. Thicker liquids dry more slowly because of their higher density, leading to a longer time for particles in the drying chamber. This may cause the particles to agglomerate and form clumps. Ultimately, this reduces powder solubility compared to evenly distributed particles with shorter drying periods.

Color measurements of spray-dried black grape juice powder using MD and MD+GA as EA

Table 3 shows the color properties of spray-dried black grape juice powder. The lightness (L^*) ranged from 78.61 to 82.62, redness (a^*) from 11.38 to 14.69, yellowness (b^*) from -2.46 to -2.54 , hue (h°) from -9.52 to -12.58 , and chroma from 11.66 to 14.90. The addition of GA significantly affected both L^* and a^* values ($p < 0.05$), but no significant difference was observed in the b^* value of spray-dried powder ($p \geq 0.05$). The L^* value of grape juice powder encapsulated with MD is significantly higher than that of MD+GA ($p < 0.05$). This shows that adding GA resulted in higher darkness of spray-dried powder. In addition, negative b^* values represent the bluish tint on spray-dried powder (Figure 2B).

A higher a^* value was observed after the addition of GA. As a result, the powder with MD+GA showed a slight pinkish or reddish tint compared to when only MD was applied. MD+GA has been reported to retain the color of spray-dried powder better than either MD or GA (Qadri et al., 2022). Hay et al. (2024) reported that encapsulation of anthocyanins from *Antidesma erosre* using MD and MD+GA at an inlet temperature of 150°C resulted in a significantly higher redness in powder with MD+GA ($a^* = 15.96$) than powder with MD ($a^* = 12.85$), which is similar to the current findings, suggesting that MD+GA protects the color of anthocyanins during drying processes. Hence, choosing a suitable EA is a crucial aspect of the spray-drying process, and it plays a substantial role in preserving color and the overall quality of black grape juice powder.

The hue angle of spray-dried grape juice powder using MD was significantly lower than that of powder with MD+GA, indicating a bluish-red or purplish tone of powder. In contrast, the hue of powder with MD+GA reflected a less purple or warmer red tone. These results suggest that MD+GA resulted in a color shift from purple to red, indicating better preservation of grape juice color than using MD alone. In addition, the chroma value indicating the purity color of spray-dried powder showed that using MD+GA as an EA significantly produced a more intense and saturated color than MD alone. This corroborates that GA enhances and preserves color stability and intensity in spray-dried powder.

Antioxidant properties of spray-dried black grape juice powder using MD and MD+GA as EA

Figure 3 shows the TPC, TFC, TMAC, and antioxidant activities of black grape juice powder with different EAs at an inlet temperature of 160°C . Higher values of TPC (33.36 mg GAE/100 g DM), TFC (351.39 mg QE/100 g DM), TMAC (8.69 mg/100 g DM), reducing power (351.39 $\mu\text{mol TE}/100$ g DM), and radical scavenging activity (1352.73

$\mu\text{mol TE}/100$ g DM) were observed with the MD+GA encapsulated powder than with MD alone. Laureanti et al. (2023) reported a similar finding where the combination of MD+GA (1:1) (w/v) had a better preservation of bioactive compounds in pink pepper extract. Furthermore, Nthimole et al. (2022) reported that microencapsulation of raspberry juice powder with MD+GA resulted in higher TPC, TFC, and antioxidant activities than MD or GA alone. These findings suggest that MD+GA as an EA possibly introduces a synergistic effect that results in better protection of bioactive compounds. Nevertheless, the current study only compares the antioxidant properties of spray-dried powder, and lacks information on the initial antioxidant concentration of grape juice, which can provide a clearer impact of spray-drying operation on the preservation of bioactive compounds in spray-dried powder. However, when comparing only spray-dried powders, assuming a similar antioxidant concentration of grape juice at the preparation of juice, it is notable that MD+GA is a promising approach for preserving antioxidants in grape juice.

Gum Arabic is known for its potential to act as a stabilizer and protect bioactive compounds from degrading because of environmental factors (Laureanti et al., 2023). When GA is used as an EA, it provides a protective shield around these bioactive compounds, helping to maintain their structural integrity and bioactivity (Laureanti et al., 2023). Overall, the choice of EA significantly impacts the TPC, TFC, TMAC, and antioxidant activity of spray-dried anthocyanin-rich powder. The combination of EA, such as MD+GA, is more effective at retaining the bioactive compounds and antioxidant activity than using EA alone. However, the current study did not evaluate the encapsulation efficiency of spray-dried powder. In this study, we assessed the spray-dried powders' TMAC, antioxidant activities, TFC, and TPC. These values serve as indirect indicators of encapsulation performance. The high retention of antioxidant activity and anthocyanin content after spray-drying, particularly under optimized condition concentrations and inlet temperatures, suggests effective encapsulation and protection of bioactive compounds.

Spray-dried black grape juice powder with different inlet temperatures

Process yield, feed flow rate, and outlet temperature for spray-dried black grape juice powder at different inlet temperatures

In Table 3, MD+GA, which yielded the highest levels of TMAC, TFC, and TPC, along with the most potent antioxidant activities, was used to determine the most effective inlet temperatures (150, 160, 170, and 180°C). Table 4 shows the process yield, feed flow rate, and outlet temperature of the spray-drying process over different

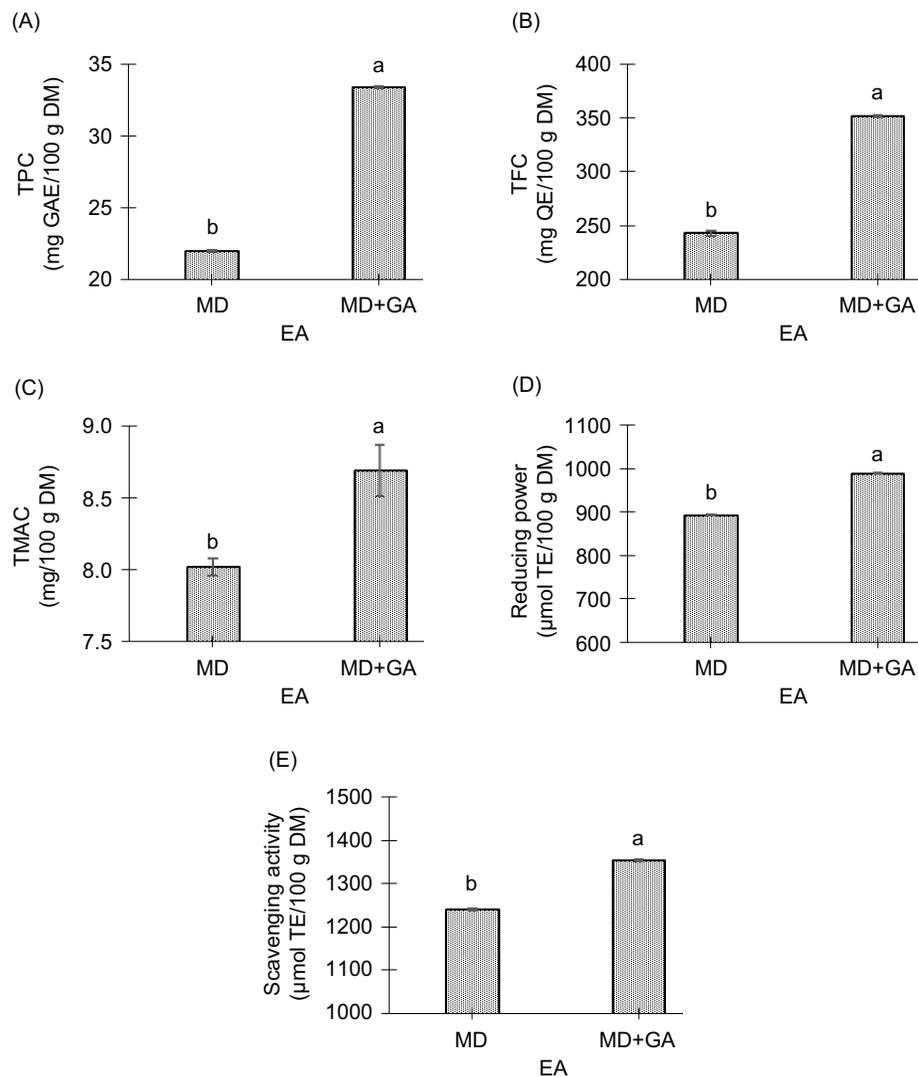


Figure 3. (A) TPC, (B) TFC, (C) TMAC, (D) reducing power, and (E) scavenging activity of black grape juice powder with different EAs at an inlet temperature of 160°C. MD: maltodextrin; GA: gum Arabic; EA: encapsulating agent; TPC: total phenolic content; TFC: total flavonoid content; and TMAC: total monomeric anthocyanin content.

inlet temperatures. The process yield of spray-dried powder significantly reduces with increase in inlet temperature, ranging from 38.25% to 54.55% ($p < 0.05$).

Black grape juice with high TSS (42.85 ± 0.07 °Brix) can become stickier or prone to caking at higher temperatures, leading to particles adhering to surfaces within the spray-drying equipment. This can reduce the efficiency of drying process and result in lower yields. Decrease in process yield at higher inlet temperatures is possibly due to rapid evaporation during drying, which can cause crust formation on powder. This can also lead to stickiness and a reduction in yield. In addition, the thermal degradation of anthocyanins at high inlet temperatures may reduce the process yield. Other reasons, such as improper

encapsulation, where rapid drying causes faster drying only on the surface of the powder, can cause the formation of crusts, which usually trap moisture and make it rupture easily. The reduced powder yield usually occurs when a higher inlet temperature is used, which leads to the rapid degradation of anthocyanins and other bioactive compounds in the sample. For instance, Decker *et al.* (2024) used an inlet temperature (120–170°C) to encapsulate grape pomace extract using MD. The authors revealed that the process yield was highest at 140°C and started to decline at 150°C because of the degradation of anthocyanins. However, the current study showed higher TMAC at a higher inlet temperature of 180°C; therefore, the degradation of anthocyanins might not be the reason for the decline in process yield in this study. Other possible

Table 4. Physical analyses of spray-dried black grape juice powder encapsulated using MD+GA at different inlet temperatures.

Inlet temperature (°C)	150	160	170	180
Process yield (%)	54.55 ± 0.07 ^a	50.40 ± 1.27 ^b	43.30 ± 0.85 ^c	38.25 ± 1.20 ^d
Feed flow rate (g/min)	3.68 ± 0.13 ^a	4.06 ± 0.08 ^a	3.79 ± 0.15 ^a	3.54 ± 0.19 ^a
Outlet temperature (°C)	88–100	92–107	98–123	102–115
Moisture content (% dry basis)	2.52 ± 0.33 ^a	1.78 ± 0.16 ^a	2.29 ± 0.27 ^a	2.00 ± 0.72 ^a
Hygroscopicity (% dry basis)	13.69 ± 0.35 ^b	20.15 ± 0.47 ^a	14.50 ± 0.37 ^b	19.60 ± 0.78 ^a
Bulk density (g/mL)	0.47 ± 0.04 ^a	0.37 ± 0.01 ^{a,b}	0.29 ± 0.01 ^b	0.32 ± 0.06 ^b
Solubility (s/g)	125.20 ± 1.41 ^b	149.4 ± 0.85 ^a	126.9 ± 2.40 ^b	129.35 ± 2.62 ^b
L*	88.12 ± 0.08 ^a	86.53 ± 0.29 ^c	14.15 ± 0.07 ^a	14.03 ± 0.25 ^a
a*	12.47 ± 0.13 ^b	13.98 ± 0.46 ^a	14.15 ± 0.07 ^a	14.03 ± 0.25 ^a
b*	3.45 ± 0.04 ^b	3.05 ± 0.08 ^c	3.54 ± 0.02 ^b	3.80 ± 0.06 ^a
Hue	15.46 ± 0.01 ^a	12.31 ± 0.06 ^d	14.05 ± 0.01 ^c	15.16 ± 0.02 ^b
Chroma	12.94 ± 0.01 ^b	14.31 ± 0.33 ^a	14.59 ± 0.05 ^a	14.58 ± 0.18 ^a

Notes: Each value represents mean ± standard deviation (n = 3). Values with the same superscript alphabet in each column are not significantly different ($p > 0.05$).

MD: maltodextrin; GA: gum Arabic.

reasons, such as higher temperatures, can also cause drying to occur instantly on the outer surface, resulting in a crust. This can trap moisture, leading to sub-optimal protection against moisture and easy rupture (Tsatsop *et al.*, 2025). Moreover, rapid drying at high temperatures may cause the targeted compounds to not be entrapped properly by EA, which influences the final recovery.

Meanwhile, the outlet temperature ranged from 88°C to 123°C. This temperature range has exceeded the range (70–90°C) suggested by Catalkaya *et al.* (2022) and Santos *et al.* (2020) to avoid degradation of heat-sensitive material. Santos *et al.* (2020) reported that an outlet temperature of 70°C during spray-drying of blackberry pomace resulted in higher anthocyanin stability. Meanwhile, Catalkaya *et al.* (2022) demonstrated that outlet temperature at 90°C resulted in a successful encapsulation, but higher temperatures led to anthocyanin degradation. However, if the powder is not exposed to temperatures exceeding 90°C for prolonged periods, further degradation of bioactive compounds can be prevented using MD+GA.

Physical analysis of spray-dried grape powder at different inlet temperatures

Based on Table 4, the moisture content of spray-dried black grape juice powder encapsulated using MD+GA ranged from 1.78% to 2.52%, with no significant differences observed across various inlet temperatures ($p \geq 0.05$). Contradicting findings were reported by Santhalakshmy *et al.* (2015), where higher inlet temperatures (160°C) produced higher moisture content (4.18 ± 0.09%) of jamun fruit juice powder.

The rise in inlet temperatures correlated with an increase in the moisture content of powders, possibly attributed to the formation of a crust on the droplet surface. In spray-drying, the drying process of a droplet unfolds in two phases. Predominant drying occurs by vaporizing free moisture from the droplet's surface. Subsequently, the drying rate diminishes due to the formation of a crust on the droplet's surface and the concentration of unbound water within the inner part of the droplet (Nguyen *et al.*, 2021).

Moreover, significantly higher hygroscopicity was observed in the powder produced at inlet temperatures of 160°C (19.80 ± 0.49%) and 180°C (19.21 ± 0.90%) ($p < 0.05$). A previous study conducted by Santhalakshmy *et al.* (2015) on jamun fruit juice powder documented a similar finding, where higher inlet temperature (160°C) resulted in higher hygroscopicity (25.33 ± 0.57%) of powder. According to Boel *et al.* (2020), higher inlet temperatures led to rapid evaporation of liquid components during spray-drying process. This rapid evaporation resulted in smaller droplets and particles with a higher surface area per unit mass. Smaller particles tend to be more hygroscopic, as they absorb moisture more readily because of their increased surface-to-volume ratio.

The bulk density of spray-dried black grape juice powder showed a decreasing trend with the increasing inlet temperatures. The powder's bulk density ranged from 0.29% to 0.47%, where 150°C significantly showed the highest bulk density of powder ($p < 0.05$). This finding is supported by Mishra *et al.* (2014), where a lower bulk density (0.49 ± 0.03%) of amla juice powder was produced

when a higher inlet temperature (200°C) was used. As inlet temperature increases, evaporation rate accelerates, thereby rapidly drying the feed mass. This induces porosity and fragmentation in powder (Sidlagatta *et al.*, 2020), reducing bulk density.

A significantly higher solubility (149.4 ± 0.85 s/g) of black grape juice powder was observed at 160°C ($p < 0.05$). These findings aligned with Santhalakshmy *et al.* (2015), where the highest solubility ($99.67 \pm 0.58\%$) of jamun fruit juice powder was obtained at 160°C. Elevated temperatures in the drying phase can induce greater porosity and fragmentation in powder particles, leading to reduced particle size and an increased surface area. This process facilitates the rapid dissolution of powder when it is introduced into a solvent. The immediate characteristic of a powder is described as its capacity to dissolve in water. Therefore, an optimal powder would exhibit rapid and thorough wetting, submerge instead of float, and disperse or dissolve seamlessly without forming lumps.

A strong negative correlation between outlet temperature and bulk density of black grape juice powder was observed ($r = -0.957$, $p < 0.05$), suggesting that spray-drying performed at increased outlet temperatures resulted in spherical hollow particles distinguished by a smooth surface and thinner walls, linked to their reduced bulk density.

Color measurements of spray-dried black grape juice powder at different inlet temperatures

Meanwhile, the color analysis of black grape juice powder with different inlet temperatures showed that the L^* value ranged from 86.53 to 88.12, a^* value from 12.47 to 14.15, b^* value from 3.05 to 3.80, hue ($^\circ$) from 12.31 to 15.46, and chroma from 12.94 to 14.59. Increasing inlet temperatures significantly affected the color properties of spray-dried powder ($p < 0.05$) (Table 4). Higher inlet temperatures produced darker powder, with high redness and yellowness (Figure 4). These findings are similar to that of Nayaka *et al.* (2020), who observed a darker and more intense red color of spray-dried avocado powder when elevated inlet temperatures were used. A study done by Bashir *et al.* (2022) on an investigation into the quality attributes of spray-dried apricot powder revealed that powders exhibiting elevated hygroscopicity demonstrated an increased capacity to absorb moisture. This subsequently led to changes in the color of the powder.

The hue value was significantly higher in spray-dried powder when an inlet temperature of 150°C was used ($p < 0.05$). However, no linear trends were observed. The lowest hue was observed at an inlet temperature of 160°C. The chroma ranged from 12.94 to 14.59. The lowest chroma was observed at an inlet temperature of

150°C. Increasing the inlet temperature did not significantly change the chroma value of spray-dried powder, suggesting that a temperature of 160–180°C can still maintain the intensity of spray-dried powder color when MD+GA was used.

A high negative correlation between L^* and hygroscopicity of spray-dried powder ($r = -0.986$, $p < 0.05$), suggested that higher hygroscopicity resulted in a lower L^* value. The study concluded that elevated temperatures, particularly above the melting point of sugar, could promote caramelization, contributing to color changes in the spray-dried powder, thus making it darker.

Antioxidant properties of spray-dried black grape juice powder at different inlet temperatures

Phenolic compounds are susceptible to oxidation during drying process. Higher drying temperatures accelerate the reaction rate of phenols, making lower drying temperatures more favorable for achieving a higher retention rate of these compounds. Volatile degradation primarily occurs through heat volatilization and dissipation. Consequently, opting for lower drying temperatures increases the retention rates of bioactive and volatile compounds (ElGamal *et al.*, 2023).

The significantly highest TPC (351.75 mg GAE/100 g DM) and TFC were observed at 160°C ($p < 0.05$) (Figure 5). Meanwhile, the lowest inlet temperature (150°C) showed the lowest TPC (220.88 mg GAE/100 g DM) and TFC (21.87 mg QE/100 g DM). Increased inlet temperature resulted in declined TPC values, thereby indicating thermal degradation of heat-sensitive polyphenols, which reduced TPC value.

Vidović *et al.* (2019) observed a declining pattern of TPC in spray-dried aronia berries at temperatures exceeding 140°C. However, the authors observed an increase in TPC value if the temperature was lower than 140°C. This discrepancy may be attributed to extremely low inlet air temperatures, causing more product retention on dryer walls and prolonging powder's exposure to hot air. Consequently, despite lower temperature, this extended exposure contributed to increased TPC losses.

However, we noted the highest TMAC (9.64 mg/100 g DM) was obtained when an inlet temperature of 180°C was used. Preserving grape juice at lower temperatures has the potential to minimize the breakdown of anthocyanins. Although elevated temperatures typically accelerate degradation reactions, the stability of powder is influenced by several factors, such as composition of grape juice, presence of co-factors, and the overall conditions of the process (Ijod *et al.*, 2024).



Figure 4. Color of spray-dried black grape juice powder at different inlet temperatures.

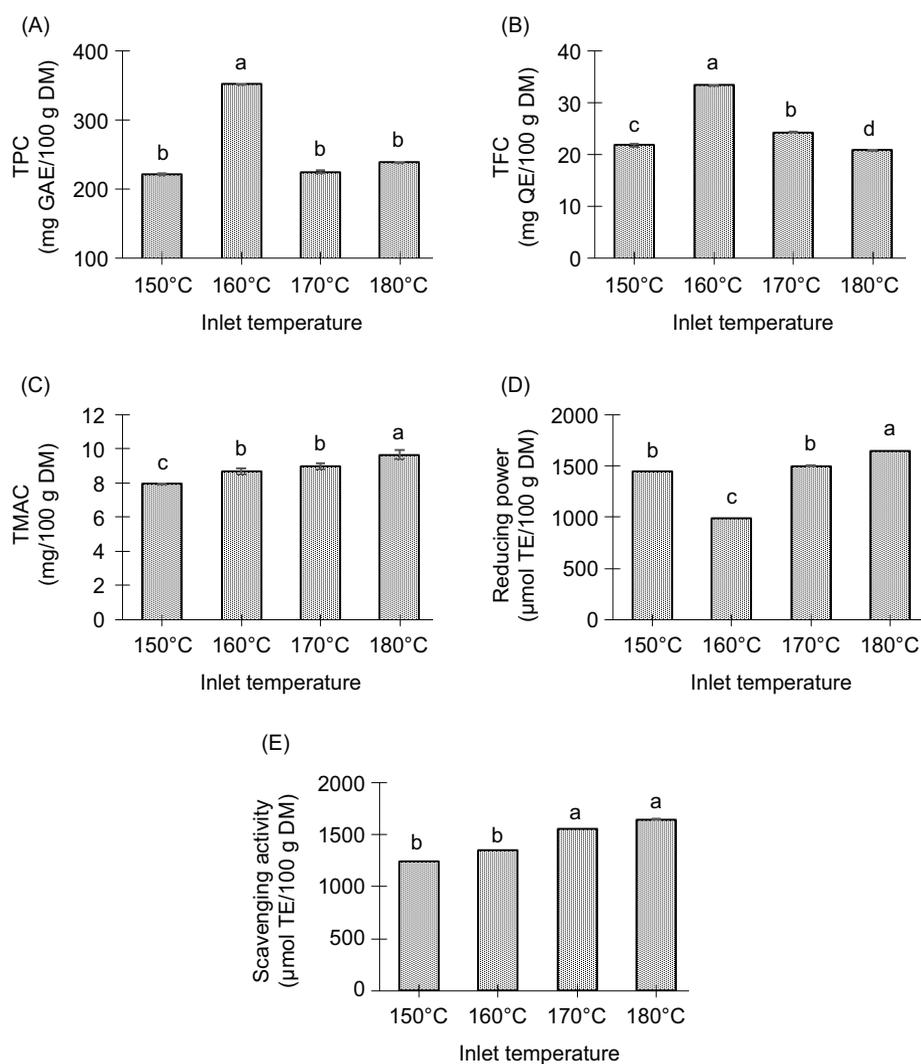


Figure 5. (A) TPC, (B) TFC, (C) TMAC, (D) reducing power, and (E) scavenging activity of spray-dried black grape juice powder with MD+GA as an EA at different inlet temperatures. MD: maltodextrin; GA: gum Arabic; EA: encapsulating agents; TPC: total phenolic content; TFC: total flavonoid content; and TMAC: total monomeric anthocyanin content.

The highest reducing power and radical scavenging activity were observed in powder at an inlet temperature of 180°C. These findings aligned with that of Vasile *et al.* (2023), who demonstrated higher antioxidants, polyphenols, and anthocyanins in roselle extract powder after using an inlet temperature of 180°C. This pattern could be linked to activating or transforming certain phenolic compounds with high antioxidant properties, leading to a high antioxidant capacity. Vasile *et al.* (2023) mentioned that higher inlet temperature promotes the release of insoluble or bound compounds with high antioxidant activity, which could be a factor for higher antioxidants in powder.

There is a strong positive correlation between TMAC and radical scavenging activity ($r = 0.952, p < 0.05$), suggesting that inlet temperature (180°C) successfully minimizes the degradation of anthocyanins and their antioxidant activities. Besides this, a strong positive correlation between feed flow rate and TPC ($r = 0.968, p < 0.05$) suggests that a higher flow rate promotes faster drying. It reduces the degradation of phenolic compounds in black grape juice powder.

Morphology of powder

Figure 6 shows the morphology of spray-dried black grape juice powder with different EAs and inlet temperatures in scanning electron microscopy (SEM). Powder encapsulated with MD+GA at an inlet temperature of 180°C showed the smoothest surface of

particles and was smaller in size. However, the particle was not completely separated and was forming a single particle. The previous findings by Mishra *et al.* (2014) and Vickovic *et al.* (2023) reported that higher temperatures are most likely to produce a smoother surface of particles because of the increased drying and water evaporation rate. In this case, the smooth surface of amla juice powder observed by Mishra *et al.* (2014), similar to spray-dried black grape juice powder, highlighted the effects of higher inlet temperature in producing smaller particles because of a rapid drying proportion. The powder encapsulated with MD at an inlet temperature of 160°C showed unseparated powder particles. In addition, the surface of powder particles showed a rough and caked surface.

Meanwhile, spray-dried grape juice powder using MD+GA at 160°C (Figure 6A) and 170°C (Figure 6C) showed a continuity of particulate matrix, possibly indicating that the spray-drying process might not have occurred well. A single and separated particle is usually produced when the atomization process works well. The MD+GA combination may soften or melt slightly at certain temperatures. This causes the particles to form a continuity, and unseparated and sticky mass. However, modifying the MD and GA proportions can overcome this issue. In addition, the use of other EAs, such as waxy starch, can improve the stability of powder during storage and reduce the degree of caking (Adetoro *et al.*, 2020).

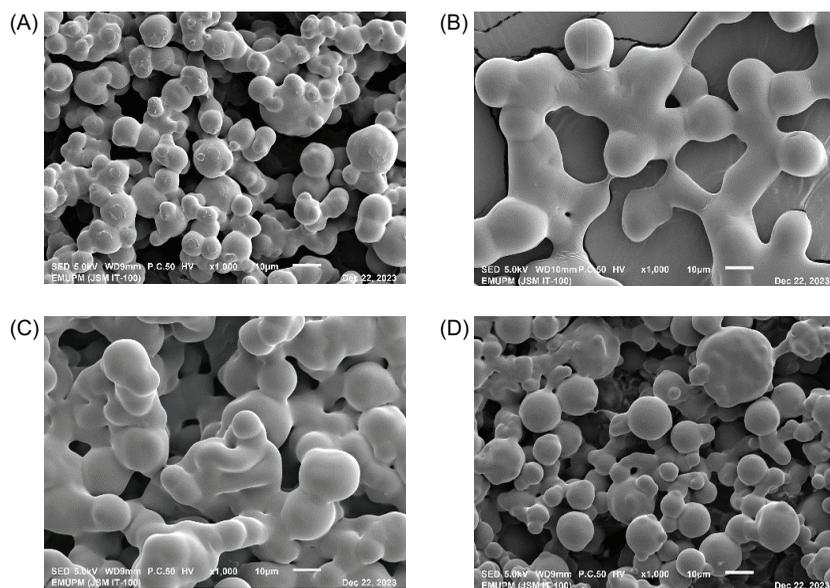


Figure 6. Micrographs of particles at different inlet temperatures and different types of EA (A) 160°C, MD, (B) 160°C, MD+GA, (C) 170°C, MD+GA, and (D) 180°C, MD+GA at 1,000× magnification. MD: maltodextrin; GA: gum Arabic; and EA: encapsulating agent.

Conclusions

The best concentrations of EAs were 40% and 50% (w/v) because of better process yield. However, 40% was selected due to the lower amount of MD used. The MD+GA combination exhibited a higher TMAC with lower moisture content than MD alone. Overall, using MD+GA at an inlet temperature of 160°C resulted in significantly higher TPC and TFC. In contrast, an inlet temperature of 180°C resulted in significantly higher TMAC and antioxidant activities with lower bulk density ($p < 0.05$). In addition, SEM analysis demonstrated that the smoothest particle surfaces were observed at 180°C, compared to other spray-dried powders at 150°C, 160°C, and 170°C. However, the particle is not entirely separated as a single particle. The process parameter can be chosen according to the desired specifications of spray-dried powder. An inlet temperature of 180°C can be used for higher retention of anthocyanins and antioxidant activities, while 160°C can be used for higher phenolic and flavonoid content. Ultimately, the choice between an inlet temperature of 160°C or 180°C depends on the specific requirements of the application. Overall, this study provides valuable insights for the food industry in selecting appropriate EAs and processing conditions to preserve the stability of anthocyanins and physicochemical properties of spray-dried black grape juice powder.

Data Availability

Data are available upon request to the corresponding author.

Author Contributions

Giroon Ijod: review and editing, visualization, and investigation. Nur Fatimah Mesran: formal analysis, writing – original draft preparation, and investigation. Nur Izzati Mohamed Nawawi: investigation, data curation, and software analysis. Mohammad Rashedi Ismail-Fitry: conceptualization, review and editing, and supervision. Muhamad Hafiz Abd Rahim: conceptualization, review and editing, and supervision. Noraniz Mohd Adzahan: conceptualization, review and editing, and supervision. Ezzat Mohamad Azman: conceptualization, review and editing, methodology, validation, supervision, and fund acquisition.

Conflict of Interest

There was no conflict of interest to declare.

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