

Lyophilized Powder of *Hibiscus sabdariffa* (Roselle) Extracts using Gum Arabic and Maltodextrin as Carrier Agents

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Abstract

Freeze-drying is a process for drying foods without heat application. The physical, chemical and sensory properties of the food remain without significant changes. In this work, maltodextrin (*MD*), gum arabic (*GA*), and a blend of *MD:GA* (60:40) were used as encapsulating agents of Roselle (*Hibiscus sabdariffa*) calyces extracts. Lyophilized powders were obtained at different concentrations of encapsulating agent (0, 3, 5, and 10%, w/w). Powders were analyzed in yield and physicochemical (average size diameter (d_{50}), moisture content, water activity (a_w), bulk and compacted densities, and color), and antioxidants (anthocyanins content, total phenolic compounds, antioxidant capacity) characteristics. The yields of freeze-drying powders from different encapsulating agents ranged 82 to 95%. The average diameter (d_{50}) was higher for powders without gum (139.5±25.6 μm) than for powders with encapsulating agents (35 to 89 μm). The moisture content and a_w of the powders were in the ranges 5.3-11.2% and 0.20-0.29, respectively. The value of the red (a^*) color parameter of all powders was 37.0±2.8, decreasing as increasing the gums concentration. Powders with 3% *GA* and *MD* showed the highest amount of anthocyanins: 560.93±10.13 and 543.46±15.68 mg/100 g of powder, respectively. The highest total phenolic compounds content was observed in the powder with the 3% *MD:GA* blend (4,705.70±140.54 mg/100 g of powder). Powder with 3% *MD* showed the highest antioxidant capacity (1,766.30±31.15 mg of Trolox equivalents/100 g powder).

Keywords: microencapsulation, freeze-drying, *Hibiscus sabdariffa* powders, maltodextrin, gum arabic, anthocyanins, phenolic compounds, antioxidant capacity

1. Introduction

The encapsulation of food compounds is used to reduce their degradation due to environmental factors (such as oxygen, light, temperature and pro-oxidants) to improve their stability during processing or to control their release in the food system (Santos and Meireles, 2010). Encapsulating agents include natural polymers or lipids. Maltodextrins and gum arabic are the most commonly used encapsulating agents in spray drying for obtaining microencapsulates. Maltodextrins provide low viscosities at high concentrations and good solubility; however, its emulsifying capacity is low. Maltodextrins in a range of 10 to 20 equivalent of dextrose are the most appropriate. On the other hand, gum arabic is a very efficient encapsulating agent; it is a polymer which has 2% protein in its structure providing excellent emulsifying properties; however, at high concentrations its viscosity increases (Gharsallaoui et al., 2007). It has been seen that mixtures of these two carrier agents may provide better results in spray drying (Zhang et al., 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 great 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).

Freeze drying (lyophilization) is a preservation method where water is evaporated or eliminated by the application of vacuum and low temperatures in the processing system. It consists of evaporating water from a frozen material (sublimation) without passing through the liquid state. Lyophilization is a way to dry heat labile chemicals or food products. This preservation procedure is used for the production of milk for infants, soups, coffee, infusions, the commercial preparation of antibiotics, some vaccines, many foods and flavor products. The main advantage of this method is that products being dehydrated in a frozen state retain their shape, aroma, flavor, vitamins, nutritional value, and active ingredients. In addition, it could be applicable to most foods, facilitating their productions on site with minimal transport costs. However, it is a prolonged and expensive method (Santos and Meireles, 2010; Özkan and Bilek, 2014). Nowadays, in addition to the products above mentioned, the process is being used to obtain powders of extracts from parts of plants in order to evaluate their antioxidant, antimicrobial and coloring properties (Rajarajan et al., 2010; Vilorio-Matos et al., 2002; Muro et al., 1997).

There is little research on this technique for obtaining powders from *Hibiscus sabdariffa* extracts. Some studies have shown that powders obtained by freeze drying, using maltodextrin (mainly) as encapsulating agent, have great stability maintaining their antioxidant properties; therefore, being these powders an option for use them as colorants and flavorings for foods (Duangmal et al., 2004; Selim et al., 2008).

The aim of this work was to evaluate the effect of maltodextrin and gum arabic as encapsulating agents for obtaining lyophilized powders of extracts of *Hibiscus sabdariffa* calyces.

2. Materials and Methods

2.1 Material

Calyces from creole *Hibiscus sabdariffa* long red variety, grown in Chiautla de Tapia, Puebla, Mexico, were used. The Roselle calyces powder (*RCP*) was obtained using a Veyco stainless steel mill model MPV 100 (Mexico) with a mesh of 0.5 mm.

2.2 Methods

2.2.1 Average Particle Size

The average particle size was carried out using a Microtac S3500 particle size analyzer (Microtac Inc., Largo, FL, USA) in a range of 0.25 to 2,800 μm . The analysis was carried out in triplicate using approximately 60 mg of Roselle calyces powder (*RCP*) or 40 mg of lyophilized powder (*LP*). Curves of granulometry, accumulative retained weight, and average diameter (d_{50}) were obtained (O'Hagan et al., 2005; Cid-Ortega and Guerrero-Beltrán, 2020).

2.2.2 Roselle Extracts Concentrates (*REC*)

The Roselle extracts (*RE*) were carried out at 50 ± 0.2 °C for 30 min according to Chumsri et al. (2008) and Cid-Ortega and Guerrero-Beltrán (2020) methods using a Riossa M80T Water Bath (Rios Rocha S.A., Monterrey, Nuevo León, Mexico). The Roselle:solvent ratio was 1:10 (20 g of Roselle powder + 200 mL of 50% ethanol). Mixtures were filtered through Whatman paper No. 4 and placed in 250 mL flasks wrapped with aluminum foil (Cid-Ortega and Guerrero-Beltrán, 2020). Afterward, the ethanol was removed, for obtaining Roselle extracts concentrates (*REC*), using a Büchi RE 111 rotary evaporator (Brinkmann Instruments Inc., Switzerland) at 45 ± 1 °C and 54 cmHg of vacuum for no more than 45 min (Selim et al., 2008). The *RECs* were analyzed in antioxidant characteristics (total anthocyanins, total phenols, and antioxidant capacity).

2.2.3 Roselle Extracts Concentrates-gums (*RECG*)

A 3x3 factorial design was used in this study. Three types of gums or blends (gum arabic powder from Roller Dry (Central de Drogas S.A. de C.V., State of Mexico, Mexico), maltodextrin ED: 9-14 (CP Ingredientes S.A. de C.V., Guadalajara, Mexico), and a blend of maltodextrin:gum arabic in a proportion of 60:40) and three concentrations of gums (3, 5, and 10% w/w) were used. The gum was added to each free-ethanol extract and stirred for 15 min at room temperature (22 ± 2 °C) to obtain the *RECG*. The *RECGs* were placed into 250 mL

flasks, covered with aluminum foil and stored in refrigeration (4 °C) until drying. A control was also prepared (extract without ethanol and gum). Total soluble solids, density and viscosity were analyzed.

2.2.4 Lyophilization

The *RECGs* were placed in Petri dishes (13.76 ± 0.07 cm in diameter), covered with aluminum foil and frozen for a minimum of 72 hours in a CHTC-16E horizontal freezer (Torrey, Mexico) at -26 ± 0.5 °C. The frozen *RECG* were lyophilized in a LabConco freeze drier (LabConco Corp. Kansas, City, USA) at 20 °C and 0.005 to 0.01 mmHg of vacuum for 72 hours. The lyophilized *RECG* (Roselle powders = *RP*) were weighed, pulverized, placed in amber pharmaceutical jars, sealed with plastic wrap and capped. *RP* were stored in a desiccator with silica at room temperature (22 ± 2 °C).

2.2.5 Physicochemical Properties of Extracts

Total soluble solids (TSS)

They were measured according to the 932.14C AOAC (1995) method. A manual Atago Master-M model refractometer (Atago Co. LTD., Tokyo, Japan) with a scale of 0-32 °Bx was used. The readings were corrected at 20 °C using the values established in the reference tables of the AOAC (1995).

Density. It was determined by the 945.06 AOAC (1995) method. 10 mL pycnometers were used. Empty pycnometers (W_1), pycnometers filled with distilled water (W_2), and pycnometers with the sample (W_3) were weighed. The density (25 °C) was calculated according to Eq. (1):

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

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

Absolut viscosity (μ)

A 350-159I Cannon Fenske capillary viscometer (Cannon Instrument Co., State College, PA, USA) was used. The kinematic viscosity was obtained by multiplying the flow time (seconds) of 6.6 mL of extract at 40 °C by the constant of the viscometer (0.4754 mm²/s²) at the same temperature. For calculating the absolute viscosity (μ), Eq. (2) was used (Cannon Instrument Company, 2000):

$$\mu(cP) = \rho_s * \nu_c \quad (2)$$

where ρ_s (g/mL) is the density of the extract and ν_c (mm²/s = cSt) is the kinematic viscosity.

2.2.6 Antioxidant Characteristics

Total monomeric anthocyanins (TMA)

The *TMA*s determination was carried out according to the Lee et al. (2005) method with some modifications according to Cid-Ortega and Guerrero-Beltrán (2020). Briefly, 0.5 mL of extract or 100 mg of powder were used, made up to 10 mL with distilled water and totally homogenized with a Vortex (2900 to 3000 rpm) for 5 min; these are the Roselle extract solution (*RES*) or the powder solution (*PS*). Then, 1 mL of solution was taken and mixed with 4 mL of buffer pH 1 or pH 4.5. The solutions were allowed to stand for 30 minutes at room temperature (22 ± 2 °C). The absorbances were measured at 520 and 700 nm in a Cary 100 UV-visible spectrophotometer (Varian Inc., Palo Alto, CA, USA). A blank with distilled water was used for standardizing the equipment. Results were reported as equivalents of cyanidin-3-glucoside (C-3-G) per 100 mL of extract or per 100 g of powder according to Eq. (3).

$$TMA = \frac{A * MW * DF}{\epsilon * L} * 100 \quad (3)$$

where *TMA* is the total monomeric anthocyanins content (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/mol); *DF* is the dilution factor; *L* is the cell pathway (1 cm); ϵ is the molar extinction coefficient of cyanidin-3-glucoside (26,900 L/mol*cm); 100 is the conversion factor of mg/mL or mg/g to obtain mg/100 mL or mg/100 g, respectively.

Total phenolic compounds (TPC)

They were determined by the method reported by Singleton and Rossi (1965) method with some modifications according to Cid-Ortega and Guerrero-Beltrán (2020). Briefly, 3 mL of distilled water, 150 μL of Roselle extract solution (*RES*) or 100 μL of powder solution (*PS*) were placed in test tubes (covered with aluminum foil), separately, mixed with a Vortex (2,900 at 3,000 rpm) for 5 min. Then, 250 μL of Folin-Ciocalteu reagent were added, completely mixed and left for 8 minutes in the dark. Finally, 750 μL of 20% Na_2CO_3 was added and made up to 5 mL with distilled water. Mixtures were left for 2 hours at room temperature ($21 \pm 1^\circ\text{C}$) in the dark. The absorbances were then measured at 765 nm in a Cary 100 UV-visible spectrophotometer (Varian Inc., Palo Alto, CA, USA). A standard curve was prepared in duplicate at different concentrations of gallic acid (98.5%, Sigma): 0 - 66.4 μg . $\text{Abs} = 17.50 \pm 2.36$ (1/mg gallic acid) \times (mg gallic acid) + 0.018 ± 0.011 ($R^2 = 0.998 \pm 0.001$). Total phenolic compounds (*TPC*) were reported as gallic acid equivalents (*GAE*) per 100 mL of extract or 100 g of powder according to Eq. (4).

$$TPC = \frac{A - b}{m} * DF * 100 \quad (4)$$

where *A* is the absorbance of the sample, *b* is the intercept, *m* is the slope and *DF* is the dilution factor.

Antioxidant capacity (*AC*)

The DPPH (1,1-diphenyl-1-picrylhydrazyl) method (Brand-Williams et al., 1995) was used with some modifications (Cid-Ortega and Guerrero-Beltrán, 2020). Briefly, 2 mL of *RES* or 1.6 mL *PS* were taken and diluted with ethanol (99.5%) to make up 10 mL in a volumetric flask, mixed with a Vortex (2,900 to 3,000 rpm) for 5 min and then filtered (twice) through Whatman paper No. 5. From the filtrates, 1 mL was taken and mixed, in a test tube (covered with aluminum foil), with 1 mL of ethanol (99.5%) and 2 mL of DPPH solution (7.8 ± 0.2 mg in 200 mL of 99.5% ethanol), perfectly mixed and allowed to stand for 45 min at room temperature ($21 \pm 2^\circ\text{C}$) in the dark. Absorbances were measured at 517 nm using a Cary 100 UV-visible spectrophotometer (Varian Inc., Palo Alto, CA, USA). The antioxidant capacity (*AC*) was calculated as percentage of inhibition according to Eq. (5).

$$I (\%) = \frac{Ac - As}{Ac} * 100 \quad (5)$$

where *Ac* is the absorbance of the control and *As* is the absorbance of the sample. A standard curve was prepared in duplicate with different concentrations of trolox (6-hydroxy-2, 5, 7, 8 tetramethylchrome-2, 97% carboxylic acid, Aldrich) (*T*): 0-28.7 μg . $I (\%) = 3278.9 \pm 195.8$ (1/mg *T*) \times (mg *T*) + 2.9 ± 1.3 . $R^2 = 0.989 \pm 0.007$. Results were expressed as trolox equivalents (*TE*) per 100 mL of extract or per 100 g of powder, according to Eq. (6).

$$AC = \frac{A - b}{m} * DF * 100 \quad (6)$$

where *A* is the absorbance of the sample, *b* is the intercept, *m* is the slope and *DF* is the dilution factor.

2.2.7 Physicochemical Properties of Powders

Yield (*Y*).

It was calculated according to the amount of total soluble solids (*TSS*) in the encapsulated extract and the amount of powder obtained (Fazaeli et al., 2012), according to Eq. (7).

$$Y (\%) = \frac{\text{Amount of powder}}{\text{Amount of TSS}} * 100 \quad (7)$$

Moisture content

It was determined according to the 934.06 AOAC (2000) method. A Cole Parmer vacuum oven (Chicago, Illinois, USA) was used. The sample (1.0 ± 0.003 g) was dried for 8 hours at $70 \pm 1^\circ\text{C}$ at a vacuum pressure of 200 to 220 mmHg.

Water activity (a_w)

It was measured using an AQUA-LAB hygrometer model 3TE (Decagon Devices Inc. Pullman, Washington, USA) with internal control of temperature. The equipment was calibrated with distilled water and charcoal (Decagon Devices Inc., 2008). The a_w of powder was measured at $25.1 \pm 0.06^\circ\text{C}$.

Bulk density (ρ_b). It was determined according to the Jumah et al. (2000) method. One gram of powder was placed in a 10 mL graduated cylinder. The sample was tapped ten times (on a polystyrene base) from a height of

15 cm. The bulk density was calculated according to Eq. (9).

$$\rho_b = \frac{W}{V_a} \quad (8)$$

where W (g) is the weight of the powder and V_a (mL) is the volume occupied by the powder.

Tap density (ρ_t)

It was carried out according to the method reported in the Official Mexican Norm NOM-104-STPS-2001 (NOM, 2001), with some modifications. One gram of powder was placed in a 10 mL graduated cylinder and topped with a rubber stopper. Then, the sample was tapped from bottom to top for 8 min (time in which volume was no longer changed). The tap density was calculated according to Eq. (9).

$$\rho_t = \frac{W}{V_t} \quad (9)$$

where W (g) is the weight of the powder and V_t (mL) is the compacted volume occupied by the powder.

Color

A Colorgard system 05 colorimeter (BYK-Gardner Inc., Silver Spring, Maryland, USA) was used. *Powders (P)*: the measurements were carried in the reflectance mode calibrating the equipment previously with black and white mosaics. A plate with a light path of 1.9 cm in diameter was used. The sample was placed in a weighing bottle (1.8 ± 0.3 g powder) with an external diameter of 2.65 cm. *Powder solution (PS) or Roselle extract solutions (RES)*: the color was assessed in the transmittance mode using a quartz cell (Konica Minolta Sensing Inc., 2 mm) of 3 mL (Salazar-González et al., 2009; Silva et al., 2013). The L^* (luminosity: black = 0; white = 100), a^* (green to red) and b^* (yellow to blue) color parameters were measured in the *CIELab* scale. From these data the purity (color saturation, $C = [a^2 + b^2]^{1/2}$) and hue ($H = \tan^{-1}[b/a]$) were calculated.

2.2.8 Statistical Analysis

It was carried out by ANOVA with a level of significance of 0.05 using the software MINITAB® version 14.1 (Minitab Inc., 2003). To establish differences between the treatments, a Tukey-Kramer multiple comparison test was used with a P value of 0.05.

3. Results and Discussion

3.1 Roselle Extracts Concentrates (REC)

Table 1 shows volumes, weights, and TSSs for RECs used for adding different amounts of carrier agents (GA, MD, MD:GA) at different concentrations (0, 3, 5, 10% w/w). The content of TMAs, TPCs, and ACs of all these RECs is shown in Table 2. The REs used for adding GA, MD and MD:GA (60:40) had 88.86 ± 2.82 , 95.19 ± 6.84 and 84.31 ± 20.93 mg of C-3-G/100 mL, respectively, of TMAs, 591.20 ± 20.20 , 709.65 ± 85.12 , and 788.28 ± 100.19 mg GAE/100 mL, respectively, of TPCs and 251.61 ± 11.58 , 239.37 ± 12.02 and 235.62 ± 8.85 mg TE/100 mL, respectively, of ACs. All these results are global averages. The TMAs, TPCs and ACs depend strongly in the TSS content (Table 1) due to concentration or elimination of ethanol; therefore, the more concentrated the extract, the more amount of TMAs, TPCs and ACs. Similar results were observed by Cid-Ortega and Guerrero-Beltrán (2020) for extracts of *H. sabdariffa* used for obtaining powders by spray drying.

Table 1. Amount of gums required for adding to REC¹ as well as volumes, weights, and total soluble solids of the REC

Type of gum	Concentration (% w/w)	Gum (g)	Extract (mL)	Extract (g)	TSS ² (°Bx)
GA	3	2.28 ± 0.02	72.50 ± 0.87	73.90 ± 0.78	16.69 ± 0.08
	5	3.69 ± 0.16	68.67 ± 3.22	70.13 ± 3.02	15.03 ± 0.21
	10	8.42 ± 0.37	74.00 ± 3.46	75.77 ± 3.32	15.24 ± 0.39
MD	3	2.11 ± 0.17	65.00 ± 6.08	67.80 ± 5.37	15.08 ± 0.48
	5	3.46 ± 0.12	63.33 ± 2.31	65.60 ± 2.23	14.87 ± 0.27
	10	7.72 ± 0.58	68.00 ± 5.29	69.43 ± 5.19	15.02 ± 0.23
MD:GA	3	2.23 ± 0.14	70.33 ± 4.51	72.00 ± 4.37	16.35 ± 0.25
	5	3.60 ± 0.09	66.67 ± 1.53	68.27 ± 1.54	16.16 ± 0.19
	10	6.60 ± 0.83	57.33 ± 7.51	59.40 ± 7.51	16.77 ± 0.84

¹REC: Roselle extracts concentrates. ²TSS: Total soluble solids in extracts without gum. RE: Roselle extract. GA: Gum arabic. MD: Maltodextrin.

Table 2. Antioxidant characteristics of Roselle extracts concentrates (*REC*)^a

Gum	Concentration (% w/w)	<i>TMA</i> ¹ (mg C-3-G/100 mL)	<i>TPC</i> ² (mg <i>GAE</i> /100 mL)	<i>AC</i> ³ (mg <i>TE</i> /100 mL)
<i>GA</i>	3	87.92 ± 1.07b	578.07 ± 9.59b	233.16 ± 2.59b
	5	89.98 ± 1.94bc	604.90 ± 19.96bg	229.58 ± 2.98cb
	10	88.67 ± 4.42b	590.62 ± 21.55b	255.37 ± 2.70d
<i>MD</i>	3	97.36 ± 5.11cde	788.83 ± 44.97cef	253.26 ± 1.24ad
	5	99.22 ± 5.18de	731.22 ± 34.97ac	264.39 ± 1.67e
	10	89.01 ± 5.92b	608.91 ± 33.62bd	237.18 ± 1.65bf
<i>MD:GA</i>	3	57.33 ± 3.61a	671.20 ± 46.01adg	244.57 ± 10.20f
	5	90.53 ± 1.99bc	860.22 ± 36.60ef	231.95 ± 2.64b
	10	105.06 ± 5.94e	833.43 ± 74.61f	230.35 ± 3.14b

^aDifferent letter within values in the same column indicate significant differences ($p \leq 0.05$). ¹*TMA*s: total monomeric anthocyanins (cyanidin-3-glucoside equivalents). ²*TPC*s: total phenolic compounds (gallic acid equivalents). ³*AC*: antioxidant capacity (Trolox equivalents). *RE*: Roselle extracts. *GA*: gum Arabic. *MD*: maltodextrin.

3.2 Roselle Extracts Concentrates-gums (*RECG*)

3.2.1 Physical Properties

Table 3 shows the content of total soluble solids, density and viscosity for all *RECG*.

Total soluble solids (TSS). The *TSS* content (as global averages) for the *RECG* added with *GA*, *MD*, and *MD:GA* (60:40) (19.40 ± 2.43, 20.01 ± 2.57, and 21.10 ± 2.79%, respectively) did not show any significant difference ($p > 0.05$). The *REC* had the lower amount of *TSS* content (16.14 ± 0.74%) since no gum was added. Regarding the concentration of gums, the *TSS* content increased as the concentration of gum increased in the *RECG*s: 16.14 ± 0.74, 17.73 ± 0.65, 19.29 ± 0.70 and 23.49 ± 1.00% for 0, 3, 5 and 10%, respectively, global averages. Comparable results were observed by Cid-Ortega and Guerrero-Beltrán (2020) for the *RECG* used for obtaining powders by spray drying.

Table 3. Physical characteristics of Roselle extracts concentrates-gums (*RECG*)^a

Gum	Concentration (% w/w)	<i>TSS</i> (°Bx)	Density (g/cm ³)	Viscosity ¹ (mPa.s)
<i>GA</i>	3	17.03 ± 0.14abd	1.05 ± 0.01ab	2.30 ± 0.04b
	5	18.69 ± 0.30beg	1.05 ± 0.01ab	3.06 ± 0.04c
	10	22.47 ± 0.33cf	1.06 ± 0.01abc	7.19 ± 0.11d
<i>MD</i>	3	17.67 ± 0.31dj	1.06 ± 0.01abc	1.88 ± 0.03a
	5	19.05 ± 0.36eg	1.07 ± 0.01bcd	1.97 ± 0.03a
	10	23.32 ± 0.26f	1.08 ± 0.01cd	2.60 ± 0.06b
<i>MD:GA</i>	3	18.48 ± 0.12gj	1.04 ± 0.01a	2.12 ± 0.03b
	5	20.14 ± 0.07h	1.05 ± 0.01ae	2.50 ± 0.04ib
	10	24.69 ± 0.38i	1.08 ± 0.01d	3.98 ± 0.04c

^aValues with different letters within the same column have significant differences ($p \leq 0.05$). ¹Viscosity at 25 °C.

Density. The densities of the *RECG* (Table 3) added with *GA*, *MD* and *MD:GA* (60:40) were 1.05 ± 0.01, 1.07 ± 0.01, and 1.06 ± 0.02 g/mL, respectively, as global averages; however, no significant differences were observed ($p > 0.05$). The *RECG* added with *MD* and *MD:GA* (60:40) showed higher densities. About the concentration of gums, *RECG* added with 3% of gums had lower density than the extracts with 5 or 10% of gum. Janiszewska and Witrowa-Rajchert (2009) carried out a study to explore the efficiency of the microencapsulation of rosemary aroma using maltodextrin (25 and 30%), gum arabic (30%) and maltodextrin:gum arabic in a 3:1 ratio (30%). The extracts with gum had densities of 1.04, 1.05, 1.03 and 1.05 g/mL for 25% of maltodextrin, 30% of maltodextrin, 30% of gum arabic and 30% of the mixture maltodextrin:gum arabic, respectively.

Viscosity. The viscosity of the *RECG* (Table 3) added with *GA* had the highest viscosities (4.18 ± 2.28 mPa.s) than the extracts with *MD* (2.15 ± 0.34 mPa.s) or a blend of *MD:GA* (2.87 ± 0.04 mPa.s, global averages). Regarding the concentration of gum, the *RECG* with 10% had the highest viscosity compared with 0, 3 and 5% of gums (4.58 ± 2.04, 1.72 ± 0.03, 2.10 ± 0.19 and 2.50 ± 0.47 mPa s, respectively, global averages).

Gharsallaoui et al., 2007 had reported that *GA* showed excellent emulsifying properties due to the fact that its structure contains a fraction of protein (approximately 2%), which gives this property.

3.3 Roselle Powders (RPs)

3.3.1 Granulometry of Roselle Calyces Powder (RCP)

Figure 1 shows the granulometric distribution of the Roselle calyces powder used for obtaining the Roselle extracts. The average diameter (d_{50}) was 231.50 ± 18.40 μm with a moisture content of $6.45 \pm 0.43\%$.

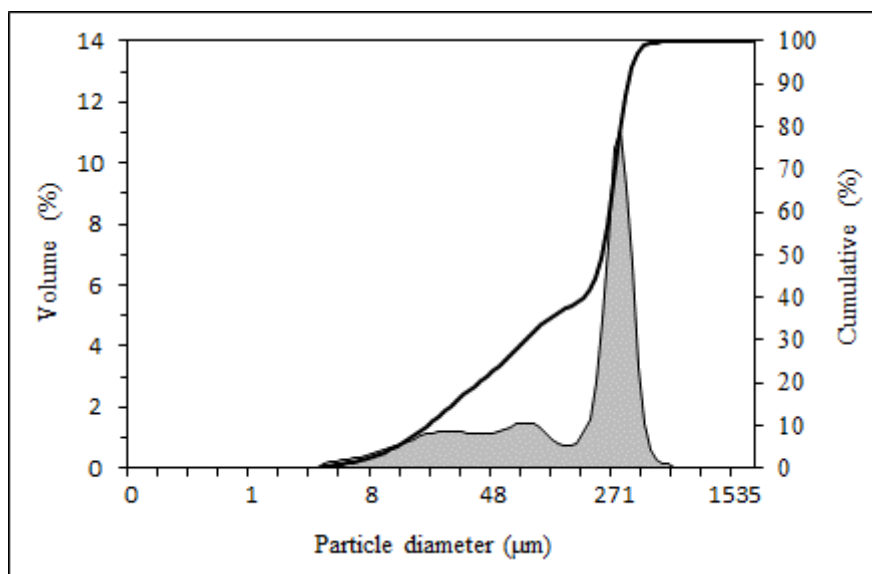


Figure 1. Particle size distribution of Roselle calyces powders

3.3.2 Yields of Powders

The yields global averages of powders of *RE* and those added with *GA*, *MD* and *MD:GA* (60:40) were of 85.89 ± 7.49 , 86.93 ± 2.58 , 90.85 ± 2.51 and $86.83 \pm 3.25\%$, respectively. The yields global averages for powders with 0, 3, 5 and 10% of gum were 85.89 ± 7.49 , 86.68 ± 3.73 , 87.97 ± 3.47 and $89.96 \pm 1.82\%$ for 0, 3, 5 and 10% of gum, respectively.

3.3.3 Physicochemical Characteristics

Table 4 shows the effect of gums on the physicochemical properties of Roselle powders.

Moisture content. The type of gum did not affect significantly ($p > 0.05$) the moisture content of powders: 8.62 ± 0.59 , 8.05 ± 2.61 , 8.01 ± 1.13 , and $8.39 \pm 1.82\%$ (global averages) of *RE*, *GA*, *MD*, and *MD:GA* (60:40), respectively. However, differences in the moisture content were observed as the gum concentration increased: 8.62 ± 0.59 , 10.09 ± 1.19 , 7.93 ± 0.59 , and $6.44 \pm 1.46\%$ for 0, 3, 5, and 10% of gum, respectively (global average). Similar behavior was reported by Farimin and Nordin (2009) when obtaining spray-dried powders from Roselle:pineapple juice (1:1) encapsulated with maltodextrin. They found that the moisture content of powders decreased as the maltodextrin concentration increased: 1.84 ± 0.30 , 1.53 ± 0.14 , $1.40 \pm 0.09\%$ for 3, 5, and 10% of gum, respectively. Similar results were reported by Fazaeli et al. (2012) when increasing the concentration of maltodextrin (DE 9) (8, 12 and 16%) in encapsulated blackberry juice by spray-drying. The moisture content of their powders ranged 1.5-2.0% using a flow rate of 800 L/h and an air entering temperature of 150°C . On the other hand, Goula and Adamopoulos (2008) reported an increase in moisture content in encapsulated powders of tomato pulp when increasing the concentration of maltodextrin in different proportions of tomato pulp:maltodextrin (4:1, 1:1 and 1:4). They reported moisture contents in the range 2.91-12.41%.

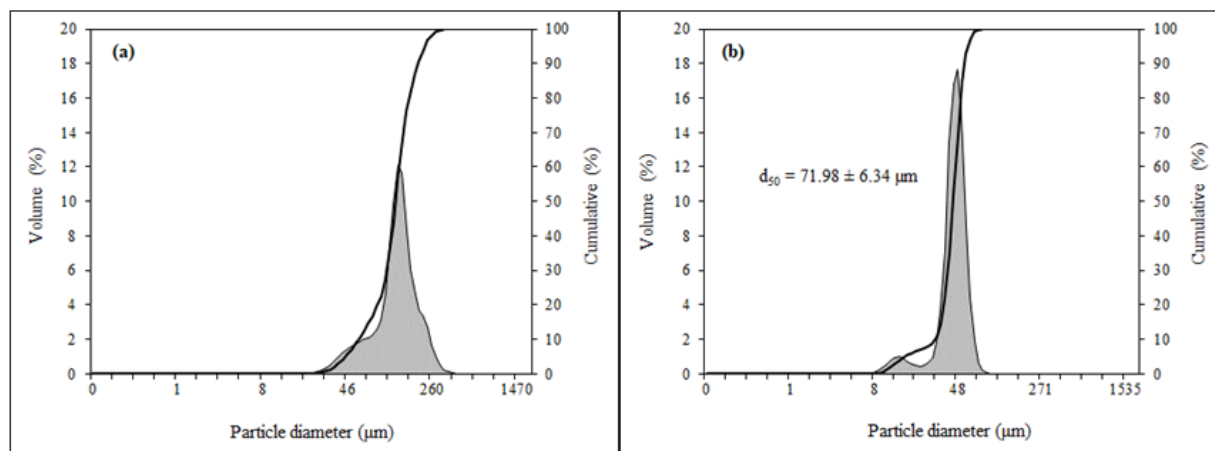
Table 4. Effect of gum types and concentrations on the physico-chemical properties of powders obtained by lyophilization^a

Gum type	Gum (% w/w)	Moisture (%)	a_w (at 25.1 ± 0.06 °C)	Average diameter d_{50} (µm)	Bulk density (g/cm ³)	Tap density (g/cm ³)
REC	0	8.62 ± 0.59ah	0.277 ± 0.023ac	139.50 ± 25.55a	0.670 ± 0.017a	0.800 ± 0.030ac
GA	3	11.20 ± 0.43b	0.237 ± 0.015abc	57.13 ± 5.46bc	0.530 ± 0.030b	0.673 ± 0.045bde
	5	7.70 ± 0.22ac	0.287 ± 0.031a	46.84 ± 15.72b	0.550 ± 0.017b	0.670 ± 0.050bde
	10	5.27 ± 0.57dj	0.250 ± 0.020ad	80.83 ± 8.28c	0.607 ± 0.040abc	0.797 ± 0.038c
MD	3	8.67 ± 0.57ae	0.197 ± 0.021bd	43.65 ± 1.57b	0.530 ± 0.044b	0.627 ± 0.064bd
	5	7.63 ± 0.72af	0.220 ± 0.000cd	44.63 ± 1.26b	0.560 ± 0.000bc	0.717 ± 0.006abc
	10	7.72 ± 1.80afg	0.277 ± 0.038ac	40.51 ± 2.09b	0.587 ± 0.046abc	0.707 ± 0.032abc
MD:GA	3	10.39 ± 0.43beh	0.197 ± 0.060bd	58.40 ± 14.24bc	0.640 ± 0.036ac	0.730 ± 0.010acde
	5	8.46 ± 0.46ah í	0.217 ± 0.015cd	71.54 ± 11.08bc	0.547 ± 0.012b	0.663 ± 0.021be
	10	6.32 ± 0.64cfij	0.247 ± 0.025ad	71.98 ± 6.34bc	0.590 ± 0.010ab	0.747 ± 0.025ace

^a Values with different letters in each column have significant differences ($p \leq 0.05$).

Water activity (a_w). The results of a_w are shown in Table 4. Regarding the type of gum, significant differences ($p < 0.05$) were observed between the REC (0.277 ± 0.023) and MD:GA (60:40) (0.220 ± 0.026) powders as well as between the GA (0.258 ± 0.030) and MD (0.231 ± 0.042) powders (global averages). About the concentration of gum, powders with 3% of gum had lower a_w (0.210 ± 0.024, global average). Powders with 0, 5 and 10% of gum had a_w s of 0.277 ± 0.0230, 0.241 ± 0.015 and 0.258 ± 0.029, respectively (global averages). The former information differs from that reported by Farimin and Nordin (2009); they reported a decrease in a_w when increasing the concentration of maltodextrin (a_w s of 0.27, 0.22 and 0.21 for 3, 5 and 10%, respectively) in spray dried powders of a mixture of Roselle extract:pineapple juice (1:1). Comunian et al. (2011) observed no significant differences in a_w s of powders of 5% chlorophyllide solution obtained with gum arabic (0.31 ± 0.10), maltodextrin (0.31 ± 0.14), and soy protein (0.28 ± 0.04) by spray drying.

Average diameter (d_{50}). The d_{50} values of Roselle powders are shown in Table 4. The gum type showed significant differences ($p \leq 0.05$) among d_{50} values of MD (42.93 ± 2.36 µm), GA (61.60 ± 17.72 µm) and MD:GA (60:40) (67.31 ± 11.67 µm) powders, all global averages. The powder of REC showed the highest d_{50} value (139.50 ± 25.55 µm). Figure 2 shows some examples of the particle size distribution of powders. All powders had a bimodal behavior. The powders showed heterogeneous particles; therefore, large particles were shaped through the process of agglomeration (Tonon et al., 2011). Comunian et al. (2011) reported a bimodal particle size distribution in powders, obtained from a 5% chlorophyllide solution, with d_{50} values between 11.2 to 19.04 µm. The powders were obtained as already mentioned above. On the other hand, Janiszewska and Witrowa-Rajchert (2009) reported d_{50} values of 55 and 29 µm for powders obtained with 25% of maltodextrin or 30% of gum arabic for encapsulated rosemary aromas obtained by spray drying.



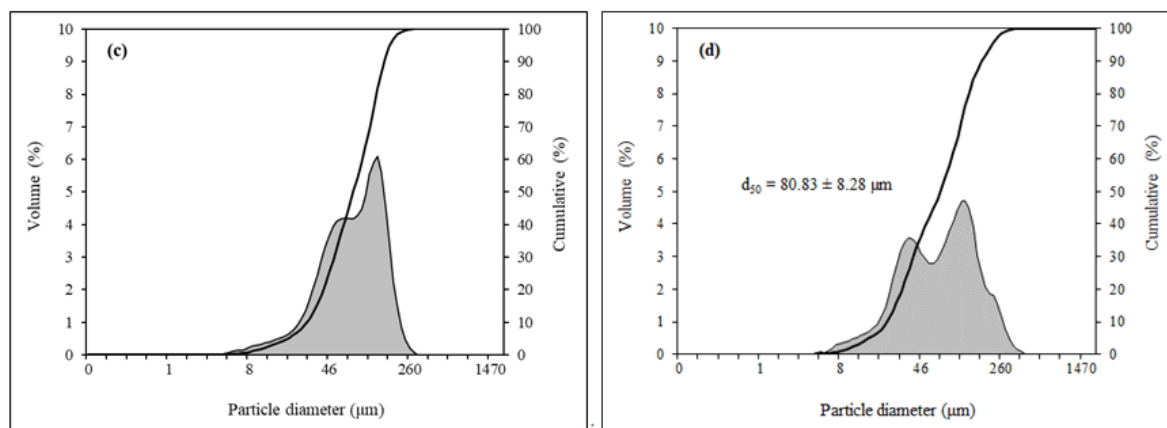


Figure 2. Particle size distribution of lyophilized powders obtained with *REC* (a); 3% *MD* (b); 10% *GA* (c), and 10% *MD:GA* (60:40) (d)

Bulk density. The bulk or apparent density of the Roselle powders is shown in Table 4. According to the type of gum, the powder of *REC* had the highest density (0.670 ± 0.017 g/mL) in comparison with the densities of *GA* (0.562 ± 0.044 g/mL), *MD* (0.559 ± 0.040 g/mL) and *MD:GA* (60:40) (0.592 ± 0.045 g/mL) powders (global averages for all powders). About the gum concentration, the *REC* powder had the highest density compared with the densities of *GA* (0.567 ± 0.064 g/mL), *MD* (0.552 ± 0.012 g/mL) and *MD:GA* (60:40) (0.594 ± 0.033 g/mL) powders. Different results were observed by Fazaeli et al. (2012); they reported decreasing bulk densities (from 0.55 to 0.35 g/mL) when increasing the concentration of maltodextrin (DE 9) (8, 12, and 16%) in powders of blackberry juice obtained by spray drying at different temperatures (110, 130, and 150 °C). Tonon et al. (2011) pointed out that the smaller the particle size, the greater the apparent density in powders of açai (*Euterpe oleracea*) juice obtained by spray drying using gum arabic and maltodextrin (10 and 20 DE). Results obtained in this work could be due to the drying conditions and types and concentrations of gums.

Tap density. Significant differences were observed ($p \leq 0.05$) between the tap density of *REC* (0.800 ± 0.030 g/mL) and *MD* (0.683 ± 0.056 g/mL, global average) powders. No significant differences ($p > 0.05$) were observed within densities of *GA* (0.713 ± 0.074 g/mL, global average) and *MD:GA* (60:40) (0.713 ± 0.042 g/mL, global average) powders. In the case of the gums concentration, the tap densities were higher for the *REC* (0.800 ± 0.030 g/mL) and 10% of gum (0.750 ± 0.048 g/mL, global average) powders in comparison with powders with 3 (0.677 ± 0.060 g/mL, global average) and 5% (0.683 ± 0.037 g/mL, global average) of gums.

3.3.4 Color of Powders

The color properties of the Roselle powders are shown in Table 5. All powders had a pale pink color.

Lightness (L^*): The *REC* powder had the lowest lightness (28.12 ± 0.48) (therefore, the darkest one) in comparison with the *GA* (36.39 ± 2.71 , global average), *MD* (36.40 ± 2.94 , global average) and *MD:GA* (60:40) (35.30 ± 4.66 , global average) powders. It was observed that, increasing the concentration of gum, powders with gum became clearer: 33.24 ± 2.58 , 35.30 ± 1.22 and 39.56 ± 2.65 for 3, 5 and 10% of gum, respectively (global averages). The lowest L^* value (29.90 ± 0.060) for powders with gum was observed in the *MD:GA* (60:40) powder at a concentration of 3% of gum. Ersus and Yurdagel (2007) reported an increase in lightness (L^*) of microencapsulated anthocyanin pigments from *Daucus carota* L. obtained by spray drying when decreasing the dextrose equivalents (10, 20-23, 28-31 DE) of maltodextrin. They also pointed out that the hue was higher for the powders obtained with maltodextrin of 28-31 DE. The authors concluded that the color of the powders became paler when increasing the DE of maltodextrin. Idham et al. (2012) reported L^* , a^* , and b^* color values for anthocyanins from Roselle extracts encapsulated by spray drying using the same gums as in this study. They reported values of 39.3, 43.1, and -0.8 for *MD*, 45.9, 34.8, and -4.3 for *MD:GA* (60:40), and 44.9, 30.3, and -6.3 for *GA* for L^* , a^* , and b^* color parameters, respectively. These values are different than those obtained in this study.

Green-red color (a^*): The *REC* powder had the lowest red coloration ($a^* = 30.11 \pm 1.23$) than powders with *GA* (38.02 ± 1.77 , global average), *MD* (37.88 ± 0.40 , global average) and *MD:GA* (60:40) (37.39 ± 1.94 , global average). The same behavior was observed for powders with 3, 5 and 10% gum: 37.18 ± 1.81 , 38.84 ± 0.66 and 37.28 ± 1.29 , respectively (global averages). The highest red coloration (39.23 ± 0.82) was observed in the

powder with 5% of GA.

Yellow-blue color (b)*: No significant differences were observed ($p > 0.05$) among the type of gum in all powders: *REC* (11.59 ± 0.90 , global average), *GA* (11.17 ± 1.81 , global average), *MD* (11.78 ± 1.06 and global average) and *MD:GA* (60:40) (12.71 ± 2.24 , global average) about the yellow color. There were no significant differences ($p > 0.05$) observed about yellowness within the powder of *REC* ($b^* = 11.59 \pm 0.90$) and the other powders: 3, 5 and 10% gum showed b^* values of 13.29 ± 1.36 , 12.50 ± 0.54 and 9.87 ± 1.19 , respectively (global averages); however, a decrease in b^* values were observed when increasing the concentration of gums.

Table 5. Effect of types and concentrations of gums on the color properties of Roselle powders obtained by lyophilization^a

Gum type	Gum (% w/w)	L^*	a^*	b^*	Hue (H°)	Purity (C)
Powder						
<i>REC</i>	0	28.12 ± 0.48 ad	30.11 ± 1.23 a	11.59 ± 0.90 ad	21.03 ± 1.06 a	32.27 ± 1.41 a
<i>GA</i>	3	35.37 ± 0.65 bc	38.97 ± 0.69 b	12.37 ± 0.56 a	17.61 ± 0.62 bd	40.89 ± 0.77 b
	5	33.98 ± 0.68 bf	39.23 ± 0.82 b	12.32 ± 0.07 a	17.44 ± 0.36 bd	41.12 ± 0.78 b
	10	39.83 ± 0.58 ce	35.87 ± 0.96 cd	8.80 ± 0.52 b	13.78 ± 0.48 cf	36.94 ± 1.05 c
<i>MD</i>	3	34.45 ± 0.45 bf	37.56 ± 0.35 bc	12.45 ± 0.38 a	18.34 ± 0.66 bd	39.57 ± 0.25 bd
	5	36.45 ± 0.75 bce	38.19 ± 0.25 b	12.15 ± 0.42 a	17.65 ± 0.63 bd	40.08 ± 0.21 bd
	10	38.29 ± 4.76 bce	37.90 ± 0.39 bc	10.74 ± 1.28 ab	15.82 ± 1.88 bcd	39.40 ± 0.36 bd
<i>MD:GA</i>	3	29.90 ± 0.60 df	35.01 ± 0.64 d	15.03 ± 0.28 c	23.24 ± 0.10 a	38.10 ± 0.69 dc
	5	35.47 ± 0.59 b	39.09 ± 0.29 b	13.03 ± 0.57 a	18.44 ± 0.85 d	41.21 ± 0.19 b
	10	40.54 ± 1.10 e	38.08 ± 1.05 b	10.06 ± 0.91 db	14.78 ± 0.93 f	39.40 ± 1.23 b
Powder in solution						
<i>REC</i>	0	71.26 ± 0.16 a	24.69 ± 0.09 a	12.56 ± 0.16 a	26.99 ± 0.15 a	27.71 ± 0.13 a
<i>GA</i>	3	73.37 ± 0.16 b	29.65 ± 0.40 b	12.84 ± 0.42 a	23.41 ± 0.45 b	32.31 ± 0.52 b
	5	75.83 ± 0.13 ce	26.65 ± 0.27 c	11.09 ± 0.13 b	22.59 ± 0.08 bd	28.86 ± 0.29 a
	10	79.89 ± 0.39 d	20.33 ± 0.24 de	8.80 ± 0.16 c	23.41 ± 0.50 b	22.16 ± 0.21 ce
<i>MD</i>	3	71.81 ± 0.74 a	29.90 ± 0.58 b	11.01 ± 0.30 b	20.22 ± 0.46 c	31.86 ± 0.60 b
	5	74.93 ± 0.77 c	26.75 ± 0.81 cf	9.81 ± 0.24 d	20.15 ± 0.18 c	28.50 ± 0.84 a
	10	80.70 ± 0.85 d	20.63 ± 1.20 deg	7.27 ± 0.33 e	19.42 ± 0.57 c	21.87 ± 1.23 c
<i>MD:GA</i>	3	76.69 ± 0.68 e	21.74 ± 0.31 eg	11.67 ± 0.20 b	28.23 ± 0.46 a	24.67 ± 0.30 d
	5	75.49 ± 0.36 ce	26.98 ± 0.21 f	11.33 ± 0.26 b	22.78 ± 0.12 bd	29.26 ± 0.20 a
	10	79.39 ± 0.57 d	21.95 ± 0.41 g	8.81 ± 0.36 c	21.87 ± 1.03 d	23.65 ± 0.34 de

^aThe values represent the mean \pm standard deviation ($n = 3$), values with different letters in each column show significant differences ($p \leq 0.05$).

Hue (H°): Regarding the hue values of powders with different types of gums (Table 5), the values of hue of powders with gum showed significant differences ($p \leq 0.05$) in comparison with the *REC* powder ($21.03 \pm 1.06^\circ$). No differences were observed within hue values of *MD* ($17.27 \pm 1.54^\circ$, global average) and *MD:GA* (60:40) ($18.82 \pm 3.73^\circ$, global average) powders. In relation to the concentration of gum, a decrease in hue values was observed when increasing the gum concentration: 21.03 ± 1.06 , 19.73 ± 2.69 , 17.84 ± 0.72 and $14.79 \pm 1.39^\circ$ (global averages) for 0, 3, 5, and 10% of gums, respectively; therefore, according to the *CIELab** color space (HunterLab, 1991), the reddish hue was decreased.

Purity (C): The *REC* powder had the lowest purity (32.27 ± 1.41) than that of powders with *GA*, *MD* and *MD:GA* (60:40): 39.65 ± 2.17 , 39.69 ± 0.39 and 39.57 ± 1.53 , respectively (global average). Regarding the concentration of gum, the *REC* powder showed the lowest purity ($p \leq 0.05$) than that of powders with 3, 5 and 10% of gum: 39.52 ± 1.32 , 40.80 ± 0.68 and 38.58 ± 1.48 , respectively (global average). The value of purity or Chroma is proportional to the amount of color or hue. This could be observed by correlating the color data of the a^* color parameter and purity of all powders (Figure 3). Salazar-González et al. (2009) obtained microencapsulated powders of Roselle extracts and mesquite gum at different concentrations (1, 2, 3, 4, and 5% w/v). They reported average values, on the Hunter scale, of 40.3 ± 0.71 , 31.93 ± 0.29 , 0.28 ± 0.00 , and 33.19 ± 0.3 for L , a , H , and C , respectively. The authors concluded that the gum concentration did not have a significant effect on the color parameters.

3.3.5 Color of Solutions of Powders

The color characteristics for the reconstituted Roselle powders are shown in Table 5. All solutions had a transparent red-purple color.

Lightness (L^*): It can be seen that, increasing the concentration of gum, the solutions were clearer: 71.26 ± 0.16 , 73.96 ± 2.22 , 75.42 ± 0.56 and 79.99 ± 0.79 for 0, 3, 5 and 10% of gum, respectively (global averages). The solutions of powers of *REC* and 3% *MD* were the darkest. Salazar-González et al. (2009) reported similar values to this research for the L^* and a^* color parameters of solutions from reconstituted powders (100 mg/7.5 mL distilled water) of Roselle extracts added with mesquite gum at different concentrations (1, 2, 3, 4 and 5% w/v). Then, the type of gum barely affected lightness; however, as the concentration of gum increases, the solutions became lighter; therefore, its purity decreases (Chroma) and consequently, its red coloration.

Green-red color (a^*): The red color of the solutions did not show significant differences ($p > 0.05$) about the type of gum; however, the solutions with 10% of gum had the lower red color (20.97 ± 0.98 , global average) than powder solutions with 0, 3 and 5% of gum (24.69 ± 0.86 , 27.10 ± 4.04 and 26.79 ± 0.46 , respectively, global averages).

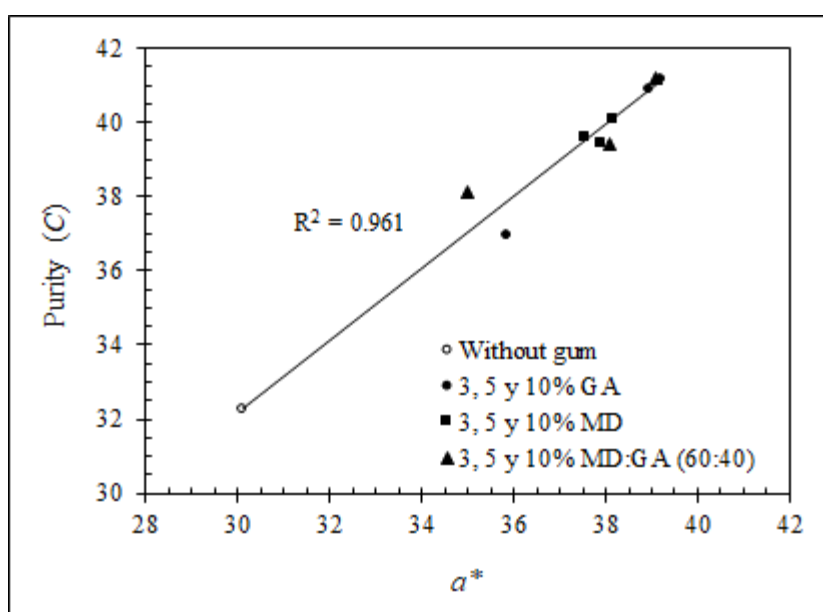


Figure 3. Correlation between the purity and the a^* color parameter of microencapsulated powders obtained by lyophilization with gum arabic (GA), maltodextrin (MD) and the blend of MD:AG

Yellow-blue color (b^*): The yellow color parameter of the solutions of powder also decreased as the gum concentration increased (12.58 ± 0.12 , 11.84 ± 0.85 , 10.74 ± 0.72 and 8.29 ± 0.81 for 0, 3, 5 and 10%, respectively, global averages).

Hue (H°). Regarding hue of solutions with different types of gums, the one with *MD* had the lowest hue (19.93 ± 0.54 , global average). About the concentration of gum, no differences ($p > 0.05$) were observed among all solutions being 23.95 ± 3.51 , 21.84 ± 1.28 and 21.57 ± 1.86 (global averages) for 3, 5 and 10% of gum, respectively. The solutions of powder of *REC* (26.99 ± 0.15 , global average) had greater hue (H) than solutions of 5 and 10% of gum.

Purity (C). The purity (C) of the solutions from different types of gum did not show any significant difference ($p > 0.05$). About the gum concentration, the lowest purity was observed for the solutions of powders with 10% of gum. No significant differences ($p > 0.05$) were observed within the solutions from 0, 3 and 5% of gum: 27.71 ± 0.13 , 29.61 ± 3.74 and 28.87 ± 0.56 , respectively (global averages).

3.3.6 Antioxidant Characteristics

Table 6 shows the *TMA*s, *TPC*s, and *AC*s for *RP*s obtained with different carrier agents and concentrations.

Total monomeric anthocyanins (TMA). The content of *TMA*s showed no significant differences ($p > 0.05$) within the types of gums: 408.51 ± 8.97 , 460.74 ± 90.96 , 447.58 ± 86.48 and 404.55 ± 55.03 mg C-3-G/100 g of dry

powder for *REC*, *GA*, *MD* and *MD:GA* (60:40), respectively (global averages). About the concentration of gums, the *REC* powder had lower amount of *TMA*s than powders with 3% of gum (489.13 ± 92.79 mg C-3-G/100 g of dry powder) (global average). No differences were observed in *TMA*s in powders with 5 and 10% of gum (470.73 ± 13.53 and 353.01 ± 16.31 mg C-3-G/100 g of dry powder, respectively, global average). *TMA*s of powders from 10% of gum had lower content of anthocyanins than powders with 3 and 5% of gum. It can be also observed that, when increasing the concentration of gums, the content of anthocyanins decreases which is very probably due to the amount of gum in the powder. Comparable results were observed by Cid-Ortega and Guerrero-Beltran (2020) for powders of *RECG* of *H. sabdariffa* obtained by spray drying.

Table 6. Effect of the type and concentration of gums on the antioxidant properties of lyophilized Roselle powders^a

Gum type	Gum (% w/w)	<i>TMA</i> ^b	<i>TPC</i> ^c (mg/100 g powder)	<i>AC</i> ^d
<i>REC</i>	0	408.51 ± 8.97a	4289.40 ± 207.07a	1722.40 ± 40.54a
<i>GA</i>	3	560.93 ± 10.13b	4179.60 ± 69.52ba	1612.60 ± 25.40bh
	5	474.81 ± 5.03cf	3618.80 ± 181.29c	1569.20 ± 40.60bcj
	10	346.48 ± 7.07de	2529.20 ± 115.66d	1486.20 ± 23.71d
<i>MD</i>	3	543.46 ± 15.68b	4004.00 ± 105.35b	1766.30 ± 31.15a
	5	458.02 ± 15.81c	3527.00 ± 133.82fc	1723.70 ± 19.83a
	10	341.27 ± 14.50d	2760.70 ± 136.18gd	1571.60 ± 70.50bj
<i>MD:GA</i>	3	363.00 ± 10.47eg	4705.70 ± 140.54h	1689.20 ± 9.85ahi
	5	479.36 ± 6.65f	4661.30 ± 154.72ih	1620.50 ± 54.48bi
	10	371.30 ± 5.09g	3935.60 ± 170.37jb	1531.40 ± 61.56dj

^aValues with different letters within the same column have significant differences ($p \leq 0.05$). ^b*TMA*s: total monomeric anthocyanins (cyanidin-3-glycoside equivalents). ^c*TPC*s: total phenolic compounds (equivalents of gallic acid). ^d*AC*: Antioxidant capacity (Trolox equivalents). *REC*: Roselle extract. *GA*: gum Arabic. *MD*: maltodextrin.

Total phenolic compounds (TPC). The content of *TPC*s (Table 6) regarding the type of gum was higher for the *REC* and *MD* powders ($4,289.40 \pm 207.07$ and $4,434.20 \pm 391.60$ mg *GAE*/100 g powder, respectively) (global averages) in comparison to the *TPC*s of powders with *GA* and *MD* (3442.50 ± 715.63 and 3430.60 ± 540.10 mg *GAE*/100 g, respectively) (global averages) gums. Regarding the gums concentration, a decrease in the content of *TPC*s was observed as the gum concentration increased ($4,296.40 \pm 323.44$, $3,935.70 \pm 549.70$ and $3,075.20 \pm 647.55$ mg *GAE*/100 g for 3, 5 and 10% of gum, respectively) (global averages). The content of *TPC*s for the *REC* powder was similar than for the concentrations of 3 and 5% of gum, and higher than that of the 10% of concentration of gum.

Antioxidant activity (AC). The *AC*s (Table 6) concerning the type of gum was higher for the *REC* and *MD* powders ($1,722.40 \pm 40.54$ and $1,687.20 \pm 96.21$ mg *TE*/100 g, respectively) (global averages) compared to the powders with *GA* and *MD:GA* (60:40) ($1,556.00 \pm 61.28$ and $1,613.70 \pm 80.24$ mg *TE*/100 g, respectively) (global averages). For the gums concentration, a decrease in the *AC*s was observed as the gum concentration increases ($1,689.40 \pm 68.33$, $1,637.80 \pm 76.43$ and $1,529.70 \pm 63.49$ mg *TE*/100 g of powder for 3, 5 and 10% of gum, respectively) (global averages). The *AC*s of the *REC* powders was similar to that of the concentration of 3% and higher than that of the concentrations of 5 and 10% of gum.

4. Conclusions

The microencapsulation of extracts from Roselle calyces by lyophilization provided high yields, as well as powders with good antioxidant and color properties. The concentration of gum is an important aspect to consider in the encapsulation of extracts from Roselle. According to the results obtained, the extracts of Roselle microencapsulated with maltodextrin and gum arabic at a concentration of 3%, allowed to obtain powders with the best antioxidant and color properties. Therefore, the use of microencapsulated powders obtained with these conditions signifies a viable option in the development of functional foods. However, it is recommended to carry out a stability study of the powders to determine the efficiency of the encapsulation process as well as the fading characteristics due to physical phenomena such as heat, oxygen, and light among others.

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