Microencapsulation of *Hibiscus sabdariffa* (Roselle) Extracts by Spray Drying Using Maltodextrin and Gum Arabic as Carriers

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Abstract

Microencapsulation by spray drying is one of the most common methods used to obtain food material powders. In this study, different gums (maltodextrin [MD], gum arabic [GA], and mixtures of MD:GA [60:40] at various concentrations [0–10% w/w]) were used to microencapsulate *Hibiscus sabdariffa* (Roselle) extracts by spray drying. The yield, physicochemical properties, and antioxidant characteristics (total monomeric anthocyanins [TMAs], total phenolic compounds [TPCs], and antioxidant capacity [AC]) of the microencapsulated Roselle powders (RP) were evaluated. The highest RP yield (73.3 ± 3.3%) was obtained with the 3% MD:GA blend. The red color (a^*) average for all powders (39.9 ± 2.0) decreased as the gum concentration increased. The 3% MD:GA RP showed the highest amount of TMAs (539.19 ± 13.27 mg cyaniding-3-glucoside equivalents/100 g) and TPCs (3,801.6 ± 125.9 mg of gallic acid equivalents/100 g of powder). The highest AC was observed with a 5% GA RP (1498.5 ± 44.0 mg of Trolox equivalents/100 g of powder).

Keywords: microencapsulation, spray drying, *Hibiscus sabdariffa*, Roselle, anthocyanins, phenolic compounds, antioxidant capacity

1. Introduction

Hibiscus sabdariffa is a plant in the Malvaceae family. It grows in tropical and subtropical regions and can have green or red calyces (Cissé et al., 2009). The red color of calyces reflects the high anthocyanin, mainly delphinidin-3-sambubioside (71.4%) and cyanidin-3-sambubioside (26.6%) content (Peng-Kong et al., 2002). These compounds are highly unstable and degrade easily, producing compounds with an undesirable color (browning). One of the main attributes of food quality is color and consumer acceptance depends greatly on color. Anthocyanins are colorful pigments from vegetable products; the stability of anthocyanins depends on various factors such as temperature, pH, oxygen, light, enzymes, and metallic ions (Idham et al., 2012; Ersus et al., 2007).

One technique that has been used to maintain the stability of pigments such as anthocyanins is microencapsulation. Microencapsulation of food materials is used to reduce pigment degradation due to environmental factors such as oxygen, light, temperature, and prooxidant agents, to increase stability during processing, to control their release, or as a food additive (Santos and Meireles, 2010). Encapsulating agents include natural polymers and derivatives of these or lipids. The most common of these are gum arabic (GA), carrageenin, maltodextrins (MDs), cyclodextrins, dextrins, chitosan, gelatin, sodium caseinate, pregelatinized starch, carboximethylcellulose, methylcellulose, hydroxypropyl methylcellulose, milk proteins (caseins and whey), lactose, corn syrup (Munin and Edwards-Lévy, 2011; Selim et al., 2008; Kolanowski et al., 2006; Vega and Roos, 2006), and mesquite gum (Ochoa-Velasco et al., 2017). The ideal substances for microencapsulation are soft and unflavored and have high solubility, emulsifying properties, and characteristics that promote drying. Furthermore, their concentrated solutions should have low viscosity to facilitate drying (Vega and Roos, 2006). MDs and GA are the encapsulating agents most commonly used with spray drying. MDs solubilize rapidly and have low viscosities at high concentrations; however, their emulsifying capacity is limited. MDs in the range of

10–20 dextrose equivalents (DEs) are the most suitable for use as encapsulating agents. On the other hand, GA is a very efficient encapsulating agent; it is a polymer with 2% of its structural proteins lending excellent emulsifying properties. However, high concentrations of GA increase the viscosity of solutions (Gharsallaoui et al., 2007). Combinations of MDs with GA used with spray drying have been shown to produce good powders (Zhang, Mou, and Du, 2007; Lopez et al., 2009; Idham et al., 2012; Fazaeli et al., 2012).

Microencapsulation is a widely used process in the food, pharmaceutical, and cosmetics industries, as well as in agricultural, veterinary, medical, chemical, biotechnological, and biomedical fields. Spray drying is a widely used economical method for encapsulating food ingredients. Particle sizes of powders obtained by this method are generally in the range of 10–50 μm; however, this size may depend on the process conditions (Gharsallaoui et al., 2007). The main advantages of this process, besides its simplicity, are its suitability for use with heat-sensitive materials because the time required at high temperatures is very short (5–30 s) (Ochoa-Velasco et al., 2017), the equipment needed is readily available, options for encapsulating materials are many, the encapsulation process is efficient, the final product is stable, and there is the potential for continuous large scale production (Santos and Meireles, 2010). The parameters that have the most influence in the spray drying process are nozzle geometry, viscosity of the feeding solution, and the inlet and outlet air temperatures (Munin and Edwards-Lévy, 2011; Gharsallaoui et al., 2007). Commercially, this technique has been used to encapsulate numerous materials, including flavor agents, fats, oils, vitamins, minerals, microorganisms, enzymes, sweeteners, and colorants (Wijaya et al., 2011).

There is little research on spray drying of Roselle extracts (REs) to obtain powders. Some researchers have found that powders obtained by spray drying using encapsulating agents (mainly MDs) are more stable and have longer lasting antioxidant properties than those obtained without encapsulating agents (Langrish and Chiou, 2008a, 2008b; Farimin and Nordin, 2009; Ochoa-Velasco et al., 2017).

The aim of this work was to evaluate the use of an MD, GA, and combination of the two as encapsulating agents to obtain Roselle powders by spray drying.

2. Materials and Method

2.1 Materials

Red Creole Roselle calyces (long variety) were acquired from Chiautla de Tapia, Puebla, Mexico to produce extracts. Roselle calyces powder (RCP) was obtained using a stainless steel VeyCo MPV Mill Model 100 (Mexico). The mill has a mesh of 0.5 mm.

2.2 Methods

2.2.1 Average Particle Size

Average particle size (APS) was determined using a Microtac S3500 Particle Size Analyzer (Microtac Inc., Largo, FL, USA) with a measuring range of 0.25–2800 μm. Approximately 30 and 60 mg of spray-dried Roselle powders (RPs) and RCP were used, respectively, to assess particle size. The analysis was performed in triplicate. Particle size distributions, mean diameters of particles (d_{50}), and cumulative weight curves were obtained for the different powders (O'Hagan et al., 2005).

2.2.2 Concentrated RE

RE was obtained with a 1:10 RCP:ethanol ratio (20 g of RCP + 200 mL of 50% ethanol) at 50 ± 0.2 °C for 30 min using a Riossa M80T water bath (Rios Rocha S. A., Monterrey, Nuevo Leon, Mexico), based on a method described by Salazar-González, Vergara-Balderas, Ortega-Regules, and Guerrero-Beltrán (2012). RE was filtered through Whatman paper No. 4, and the liquid was obtained in flasks that were then covered with aluminum foil (Cid-Ortega and Guerrero-Beltrán, 2014). To obtain concentrated REs, alcohol was removed using a Büchi rotary evaporator RE 111 (Brinkmann Instruments Inc., Switzerland) at 45 °C and 54 cmHg for no more than 45 min (Selim et al., 2008). The concentrated RE was evaluated according to volume, weight, and physicochemical (total soluble solids [TSSs], density, viscosity) and antioxidant (total monomeric anthocyanins [TMAs], total phenolic compounds [TPCs], and antioxidant capacity [AC]) characteristics.

2.2.3 Concentrated RE-gums

A 3×3 factorial design with the type or blend of gum (GA, MD, and an MD:GA [60:40] blend) and their concentration (3, 5, and 10% w/w) was used (Idham et al., 2012). GA was acquired from Central de Drogas, S. A. de C.V. (Mexico State, Mexico). MD of 9–14 DEs was acquired from CP Ingredientes S. A. de C.V. (Guadalajara, Jalisco, Mexico). The gum or blend of gums was added to the RE concentrate and stirred for 15 min at room temperature (22 ± 2 °C). Each RE-gum concentrate was placed in a 250-mL Erlenmeyer flask, covered with

aluminum foil, and stored a 4 ℃ until spray drying. RE and RE-gum concentrates were analyzed for physicochemical (TSSs, density, viscosity) and antioxidant (TMAs, TPCs, and AC) characteristics.

2.2.4 Spray Drying

A mini spray dryer (Büchi B-290, Switzerland) with a two-fluid nozzle with an orifice 0.7 mm in diameter (particle diameter of 1–25 microns) was used for spray drying. The inlet and outlet air temperatures were 180.01 ± 0.25 and 105.16 ± 3.52 ℃, respectively. Blends of RE-gum concentrates were fed into the dryer at a flow rate of 10 mL/min (Andrade and Flores, 2004; Ochoa-Velasco et al., 2017). The aspirator power of the drying system was 100% (equivalent to an airflow of 35 m³/h), and the spray drying airflow was maintained at 55 mm (equivalent to 670 L/h with a pressure of 1.05 bar). Calibration curves were constructed (10–100% pump power) to determine the percentage equivalent to 10 mL/min for each mixture (38–41%). RPs were weighed and placed in 100-mL amber glass bottles, and these bottles were capped and stored at room temperature (22 ± 2 ℃) in a desiccator containing silica. Yield, productivity, physical characteristics (moisture content, water activity [aw], average diameter, bulk and tapped densities, and color), and antioxidant characteristics (TMAs, TPCs, and AC) of RPs were determined.

2.2.5 Physical Properties of Extracts

Total soluble solids (TSSs). TSSs were measured according to the 932.14C AOAC (1995) method using a handheld refractometer (Atago Co. LTD, Tokyo, Japan). To correct values for 20 ℃, a standard set of tables were used (AOAC, 1995).

Density. Density was determined according to the 945.06 AOAC (1995) method and expressed in g per mL. Empty (W_1), filled with distilled water (W_2), and filled with sample (W_3) pycnometer weights were determined, and the density at 25 ℃ was calculated according to Eq. (1):

$$\rho(g \ / \ mL) \ = \ \left[\frac{W_2 \ - \ W_1}{W_3 \ - \ W_1} \right] \ast \ \rho_{H_2O}^{25°C} \tag{1}$$

where $\rho_{H_2O}^{25°C}$ (g/mL) is the density of water at 25 ℃.

Absolute viscosity (μ). A Cannon-Fenske capillary viscometer (Cannon Instrument Co., State College, PA, USA) was used to determine absolute viscosity. Kinematic viscosity was calculated by multiplying the time (s) of 6.6 mL of extract at 40 ℃ flowing through the viscometer per the constant of the apparatus (0.4754 mm²/s²) at the same temperature. To obtain absolute viscosity, kinematic viscosity was multiplied by the density of the extract according to Eq. (2) (Cannon Instrument Co., 2000):

$$\mu(cP) \ = \ \rho_s \ast \ \upsilon_c \tag{2}$$

where μ is the absolute viscosity (cP = mPa·s), ρ_s is the density (g/mL), and υ_c is the kinematic viscosity (mm²/s = cSt) of an extract. The absolute viscosity at 25 ℃ was calculated using Eq. (3) (Cannon Instrument Co., 2014):

$$C \ = \ C_o \left[1 \ - \ B \left(T_t \ - \ T_f \right) \right] \tag{3}$$

where C (0.4754 mm²/s²) is the constant of the apparatus at 40 ℃, C_o (mm²/s²) is the viscometer constant at the filling temperature, B (79×10⁻⁶/ ℃) is the calibration temperature factor obtained from the calibration certificate for the viscometer, T_t is the working temperature (40 ℃), and T_f is the filling temperature. Using the equation above, the constant C_o was calculated and then, using the same equation, the constant C was calculated at 25 ℃.

2.2.6 Antioxidant Properties

Total monomeric anthocyanins (TMAs). TMAs were determined according to the method described by Lee et al. (2005). First, 0.5 mL or 100 mg of extract or powder, respectively, were diluted with distilled water to reach 10 mL in a volumetric flask. The mixture was stirred for 5 min using a vortex at 2900–3000 rpm. One milliliter of each solution was diluted with buffer pH 1.0 or pH 4.5 to reach 5 mL in test tubes wrapped with aluminum foil. The blends were left for 30 min at room temperature (23 ± 2 °C) in the dark. Then, absorbances in 4-mL glass cells were measured at 520 and 700 nm using a Cary 100 UV-Vis spectrophotometer (Varian Inc., Palo Alto, CA, USA). A blank with distilled water was used to correct these absorbances. Results were calculated as mg of cyanidin-3-glucoside equivalents per 100 mL of RE or per 100 g of powder using Eq. (4):

$$TMA = \frac{A*MW*DF}{\varepsilon*L} * 100 \tag{4}$$

where *TMA* is the concentration of anthocyanins (mg/100 mL or mg/100 g), $A = (A_{520nm} - A_{700nm})_{pH=1.0} - (A_{520nm} - A_{700nm})_{pH=4.5}$, *MW* is the molecular weight of cyanidin-3-glucoside (449.2 g/mole), *DF* is the dilution factor, *L* is the cell width (1 cm), ε is the coefficient of molar extinction for cyanidin-3-glucoside (26,900 L/mole-cm), and 100 is the conversion factor for obtaining mg/100 mL of RE or mg/100 g of RP.

Total phenolic compounds (TPCs). TPCs were determined using the Phenol Folin and Ciocalteu method (Singleton and Rossi, 1965) with some modifications. Three milliliters of distilled water, 150 μL of extract solution, or 100 μL of powder solution (the same solutions prepared to determine TMAs), and 250 μL of Folin and Ciocalteu reagent were placed in test tubes that were then covered with aluminum foil. Mixtures were stirred and left for a maximum of 8 min in the dark, and then 750 μL of 20% Na_2CO_3 was added and thoroughly mixed. Distilled water was added (850 or 900 μL) to reach 5 mL, mixed thoroughly, and left for 2 h at room temperature (23 $\pm$ 2 ℃) in the dark. Absorbances were measured at 765 nm using a Cary 100 UV-visible spectrophotometer (Varian Inc.). Various standard curves were constructed with different concentrations of gallic acid (0–0.066 mg; Sigma, St. Louis, MO, USA). The standard curve for extracts and powders is represented by the equation A = 18.810 $\pm$ 1.463 (1/mg GA) * X (mg GA) + 0.023 $\pm$ 0.007 (R^2 = 0.998 $\pm$ 0.001). The amount of TPCs was calculated as mg of gallic acid equivalents per 100 mL of RE or per 100 g of powder according to Eq. (5):

$$TPC \left(\frac{mg}{100\,mL}\right) = \left(\frac{A-b}{m}\right) * DF * 100 \tag{5}$$

where *TPC* is the total phenolic compounds content (mg/100 mL or mg/100 g), *A* is the absorbance of the sample, *b* is the intercept, *m* is the slope, and *DF* is the dilution factor for the sample.

Antioxidant capacity (AC). The DPPH (1,1-diphenyl-2-picrylhydrazyl) method (Brand-Williams et al., 1995) was used with some modifications (Molyneux, 2004; Esmaeili et al., 2015). Two milliliters of extract solution or 100 μL of powder solution (the same solutions prepared to determine TMAs) were diluted with ethanol (99.5%) to reach 10 mL in a volumetric flask, stirred for 5 min using a vortex (2900–3000 rpm), and then filtered through Whatman paper No. 4. One milliliter of filtrate was placed in a test tube containing 1 mL of ethanol (99.5%) and 2 mL of DPPH solution (7.99 $\pm$ 0.24 mg in 200 mL of 99.5% ethanol). The solutions were thoroughly mixed and left for 45 min at room temperature (21.6 $\pm$ 3.3 ℃) in the dark. Absorbances were measured at 517 nm using a Cary 100 UV-Vis spectrophotometer (Varian Inc.). The AC of each solution was calculated using Eq. (6):

$$I\,(\%) = \frac{Ac-As}{Ac} * 100 \tag{6}$$

where *I* is the percent of inhibition, *As* is the absorbance of the sample and *Ac* is the absorbance of the control. Standard curves were prepared at various concentrations (0, 0.008–0.030 mg) of Trolox (6-hydroxy-2, 5, 7, 8 tetrametilcromo-2 carboxylic acid 97%). The standard curves for extracts and powders are represented by the equation I (%) = 3272.18 $\pm$ 220.83 (1/mg TE)*X (mg TE) + 0.70 $\pm$ 4.06 (R^2 = 0.978 $\pm$ 0.014), and I (%) = 3269.52 $\pm$ 251.15 (1/mg TE)*X (mg TE) + 4.78 $\pm$ 4.37 (R^2 = 0.964 $\pm$ 0.026), respectively. Results, calculated according to Eq. (7), were expressed as milligrams of TEs per 100 mL of RE or 100 g of powder.

$$AC \left(\frac{mg}{100\,mL}\right) = \left(\frac{A-b}{m}\right) * DF * 100 \tag{7}$$

where *AC* is the antioxidant capacity (mg/100 mL or mg/100 g), *A* is the absorbance of the sample, *b* is the intercept, *m* is the slope, and *DF* is the dilution factor of the sample.

2.2.7 Physicochemical Properties of Powders

Yield (Y). Yield was calculated based on the amount of TSSs in the encapsulated extract and the amount of powder obtained (Fazaeli et al., 2012) according to Eq. (8):

$$Y\,(\%) = \frac{Amount\,of\,powder}{Amount\,of\,TSS} * 100 \tag{8}$$

Moisture content. Moisture content was measured according to the 934.06 AOAC (2000) method. A Cole-Parmer (Chicago, IL, USA) vacuum oven was used to dry samples for 8 h at 70 $\pm$ 1 ℃ and a vacuum pressure of 200–220 mmHg. Moisture content was calculated as a percentage.

Water activity (a_w). Water activity was measured using an AQUA-LAB hygrometer model 3TE (Decagon

Devices Inc., Pullman, WA, USA). The temperature at the time of measurement was 25.10 $\pm$ 0.06 ℃.

Bulk density. Bulk density was measured according to the method described by Jumah et al. (2000). One gram of powder was weighed in a 10-mL graduated cylinder. The cylinder was gently tapped 10 times on a polystyrene mat from a height of 15 cm. Bulk density (ρ_a) was calculated according to Eq. (9):

$$\rho_a(g \ / \ mL) \ = \ \frac{W}{V_a} \tag{9}$$

where *W* is the weight of powder (g) and *Va* is the apparent volume (mL) occupied by the powder in the cylinder after tapping.

Tapped density. Tapped density was measured according to the Mexican Official Norm number NOM-104-STPS-2001 (NOM, 2001) with some modifications. One gram of powder was weighed in a 10-mL graduated cylinder with a rubber stopper. The cylinder was subjected to a manual vibration process so that the sample were shaken from bottom to top for 8 min (estimated time at maximum volume). The tapped density (ρ_c) was calculated according to Eq. (10):

$$\rho_c \ (g/mL) = \frac{W}{Vc} \tag{10}$$

where W is the weight of powder (g) and Vc is the compacted volume (mL) occupied by the powder in the cylinder after tapping.

Color. A Colorgard system 05 colorimeter (BYK-Gardner Inc., Silver Spring, MD, USA) was used to determine the color of powders and solutions. For powders, the color parameters were obtained in reflectance mode. A plate with a light gap 1.9 cm in diameter and external diameter of 2.65 cm was used. Samples were placed in weighing bottles for color determination. For solutions, a solution of 10 mg of powder/mL of distilled water was prepared, and color parameters were determined in transmittance mode using a 3-mL quartz cell (Konica Minolta Sensing, Inc., Kyoto, Japan) (Ochoa-Velasco et al., 2017; Silva et al., 2013). Color parameters of powders and solutions were obtained using the *CIEL*a*b** scale: *L** (lightness, 0–100), *a** (green to red) and *b** (blue to yellow). From these data, purity (color saturation, $C = [a^{*2} + b^{*2}]^{1/2}$) and hue ($H = \tan^{-1}[b^*/a^*]$) were calculated.

2.2.8 Statistical Analysis

Data were subjected to analysis of variance (ANOVA) testing using MINITAB software version 14.1. Multivariate analysis and Tukey's multiple comparison tests were used to compare differences between means. Values shown are average values. A value of 0.05 was considered significant for differences between means of treatments.

3. Results and Discussion

3.1 Antioxidant Characteristics of RE Concentrates

Table 1 shows the characteristics of RE concentrates. Roselle extracts had average initial volumes, weights, and TSSs of 73.10 $\pm$ 3.09, 7.83 $\pm$ 4.38, and 15.04 $\pm$ 0.80, respectively, and average TMAs, TPCs, and ACs of 84.10 $\pm$ 4.77, 629.97 $\pm$ 30.25, and 257.00 $\pm$ 10.68, respectively (Table 2). Statistically significant differences in TMA contents were observed (p > 0.05) for all extracts (70.65–91.26 mg/100 mL). Extracts with 3 and 5% GA and 3 and 5% MD added (462.43 $\pm$ 22.41, 457.66 $\pm$ 30.48, 466.53 $\pm$ 17.18, and 456.53 $\pm$ 28.07 mg/100 mL extract, respectively) showed less (p $\leq$ 0.05) TPCs than those in RE alone. Extracts with 3% GA and 10% MD added (227.32 $\pm$ 5.12 and 220.99 $\pm$ 15.71 mg/100 mL extract, respectively) showed significantly lower ACs than RE alone. Significant differences (p $\leq$ 0.05) in ACs for all RE-gum concentrates.

Table 1. Roselle extract concentrate and gum required for each Roselle extract-gum concentrate

Gum	Concentration (% w/w)	Gum (g)	Extract (mL)	Extract (g)	TSSs[1] (%)
RE	0	–	73.10 ±3.09	73.83 ±4.38	15.04 ±0.80
GA	3	2.77 ±0.19	88.83 ±6.53	89.43 ±6.29	16.78 ±0.03
	5	4.35 ±0.63	82.00 ±12.49	82.67 ±12.02	17.54 ±0.48
	10	9.22 ±0.64	82.17 ±6.05	82.97 ±5.75	17.18 ±0.29
MD	3	2.63 ±0.07	83.34 ±2.36	83.84 ±2.37	17.15 ±0.16
	5	4.64 ±0.28	85.21 ±5.03	85.21 ±5.03	16.91 ±0.81
	10	9.60 ±0.70	84.33 ±6.81	85.36 ±6.32	17.06 ±0.46
MD:GA	3	2.18 ±0.18	67.43 ±6.02	69.75 ±6.02	14.39 ±0.37
	5	3.97 ±0.06	72.51 ±0.87	74.77 ±0.93	14.34 ±0.11
	10	8.72 ±0.49	76.54 ±5.24	77.53 ±4.95	17.31 ±0.43

[1]TSSs: total soluble solids (at 20 ℃) in extracts without gum. RE, Roselle extract; GA, gum arabic; MD, maltodextrin.

Table 2. Antioxidant characteristics for Roselle extract concentrates[a]

Gum	Concentration	TMA[1]	TPC[2]	AC[3]
	(% w/w)		(mg/100 mL)	
RE	0	84.10 ±4.77abc	624.97 ±30.25a	257.87 ±10.68abc
GA	3	76.11 ±3.31ac	462.43 ±22.41c	227.32 ±5.12bc
	5	82.07 ±10.98abc	457.66 ±30.48c	278.59 ±41.77ab
	10	86.04 ±7.69ab	491.32 ±19.18c	235.26 ±15.01abc
MD	3	74.11 ±3.78bc	466.53 ±17.18c	251.42 ±4.42abc
	5	70.65 ±3.37c	456.53 ±28.07c	268.98 ±10.47abc
	10	76.53 ±1.91abc	499.92 ±13.04c	220.99 ±15.71c
MD:GA	3	90.95 ±0.22a	617.62 ±28.86a	281.15 ±25.60a
	5	91.26 ±1.50a	519.11 ±9.12abc	263.80 ±3.49abc
	10	83.50 ±4.02abc	590.76 ±43.98ab	251.72 ±13.90abc

[a]Different letters in the same column indicate significant differences (p ≤ 0.05) between values. [1]TMAs: total monomeric anthocyanins (cyanidin-3-glucoside equivalents). [2]TPCs: total phenolic compounds (gallic acid equivalents). [3]AC: antioxidant capacity (Trolox equivalents). RE, Roselle extract; GA, gum arabic; MD, maltodextrin.

3.2 RE-gum Concentrates

3.2.1 Physical Properties

TSSs (°Bx). Significant differences (p ≤ 0.05) were observed between average TSS contents (Table 3) for REs with GA, MD, or MD:GA added. The TSS content in REs was 15.04 ± 0.80. An increase of gums in REs increased TSS content (Table 3).

Table 3. Physical characteristics of Roselle extract concentrates[a]

Gum	Concentration (% w/w)	TSSs (°Bx)	Density (g/mL)	Viscosity (mPa s)[1]
RE	0	15.04 ±0.80e	1.03 ±0.01ab	1.66 ±0.01g
GA	3	19.03 ±0.19c	1.02 ±0.01b	2.79 ±0.05fd
	5	20.86 ±0.48b	1.03 ±0.02ab	3.68 ±0.01c
	10	24.14 ±0.17a	1.05 ±0.01ab	8.44 ±0.1a
MD	3	19.53 ±0.22bc	1.02 ±0.01b	2.07 ±0.02f
	5	20.73 ±0.39b	1.03 ±0.01ab	2.29 ±0.07e
	10	24.54 ±0.08a	1.05 ±0.01ab	2.99 ±0.07d
MD:GA	3	17.03 ±0.77d	1.05 ±0.01ab	1.94 ±0.01f
	5	18.73 ±0.59c	1.05 ±0.00ab	2.33 ±0.04e
	10	24.90 ±0.23a	1.06 ±0.01a	4.39 ±0.09b

[a]Different letters in the same column indicate significant differences (p ≤ 0.05) between values. [1]Viscosity at 25 ° C. TSSs, total soluble solids; RE, Roselle extract; GA, gum arabic; MD, maltodextrin.

Density. Densities of RE-gum concentrates (Table 3) showed significant differences ($p \leq 0.05$) among types and concentrations of gum. Extracts with the MD:GA blend added were denser (1.06 ± 0.01 g/mL) than extracts without gum (RE) or with GA and MD (1.03 ± 0.01, 1.04 ± 0.02, and 1.03 ± 0.01 g/mL, respectively). Densities of RE-gum concentrates with the three concentrations of gums also showed significant differences ($p \leq 0.05$).

Viscosity. Viscosities of RE-gum concentrates are shown in Table 3. The viscosity of RE-gum concentrates increased as gum concentration increased (1.66 ± 0.01, 2.26 ± 0.40, 277 ± 0.69, and 5.27 ± 2.45 mPa·s, for 0, 3, 5, and 10% gums, respectively). Extracts with 10% GA showed the highest viscosity, perhaps because GA has the ability to form gels due to its protein contents (Lopez et al., 2009). Significant differences ($p \leq 0.05$) in RE-gum concentrate viscosities were observed among concentrations and types of gum. Viscosity and TSS content are important for spray drying because low viscosities along with high TSS content results in better flow during atomization and higher yields (Lopez et al., 2009). Therefore, a positive correlation between viscosity and TSS content was observed with each treatment: GA ($R^2 = 0.938$), MD ($R^2 = 0.988$), and MD:GA ($R^2 = 0.980$).

3.4 Roselle Powders (Rps)

3.4.1 Granulometry of RPs

Figure 1 shows particle size distribution and cumulative percentages of RPs. The average diameter (d_{50}) of particles was 221.03 ± 3.97 mm and the moisture content was $6.45 \pm 0.43\%$.
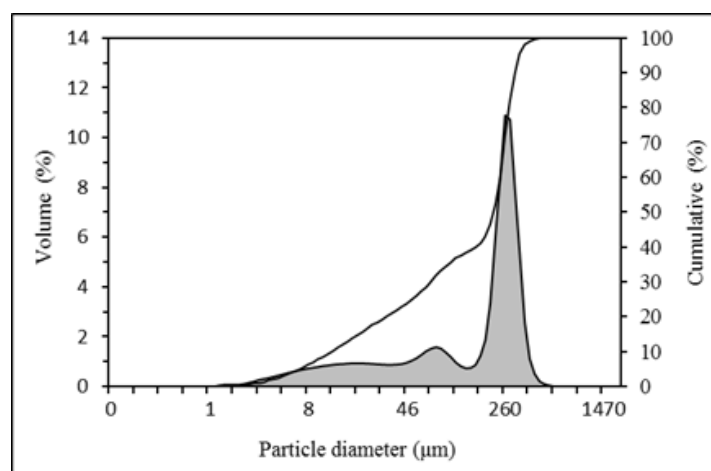

Figure 1. Particle size distribution of Roselle calyces powders

3.4.2 Yield

The yield for powder without gum was 58.19 ± 5.06cd%. The yields for powders with GA were 59.78 ± 2.47cd, 65.18 ± 5.25bc, and 68.77 ± 2.22ab% with 3, 5, and 10% gums, respectively. The yields for powders with MD were 56.46 ± 0.97d, 59.81 ± 1.49cd, 72.07 ± 2.12ab% with 3, 5, and 10% gums, respectively. The yields for powders with MD:GA were 76.38 ± 3.24a, 72.75 ± 1.16ab, and 70.79 ± 2.60ab% with 3, 5, and 10% gums, respectively. Regarding gum type, MD:GA RP showed the highest yield ($73.3 \pm 3.3\%$), and the RE and GA and MD RPs showed similar ($p > 0.05$) lower yields of 58.19 ± 5.06, 59.78 ± 2.47, and $59.81 \pm 1.49\%$, respectively. Significant differences ($p \leq 0.05$) in yields were observed with 3, 5, and 10% gums (64.2 ± 9.5, 65.9 ± 6.3, and $70.5 \pm 2.5\%$, respectively). The control RP (of RE) showed a low yield ($p \leq 0.05$). Ochoa-Velasco et al. 2017) obtained an average yield of $73.7 \pm 1.5\%$ for RPs microencapsulated by spray drying using mesquite gum at different concentrations (1, 2, 3, 4, and 5% w/v).

3.4.3 Physicochemical Characteristics

Moisture content. In general, RPs with the highest ($p > 0.05$) moisture contents were the following: 3.34 ± 0.30 and $3.29 \pm 0.27\%$ for 3 and 5% MD:GA, respectively; 3.29 ± 0.27 for RE; and 3.09 ± 0.24 and $2.29 \pm 0.15\%$ for 10 and 3% GA, respectively (Table 4). No significant differences ($p > 0.05$) were observed in moisture contents with gum concentrations of 0, 3, 5, and 10% (3.29 ± 0.27, 2.68 ± 0.57, 2.48 ± 0.68, and $2.60 \pm 0.41\%$, respectively). Gonzales-Palomares et al. (2009) reported a moisture content of 4% in spray-dried powders using inlet and outlet temperatures of 180 and 80 °C, respectively, for control REs, which is similar to values obtained

in this study (3.29 $\pm$ 0.27%). Likewise, Ochoa-Velasco et al. (2017) reported an average moisture content in spray-dried microencapsulated powders from REs using mesquite gum as an encapsulating agent at different concentrations (1, 2, 3, 4, and 5% w/v) of 2.29 $\pm$ 0.45%. This value is similar to the average moisture content (2.59 $\pm$0.55%) in RPs with different gums obtained in this study.

Table 4. Effect of gum type and concentration on the physicochemical properties of Roselle powders[a]

Gum type	GC(% w/w)	Moisture (%)	a_w (at 25.1 $\pm$0.06 °C)	Average diameter d_{50} (μm)	Bulk density (g/mL)	Tap density (g/mL)
RE	0	3.29±0.27a	0.183±0.012def	12.16±1.01cd	0.380±0.020a	0.483±0.021b
GA	3	2.59±0.15bc	0.167±0.012ef	9.69±0.59abe	0.220±0.010c	0.303±0.006b
	5	2.36±0.30bcd	0.210±0.010bcd	12.15±0.20cd	0.150±0.010d	0.233±0.015e
	10	3.09±0.24ab	0.247±0.006a	14.92±0.25ab	0.153±0.012d	0.250±0.017e
MD	3	2.12±0.19cd	0.153±0.015f	9.05±0.49c	0.240±0.010c	0.307±0.015b
	5	1.79±0.18fd	0.173±0.006ef	9.69±0.54de	0.163±0.012b	0.233±0.021e
	10	2.48±0.16bc	0.163±0.015f	13.93±0.51abc	0.110±0.000e	0.160±0.000f
MD:GA	3	3.34±0.30a	0.200±0.020cde	9.12±0.17e	0.353±0.021a	0.540±0.035a
	5	3.29±0.12a	0.230±0.010abc	12.42±1.53bc	0.300±0.010b	0.370±0.010c
	10	2.23±0.08cd	0.240±0.010ab	16.20±2.02a	0.097±0.012e	0.133±0.015f

[a]Different letters in the same column indicate significant differences (p ≤ 0.05) between values. GC, gum concentration; RE, Roselle extract; GA, gum arabic; MD, maltodextrin.

Water activity (a_w). The stability of many foods depends on water activity (Fennema, 1985). High a_w indicates high free water content and thus low food stability. Table 4 shows low a_{ws} for all RPs. Average a_{ws} for RPs with MD, GA, and MD:GA were 0.163 $\pm$ 0.014, 0.208 $\pm$ 0.036, and 0.223 $\pm$ 0.022, respectively. Significant differences (p ≤ 0.05) were observed among types and concentrations of gums. The highest values were observed with 10% GA.

Average diameter (d_{50}). Significant differences were observed between mean diameters of RE, GA, MD, and MD:GA RPs (12.16 ± 1.01, 12.25 ± 2.29, 10.89 ± 2.34, and 12.58 ± 3.32 μm, respectively (Table 4). Diameters increased significantly (0.5 $\pm$ 9.29, 11.42 $\pm$ 1.54, and 15.35 $\pm$ 2.33 μm; R^2 = 0.990) as gum concentrations increased from 3 to 10%. The d_{50} of RE RP (12.16$\pm$1.01 μm) was similar (p > 0.05) to those of 3 and 5% RPs and lower (p ≤ 0.05) than that for 10% RPs. Particle sizes showed a unimodal distribution for RE, MD (Figure 2a), and GA RPs and a bimodal distribution for MD:GA RP (Figure 2b). Therefore, GA and MD RP particle sizes were more homogeneous and MD:GA RPs were more heterogeneous. This distribution could be attributed to agglomeration, which causes larger particles to form (Tonon et al., 2011). Figure 3 shows the TMA content for all encapsulated powders. An increase in d_{50} was observed with a decrease in anthocyanin content. This behavior was very similar to what was observed for TPCs.

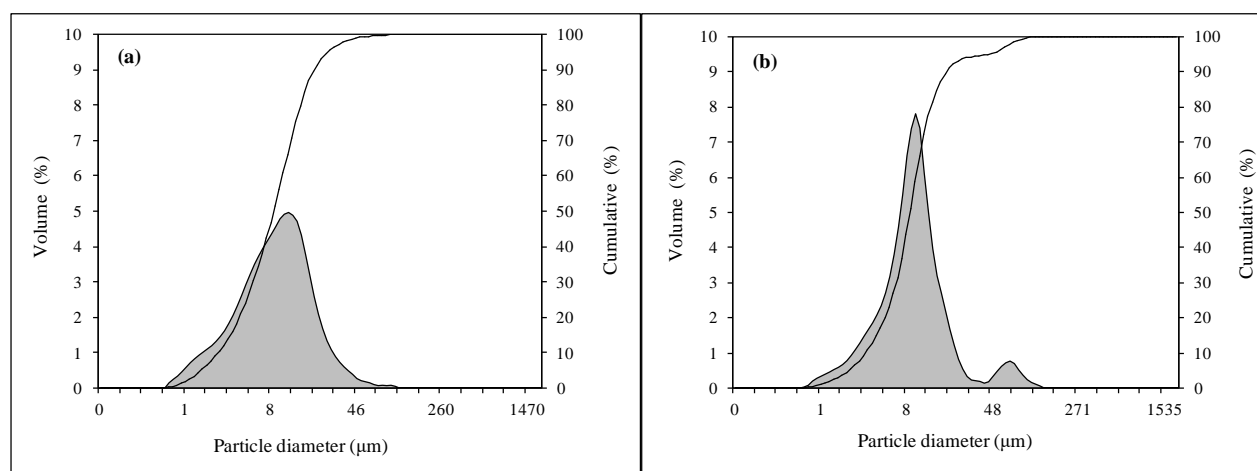Figure 2. Particle size distribution of Roselle calyces, powders with 3% maltodextrin (MD) (a) and 3% maltodextrin:gum arabic (MD:GA) (b)
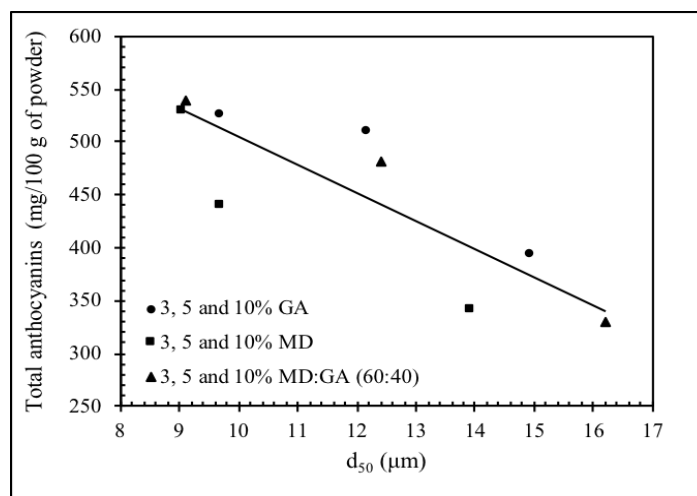
Figure 3. Correlation between total monomeric anthocyanin content and average diameter (d_{50}) of particles in microencapsulated Roselle calyces powders with gum arabic (GA) and maltodextrin (MD)

Bulk density. Significant differences ($p \le 0.05$) were observed between bulk densities of RPs with different gum types (Table 4). RE RPs showed a higher average bulk density (0.380 ± 0.020 g/mL) than RPs with GA (0.174 ± 0.035 g/mL), MD (0.171 ± 0.057 g/mL), or MD:GA (0.250 ± 0.118 g/mL). As the concentration of gums increased, bulk densities of RPs decreased ($p \le 0.05$): average densities of 0.271 ± 0.064, 0.204 ± 0.072, and 0.120 ± 0.027 g/mL were observed for 3, 5, and 10% gums, respectively. The highest bulk density was observed for RE (0.380 ± 0.020 g/mL) and for RPs with 3% MD:GA (0.353 ± 0.021 g/mL).

Tapped density. Table 4 shows the tapped densities of RPs. Tapped densities showed trends that were similar to those of bulk densities; however, because the RPs were tapped, all densities were higher.

3.4.4 Color of Powders

Lightness (*L**). Significant differences were observed between RPs based on type and concentration of gums (Table 5). RE RPs (41.15 ± 1.00) were darker than those with GA (55.76 ± 1.90), MD (57.14 ± 3.97), and MD:GA (52.42 ± 5.22). Lightness of RPs increased with increasing gum concentration, with lightness measures of 41.15 ± 1.00, 51.51 ± 3.18, 54.36 ± 3.03, and 59.45 ± 2.07 for 0, 3, 5, and 10% gums, respectively. Ochoa-Velasco et al. (2017) reported an average lightness value of 40.3 ± 0.71 for microencapsulated RPs obtained by spray drying using mesquite gum at different concentrations (1, 2, 3, 4, and 5% w/v); however, no significant differences were observed with the different microencapsulated powders. The authors concluded that gum concentration did not have a significant effect on all color properties. Idham et al. (2012) reported color parameters of RPs with the same gums used in this study, but they purified anthocyanins before they were mixed with the carrier for spray drying. They obtained *L**, *a**, and *b** values of 44.9, 30.3, and − 6.3 for RPs with GA; 39.3, 43.1, and −0.8 for RPs with MD; and 45.9, 34.8, and −4.3 for RPs with MD:GA. Gums were added to the extracts to reach a concentration of 20%. The mixtures were fed into the spray dryer at a flow rate of 9.5% with inlet and outlet temperatures of 150 and 110 ℃, respectively.

Table 5. Effect of gum type and concentration on the color properties of spray-dried powders and powders in solution[a]

Gum type	GC (% w/w)	L^*	a^*	b^*	H (°)	C
Powder						
RE	0	41.15±1.01h	42.68±0.26a	16.31±0.25a	20.92±0.40a	45.69±0.17a
GA	3	54.25±0.94de	40.66±0.33bc	11.17±0.41dcd	15.36±0.46bc	42.17±0.40cd
	5	55.59±2.33cde	39.77±0.95cd	10.31±0.64d	14.53±0.54c	41.08±1.08be
	10	57.43±0.66bc	38.72±0.86d	7.63±0.42e	11.15±0.63e	39.47±0.85fg
MD	3	52.83±0.27ef	41.11±0.09bc	12.49±0.11c	16.90±0.16b	42.97±0.08bc
	5	56.74±0.68bcd	39.16±0.20d	10.52±0.18d	15.04±0.17c	40.55±0.24ef
	10	61.86±1.00a	36.55±0.59e	8.11±0.17e	12.51±0.07de	37.43±0.60h
MD:GA	3	47.45±0.98g	41.83±0.23ab	14.76±1.06b	19.43±1.36a	44.37±0.26ab
	5	50.75±0.59f	40.97±0.22bc	12.50±0.34c	16.96±0.36b	42.83±0.30c
	10	59.05±0.82ab	37.03±0.36e	8.42±0.15e	12.81±0.10d	37.97±0.39gh
Powder in solution						
RE	0	68.32±0.24e	35.18±0.46a	14.29±0.08a	22.10±0.15a	37.97±0.46a
GA	3	71.86±0.75d	31.12±0.78b	11.65±0.26bc	20.53±0.31ab	33.24±0.80b
	5	74.71±2.29bc	29.97±1.27bc	11.13±1.00cd	20.34±0.98ab	31.97±1.52bc
	10	79.63±0.80a	23.57±0.67e	8.89±0.41e	20.66±1.06ab	25.19±0.64e
MD	3	72.11±0.71cd	30.19±0.52b	11.98±0.23bc	21.64±0.08a	32.48±0.57b
	5	76.19±0.88b	26.80±0.60d	10.10±0.17d	20.65±0.12ab	28.65±0.62d
	10	80.01±0.22a	22.22±0.62ef	7.95±0.13e	19.68±0.25b	23.60±0.63ef
MD:GA	3	72.59±0.60cd	31.38±0.41b	12.58±0.27b	21.85±0.17a	33.81±0.48b
	5	75.63±0.27b	28.14±0.41cd	11.21±0.20cd	21.73±0.47a	30.29±0.38cd
	10	80.44±0.83a	21.23±0.73f	8.49±0.25e	21.81±1.11a	22.87±0.63f

[a]Different letters in the same column indicate significant differences (p ≤ 0.05) between values. GC, gum concentration; RE, Roselle extract; GA, gum arabic; MD, maltodextrin.

Green-red color (a^*). Green-red color values (a^*) decreased significantly, as gum concertation increased, with averages of 39.72 ±1.07, 38.94 ±2.01, 39.94 ±2.23 for RPs with GA, MD, and MD:GA, respectively. RE RPs had the highest red color value (42.68 ±0.26). The average green-red color values by gum concentration were 42.68 ±0.26, 41.20 ±0.55, 39.97 ±0.94, and 37.43 ±1.13 for 0, 3, 5, and 10% gums.

Yellow-blue color (b^*). Yellow-blue color values (b^*) were significantly different among different concentrations and types of gum. The overall averages were 9.70 ±1.66, 10.38 ±1.91, and 11.89 ±2.84 for RPs with GA, MD, and MD:GA, respectively. RE RPs had the highest b^* value (16.31 ±0.25). An increase in gum concertation was associated with a decrease in b^* values, with values of 16.31 ±0.25, 12.81 ±1.67, 11.11 ±1.11, and 8.05 ±0.42 for 0, 3, 5, and 10% gums, respectively.

Hue (H). RE RPs had a higher H value (20.92 ±0.40°) than RPs with GA (13.68 ±1.99°), MD (14.82 ±1.91°), or MD:GA (16.40 ±2.98°). These differences among gum types were significant. H values were also associated with gum concentrations: as gum concentrations decreased, H values increased (20.92 ±0.40°, 17.23 ±1.92°, 15.51 ±1.16°, and 12.16 ±0.83° for RPs with gum concentrations of 0, 3, 5, and 10%, respectively). Hue values are located in the red-yellow segment (0–90°) of the color space; however, these values tend to lie in the deep red or purple color areas (McLaren, 1986).

Purity (C). The purity (chroma) of all RPs showed trends that were similar to those of hue. Significant differences were observed based on type of gum added, with purities of RPs with GA, MD, and MD:GA of 40.91 ±1.38, 40.32 ±2.43, and 41.72 ±2.90, respectively. RE RPs were purer (45.69 ±0.17) than RPs with GA, MD, or MD:GA. The purity of powders were found to decrease as gum concentration increased, with purity values of 45.69 ±0.17, 43.17 ±0.99, 41.49 ±1.18, and 38.29 ±1.07 with gum concentrations of 0, 3, 5, and 10%, respectively. RE RPs showed the highest purity (p ≤ 0.05). Purity values specify the position of colors between gray and a pure hue (saturation). Therefore, the purity or chroma of a color is proportional to the amount of color it has (McLaren, 1986). Figure 4 illustrates the correlation between a^* and purity values for all treatments; an increase in the a^* value was associated with an increase hue purity.
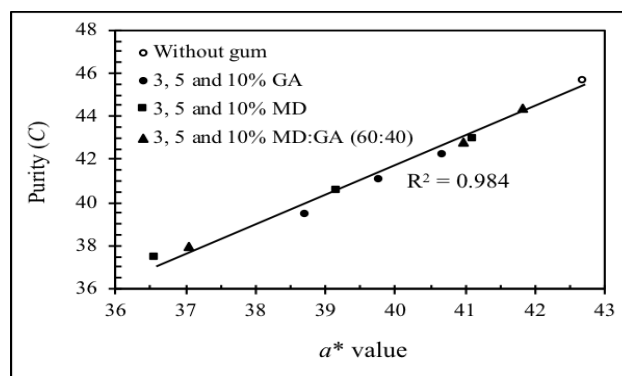
Figure 4. Correlation between purity and green-red color values for microencapsulated Roselle calyces powders with different gums

3.4.5 Color of Powders in Solution

Table 5 shows the color parameters for RPs in solution. Significant differences ($p \leq 0.05$) were observed between RPs with different types and concentrations of gums. L^*, a^*, and b^* color parameters and purity of RPs in solution were similar to those for dry RPs. The lightness (L^*) of powders in solution was higher (68–80) than that of powders because the solutions were dark red-purple but transparent. The RE RPs in solution were the darkest of all solutions. The red color (a^*) of solutions decreased as gum concentration increased. The b^* (yellow) color values for solutions were also similar to those for powders. The purity of solutions was less than those for the powders (23–38). Purities of solutions with RE (22.10 $\pm$ 0.15) and MD:GA (21.79 $\pm$ 0.61) RPs were significantly different ($p \leq 0.05$) from those of solutions with GA (20.51 $\pm$ 0.75) and MD (20.66 $\pm$ 0.86) RPs. Significant differences ($p \leq 0.05$) were also observed between RP solutions with 0, 3, 5, and 10% gum. Ochoa-Velasco et al. (2017) evaluated the color values for reconstituted RPs (100 mg/7.5 mL of distilled water) encapsulated with mesquite gum at different concentrations. The average L, a, and b color value ranges were 23.96 $\pm$ 0.66–30.54 $\pm$ 0.05, 32.83 $\pm$ 3.83–40.37 $\pm$ 0.39, and 14.56 $\pm$ 1.77–18.66 $\pm$ 0.08, respectively. These results differ from those reported in this work, possibly because of the difference in gum type, as well as solution concentration.

3.4.6 Antioxidant Characteristics

Table 6 shows TMAs, TPCs, and AC for the different RPs.

TMAs. The TMA contents in GA (476.39 $\pm$ 64.18 mg/100 g), MD (437.38 $\pm$ 79.55 mg/100 g), and MD:GA (450.10 $\pm$ 91.38 mg/100 g powder) RPs were significantly different. RE RPs had higher ($p \leq 0.05$) TMA content (665.39 $\pm$ 9.34 mg/100 g) than the other RPs. As gum concentration increased, TMA content decreased: 3% RPs showed higher TMA content than 5 and 10% RPs, with values of 531.62 $\pm$ 14.22, 477.10 $\pm$ 35.22, and 355.15 $\pm$ 30.05 mg cyanidin-3-glucoside equivalents/100 g of powder for 3, 5, and 10% gums, respectively.

TPCs. The TPC content in GA (2827.9 $\pm$ 364.8 mg/100 g), MD (499.4 $\pm$ 2956.1 mg/100 g), and MD:GA (3160.1 $\pm$ 549.4 mg/100 g of powder) RPs were significantly different. RE RPs had the highest ($p \leq 0.05$) TPC content (4929.4 $\pm$ 175.4 mg/100 g). As the gum concentration increased, TPC content decreased: RPs with 3% gum showed higher TPC content than RPs with 5 and 10% of gum, with values of 3472.9 $\pm$ 329.5, 167.3 $\pm$ 3046.2, and 2424.9 $\pm$ 138.0 mg/100 g of powder for 3, 5, and 10% gums, respectively.

AC. No significant differences ($p > 0.05$) were observed between the ACs of RE (1284.9 $\pm$ 20.8 mg/100 g), GA (1275.3 $\pm$ 185.2 mg/100 g), MD (1187.2 $\pm$ 146.4 mg/100 g), and MD:GA (1186.3 $\pm$ 102.9 mg TE/100 g of powder) RPs. Regarding gums concentration, RPs with 5% gum showed higher antioxidant capacity (1382.2 $\pm$ 106.0 mg ET/100 g powder) than RPs with 3% (1186.0 $\pm$ 94.5 mg/100 g) and 10% (1080.5 $\pm$ 45.5 mg/100 g powder) gum.

Table 6. Effect of gum type and concentration on the antioxidant properties of spray-dried powders[a]

Gum type	GC(% w/w)	TMAs[b]	TPCs[c]	AC[d]
		(mg/100 g of dry powder)		
RE	0	665.39 ±9.34a	4929.4 ±175.4a	1284.9 ±20.8bc
GA	3	525.37 ±19.34b	3090.2 ±130.0d	1226.9 ±115.5bcd
	5	510.33 ±32.46bc	3036.5 ±161.3d	1498.5 ±44.0a
	10	393.45 ±7.72e	2357.0 ±92.4e	1100.6 ±46.0d
MD	3	530.30 ±4.46b	3527.0 ±165.6bc	1120.7 ±95.1cd
	5	440.20 ±9.98b	2950.8 ±181.0d	1368.2 ±17.3ab
	10	341.65 ±5.29f	2390.4 ±111.6e	1072.6 ±56.2b
MD:GA	3	539.19 ±13.27b	3801.6 ±125.9b	1210.4 ±11.7bcd
	5	480.76 ±9.37cd	3151.3 ±110.1cd	1280.0 ±83.5bc
	10	330.35 ±16.29f	2527.5 ±155.5e	1068.5 ±31.7d

[a]Different letters in the same column indicate significant differences ($p \leq 0.05$) between values. [b]TMAs: total monomeric anthocyanins (cyanidin-3-glucoside equivalents). [c]TPCs: total phenolic compounds (gallic acid equivalents). [d]AC: Antioxidant activity (Trolox equivalents). GC, gum concentration; RE, Roselle extract; GA, gum arabic; MD, maltodextrin.

3. Conclusions

Based on the drying conditions used in this study, the microencapsulated RPs obtained with a mixture of MD and GA (60:40) as a carrier were the preferred powders because of its higher yields and better antioxidant and color characteristics. However, the red color ($a*$) average for all powders decreased as the gum concentration increased which is due to the gum concentration. In addition, the 3% MD:GA RP showed the highest amount of TMAs (cyaniding-3-glucoside equivalents/100 g) and TPCs; however, TMAs and TPCs were well maintained in all MD:GA RPs. These results indicate that microencapsulated powders can be used successfully to produce attractive functional foods as well as imparting flavor characteristics to foods. However, a stability study should be conducted with these RPs to evaluate their carrier efficiency. A study of MD:GA mixtures at different ratios than those used in this work should also be conducted to optimize yields and physicochemical properties of RPs obtained. Therefore, more studies about stability of color, solubility, moisture sorption characteristics, and maintenance of antioxidant properties are required.

Acknowledgments

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