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Microencapsulation of grape seed oil by spray drying

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Abstract

In the search for alternative wall materials to replace gum Arabic (GA), a good but expensive encapsulating agent, this work aimed to evaluate the effectiveness of maltodextrin 10DE in combination with GA (GA/MD, 50:50 ratio) for the microencapsulation of grape seed oil by spray drying. The addition of maltodextrin to gum Arabic did not influence the mean particle diameter, powder bulk density, encapsulation efficiency or the total oil retained in the microspheres. Although the oil encapsulated with GA showed greater retention of phenolic compounds after spray drying, the sample encapsulated with GA/MD had greater ferric reduction antioxidant power and DPPH radical scavenging activity, and a lower peroxide index.

Keywords: microencapsulation; grape seed oil; antioxidant activity; maltodextrin; encapsulation efficiency; lipid oxidation.

Practical Application: Gum Arabic (GA), one of the most used wall materials for microencapsulation of oils by spray drying, has been already evaluated in several works; however, more studies about the influence of maltodextrin in combination with GA are needed. In the current work, similar encapsulation efficiency and better preservation with respect to antioxidant activity and lipid oxidation of grape seed oil are observed for maltodextrin combined with GA. This combination could be an alternative to the increasing prices of gum Arabic and its oscillation in supply.

1 Introduction

Nowadays, consumers are being encouraged to increase their intake of functional foods, leading to an increase in the development of such products by the food industry. In this context, animal fats are being replaced by vegetable oils in the diet due to their healthier aspects. Due to the high concentration of antioxidant substances such as polyphenols in grape seed oil, it could be used as an antioxidant in the conservation of food or to protect cells from oxidative damage by free radicals (Zhao et al., 2017).

Moreover, grape seed oil is rich in linoleic acid, the polyunsaturated fatty acid, and oleic acid, the monounsaturated fatty acid (Zhao et al., 2017). Since the monounsaturated (MUFA) and polyunsaturated (PUFA) fatty acids have been associated with the prevention of various disorders in humans (Al-Jawadi et al., 2018; Cicero & Colletti, 2017; Wijendran & Hayes, 2004), grape seed oil could be incorporated in food systems to increase the nutritional value and improve the beneficial effects on human health. However, due to its high content of PUFA, grape seed oil is chemically unstable and susceptible to oxidative degradation when exposed to light, oxygen and high temperatures, resulting in a loss of nutritional value and the production of undesirable off-flavors (Frankel, 2012). Therefore, it is necessary to protect grape seed oil during its handling, storage and transportation.

Microencapsulation is a technology that can be applied to retard lipid oxidation and decrease volatility, improving stability of oils and flavors. This technique consists of forming a continuous thin coating around solid particles, liquid droplets or gases, such that they are fully contained within the capsule wall. The principle of microencapsulation is to create a physical barrier between the active compounds and adverse environmental conditions and the food matrix (Martins et al., 2017). Spray drying, an economical and flexible process, is the method commonly used for microencapsulation, which converts liquids into powders with easier handling, storage and transportation and makes its uniform mixing in food formulations easier.

One of the most commonly used wall materials for the microencapsulation of hydrophobic compounds by spray drying is gum Arabic due to its good emulsifying properties, high solubility and low viscosity (Comunian & Fávaro-Trindade, 2016). Despite its desirable properties as an encapsulating agent, gum Arabic is an expensive ingredient and its production is susceptible to climatic and political turbulence, resulting in some supply problems (Kalušević et al., 2017). Thus the total or partial substitution of gum Arabic by other wall materials has been considered by several researchers. Maltodextrins have been widely used for the microencapsulation of food due to their low cost, high solubility and low viscosity at high concentration. However, the greatest disadvantage of this encapsulating agent is its lack of emulsifying property due to its limited affinity for hydrophobic components (BeMiller & Huber, 2007). Thus, the use of gum Arabic combined with maltodextrin could offer a good compromise between cost and effectiveness.

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With a view to finding alternative wall materials that could replace gum Arabic (GA), a good but expensive encapsulating agent, this work aimed to evaluate the effectiveness of maltodextrin in combination with GA (GA/MD, 50:50 ratio) for the microencapsulation of grape seed oil by spray drying.

2 Materials and methods

2.1 Material and sample preparation

Grape seeds from the cultivars Isabel and Bordô (Agroindustrial Cooperative Winegrowers, Marialva, PR, Brazil) were dried in an oven-dryer with air circulation (TE-394/2, Tecnal, Piracicaba, Brazil) at 60 °C for 6 h and stored in airtight bottles at -22 °C. Gum Arabic (GA) Instantgum BB (Nexira, São Paulo, Brazil) and maltodextrin 10DE (MD) Mor-Rex 1910 (Ingredion Brazil, Mogi Guaçu, Brazil) were used as the wall materials. DPPH 2,2-diphenyl-1-picrylhydrazyl and TPTZ 2,4,6-tris(2-pyridyl)-S-triazine were used as reagent, and fatty acid methyl esters and Trolox 6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid as standards (Sigma-Aldrich, Saint Louis, USA). All the reagents were of analytical grade.

2.2 Ultrasound–assisted extraction of grape seed oil

Ultrasound-assisted extraction of grape seed oil was carried out in a sonicator (Q700, QSonica, Newtown, USA) with a maximum power output of 700W. Grape seeds were ground using a seed mill (A11, Ika, Staufen, Germany) for 20 s, sieved to standardize the particle size to 0.841 mm and dispersed in hexane in a jacketed beaker as described by Porto et al. (2013) at 15 °C, controlled by a thermostatic bath with external circulation (TE-2005 model, Tecnal Piracicaba, Brazil). A titanium probe (½" in diameter) was used at a frequency of 20 kHz for 30 min and wave amplitude of 42 μm. The extraction conditions were determined according to previous studies. After extraction, the suspension was vacuum filtered through filter paper, and the solvent removed using a rotary evaporator under vacuum (MA120, Marconi, Piracicaba, Brazil) at 50 °C for 15 min (Porto et al., 2013).

2.3 Emulsion preparation and characterization

The wall materials used were gum Arabic (GA) and maltodextrin combined with gum Arabic (GA/MD, 50:50). The total solids concentration (wall material + oil) in the emulsion and oil concentration in relation to the total solids were 30% (Paramita et al., 2010) and 10% (Garcia et al., 2012; Frascareli et al., 2012) respectively. The wall material was dissolved in distilled water at 40°C and cooled to room temperature. About 250 ml of emulsion was formed by mixing the grape seed oil with the wall material solution using a rotor-stator homogenizer (MA102, Marconi, Piracicaba, Brazil) operating at 16,000 rpm for 5 min.

Emulsion stability

Ten milliliters of emulsion were placed in a 25 ml graduated cylinder and stored at room temperature for 4 and 24 h. The emulsion stability was evaluated by observing the occurrence of phase separation (McClements, 2007).

Emulsion droplet size

After emulsion formation, aliquots of samples were placed on slides, covered with coverslips and observed by optical microscope (leica DMLS, Wetzlar, Germany). Image capture was performed by digital camera (Motican 2500 UBS 2.0) using a 40× objective. The diameters of five hundred droplets were measured using the image processing software Motic Images Plus 2.0. The emulsion droplet size was expressed as Sauter mean diameter D_{32}, calculated by Equation (1):

$$D_{32} = \frac{\sum_{i=1}^{n} z_i d_i^3}{\sum_{i=1}^{n} z_i d_i^2} \quad (1)$$

Where: z_i is the number of droplets with diameter d_i.

2.4 Microencapsulation of grape seed oil by spray drying

Microencapsulation was carried out in a laboratory scale spray dryer (SD-05, LabPlant, Chelmsford, England) with a two-fluid atomizer spray nozzle with an orifice of 0.7 mm in diameter. For all experiments, about 250 ml of emulsion were fed into the drying chamber using a peristaltic pump, obtaining about 25 g of powder. For both emulsions, spray drying was carried out three times for subsequent analysis. The process conditions were: inlet and outlet air temperatures of 180 °C ± 3 °C and 105 °C ± 8 °C, respectively; feed and drying air flow rates of 350 mL/h and 73 m^3/h ± 3 m^3/h, respectively; and compressor air pressure of 1.8 bar. Air flow rate and compressor air pressure were chosen according to spray dryer manufacturer's recommendation. Other process conditions were based on preliminary test.

The microspheres were evaluated with respect to surface oil content, encapsulation efficiency, oil retention, moisture content, water activity, bulk density, particle size and morphology. In order to verify the effect of spray drying on the total phenolic compounds content, antioxidant activity, peroxide value and fatty acid profiles, these assays were determined in microspheres and grape seed oil before spray drying (control sample).

2.5 Powder evaluation

Extraction of grape seed oil from the microspheres

Grape seed oil was extracted from the microspheres (Bae & Lee, 2008) to be analyzed for the total oil and phenolic compound contents by Folin-Ciocalteau method, antioxidant activity by DPPH and FRAP methods and fatty acid profiles by gas chromatography.

Encapsulation efficiency and oil retention by the microspheres

Total oil content in the microspheres (*TO*, %) was determined as the ratio of the oil mass to powder mass. Surface oil content (*SO*, %) was obtained according to Bae & Lee (2008). The assays were carried out in duplicate.

Encapsulation efficiency (*EE*, %) and oil retention (*RO*, %) were calculated according to Equations 2 and 3, respectively.

$$EE = \left(\frac{TO - SO}{TO}\right) \times 100 \quad (2)$$

$$RO = \left(\frac{TO}{TO_{initial}} \right) \times 100 \quad (3)$$

Where: $TO_{initial}$ is the initial oil concentration before spray drying in relation to the total solids, corresponding to 10%.

Total phenolic compound content and antioxidant activity

Phenolic compounds in the grape seed oil (control sample) and oil encapsulated with GA and GA/MD were extracted with methanol:water solution (90:10 v/v) (Bail et al., 2008) and quantified according to the Folin-Ciocalteau method (Singleton & Rossi, 1965) in triplicate. The extract was mixed with water, Folin-Ciocalteau reagent and 10% sodium carbonate solution. After 60 min, the absorbance was measured in an UV-visible spectrophotometer (Libra S22, Biochrom, Cambridge, UK) at 765 nm. The results were correlated with a gallic acid standard curve (Dynamic, Diadema, Brazil) with concentrations varying from 6 to 30 mg of gallic acid, and expressed as mg gallic acid/g oil.

Antioxidant activities were evaluated in duplicate by DPPH (Tuberoso et al., 2007) and FRAP methods (Benzie & Strain, 1996; Tuberoso et al., 2007). Analytical curves prepared with different concentrations of Trolox (0.1-3.0 mM for DPPH method and 0.05-0.6 mM of Trolox for FRAP method) were used to calculate the results in mM Trolox/g oil.

Fatty acid composition

Fatty acids were hydrolyzed and transesterified according to ISO (International Organization for Standardization, 1978). Fatty acid methyl esters were evaluated using a gas chromatograph (17A model, Shimadzu, Kyoto, Japan) equipped with a flame ionization detector and capillary column (100 m × 0.25 mm) with 0.25 mM cyanopropylpolysiloxanes SII 88. The temperature ramp of the column was: 65 °C for 15 min; increased at 10 °C.min^{-1} to 165 °C, maintained for 2 min; increased at 4 °C.min^{-1} to 185 °C, maintained for 8 min; then increased at 4 °C.min^{-1} to the final temperature of 235 °C and held for 5 min. The detector and injector temperatures were both 260 °C using Split 1/100. The carrier gas (N_2), auxiliary gas (H_2), flame gas (H_2) and synthetic air flow rates were 1.2, 30, 30 and 300 ml min^{-1}, respectively.

Peroxide index

After grape seed oil extraction from the microspheres (Partanen et al., 2008), peroxide index was determined in duplicate (IDF, 2005) using a Fe^{+3} standard curve (1 to 20 µg) and expressed as milliequivalents of peroxide/kg oil (Shantha & Decker, 1994).

Physicochemical properties of the powders

Moisture content was determined gravimetrically in duplicate in an oven at 105 °C for 6 h (Association of Official Analytical Chemists, 1997). A thermohygrometer Aqualab (4 TEV, Decagon, Pullman, USA) was used to measure water activity at 25 °C.

Bulk density was calculated, in duplicate, by dividing the mass of powder by the volume occupied in the cylinder after tapped by hand 50 times. Particle size distribution, mean diameter particle D_{43}, surface area and span values were determined, in triplicate, using a laser light scattering analyzer (Mastersizer Laser Scattering Spectrometer, model 2000, Malvern, UK). The particles were dispersed using 99.5% ethanol.

Microsphere microstructures were analyzed by a scanning electron microscope (FEI Quanta 200, FEI Company, Netherlands) and the images acquired using XTM 2001 Fei company software. The samples were fixed to SEM stubs using double-faced carbon tape, and coated with gold (purity of 99.6%) in a sputter coater (SCD 050, Bal-Tec, Liechtenstein) at a coverage rate of 0.51 Å/s for 98 s, 40 mA and 5 × 10^{-1} mbar.

2.6 Statistical analysis

The results were analyzed by the analysis of variance (ANOVA) and Tukey test at 5% significance, using Statistica software (Statsoft, Tulsa, USA).

3 Results and discussions

3.1 Evaluation of the emulsions

Since emulsions are thermodynamically unstable systems, the formation of an oily layer on the emulsion can result in poor microencapsulation efficiency (Garcia et al., 2012). All the emulsions remained stable for 4 h, presenting no phase separation. However, the emulsion prepared with GA presented some oil droplets on the surface after 24 h, and the emulsion with GA/MD showed phase separation, which could be attributed to a deficiency in the emulsifying properties of maltodextrin.

The droplet size distributions of the emulsions (Figure 1a) ranged from 0 to 10-11µm. The mean droplet diameters

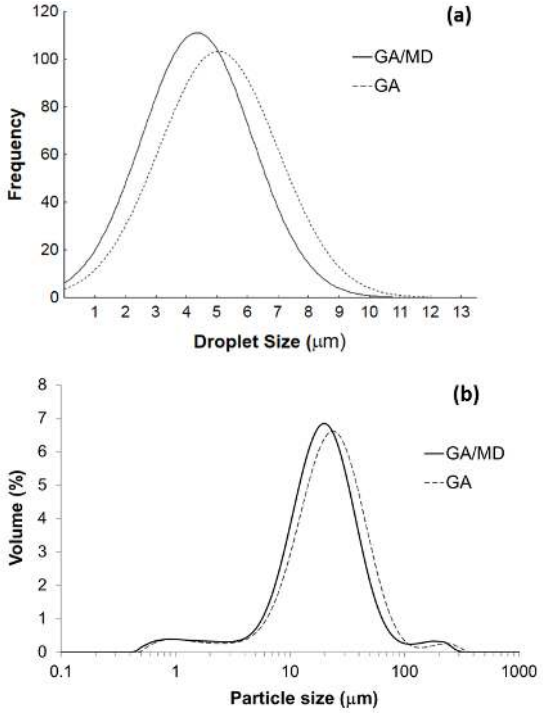

Figure 1. Size distributions: (a) of droplets in the emulsions and (b) of microspheres in the powders.

of the samples formulated with GA and GA/MD were 6.47 ± 0.06 and 5.80 ± 0.11 μm, respectively.

Smaller droplet sizes are desirable, since larger sizes result in poor encapsulation efficiency (Linke et al., 2017; Soottitantawat et al., 2003). The grape seed oil emulsions presented a negative aspect in relation to this property, requiring further studies to decrease the droplet size, such as the use of other types of homogenizer (high pressure valve or microfluidizer). The mean droplet size of the emulsion with GA/MD was lower than that formulated with GA. This result was unexpected, since the addition of maltodextrin to the wall material decreases the gum Arabic concentration, which implies less surface active material to fully coat the entire oil droplet, and hence larger droplet sizes would be expected. However, this finding could be related to emulsion viscosity. Dokic et al. (1998) reported that maltodextrin solution, a Newtonian fluid, presents lower viscosity (about 25 mPa.s for concentration of 30% at 20 °C). On the other hand, gum Arabic solution 30% has an apparent viscosity ranging from 125 to 110 mPa.s at 20 °C and shear rate up to 300 s^{-1} (Gómez-Díaz et al., 2008). Thus, the presence of maltodextrin in the wall material could have reduced the emulsion viscosity, resulting in smaller droplets, because there was more energy available for emulsification.

3.2 Surface oil content, oil retention and encapsulation efficiency

Both samples presented high amount of surface oil (about 30% of total oil) and low encapsulation efficiency (Table 1), which might be attributed to the larger emulsion droplets (6.5 μm and 5.8 μm). Sharif et al. (2017) obtained higher encapsulation efficiencies (87.0% to 92.1%) due to lower droplet mean diameters (119.8 to 217.6 nm μm) when compared with the current work. According to Jafari et al. (2008), small oil droplets will be entrapped more efficiently within the wall material matrix, and the emulsion will also be more stable during the atomization and drying processes. The larger the oil droplets are, the greater the breakup during atomization of the emulsion in the spray dryer chamber changing the size distribution (Munoz-Ibanez et al., 2015). This breakup of the emulsion favors the increase in of the surface oil, decreasing the encapsulation efficiency (Soottitantawat et al., 2003).

Although no significant difference was observed between the encapsulation efficiencies of the microspheres, the powder with GA/MD had higher surface oil content than that with GA (Table 1), despite its emulsion presented smaller oil droplets. These results showed that the amount of surface oil increased when maltodextrin was added to wall material composition, probably due to its low emulsifying capacity. Such result is consistent with the emulsion stability, where the emulsion GA/MD was less stable than the emulsion with GA.

All the samples presented high oil retention (Table 1) after the spray drying process. According to Rosenberg et al. (1985), this property is affected by several factors; however, the type of encapsulating agent did not significantly influence the oil retention of the samples.

Table 1. Characterization of powder particles prepared with gum Arabic (GA) and a combination of gum Arabic plus maltodextrin (GA/MD).

Parameters	GA	GA/MD
Total oil content (%)	9.19 ± 0.14 a	9.13 ± 0.09 a
Surface oil content (%)	2.95 ± 0.1 b	3.30 ± 0.04 a
Oil retention (%)	92.00 ± 1.41 a	91.50 ± 0.71 a
Encapsulation efficiency (%)	67.92 ± 1.55 a	63.47 ± 0.49 a
Moisture content (%, wet basis)	5.51 ± 0.07 a	4.15 ± 0.3 b
Water activity	0.21 ± 0.02 b	0.30 ± 0.01 a
Bulk density (g/cm^3)	0.39 ± 0.00 a	0.40 ± 0.00 a
Span	2.01 ± 0.03 a	2.00 ± 0.05 a
Mean particle diameter D_{43} (μm)	27.28 ± 0.51 a	26.96 ± 0.46 a
Surface area (m^2/g)	0.60 ± 0.004 b	0.61 ± 0.01 a

Means ± standard deviation. Values followed by the same letter did not differ statistically according to the Tukey test at 5%.

3.3 Fatty acid profile and peroxide index

No significant differences in the fatty acid compositions were observed between samples (Table 2), which had higher PUFA than MUFA contents, mainly the linoleic acid content. Thus the inclusion of grape seed oil in the human diet can bring health benefits, since linoleic acid is an essential fatty acid and regulates low density lipoprotein (LDL) (Wijendran & Hayes, 2004).

Due to high unsaturated fatty acid content (Table 2), grape seed oil was vulnerable to lipid oxidation during spray drying, favoring to the formation of fat peroxides by the reaction with molecular oxygen (Table 2). In addition, the microspheres presented low encapsulation efficiency (Table 1), leading to exposition of surface oil to high temperature during the stay of the particles in the cyclone, which supplies more energy for the formation of peroxides.

Although the samples encapsulated with GA and GA/MD presented no significant differences with respect to encapsulating efficiency (Table 1), the latter was more effective in protecting the lipid fraction inside the microspheres against oxidation, than the sample with GA. This result may be related to the hydrophilic nature of maltodextrin, which could have decreased the oxygen (hydrophobic substance) permeability of the microsphere wall (Dzondo-Gadet et al., 2005), resulting in a reduction in oxygen mobility from the atmosphere surrounding the surface particles into the encapsulated oil.

3.4 Total phenolic compound content and antioxidant activity

The control sample showed a higher total phenolic compound (TPC) content than the oils encapsulated (Table 2). After spray drying process, only 47% and 60% of TPC were preserved in microspheres formulated with GA and GA/MD, respectively. This act could be due to lower encapsulation efficiencies obtained for both microspheres (Table 1), in which significant amount of surface oil containing phenolic compounds was exposed to oxygen and high temperature. Thus, the poor nutritional retention showed possible degradation of phenolic compounds during spray drying process. However, it is important highlighted that

Table 2. Fatty acid composition, antioxidant activity, phenolic compound content and peroxide index of the control and microencapsulated samples of the grape seed oil.

		GA	GA/MD	Control
Fatty acids (%)	c 14:0	0.1 ± 0.0 a	0.1 ± 0.0 a	0.1 ± 0.0 a
	c 16:0	8.8 ± 0.3 a	8.8 ± 0.2 a	8.8 ± 0.1 a
	c 18:0	3.3 ± 0.1 a	3.2 ± 0.0 a	3.2 ± 0.1 a
	c 18:1	20.9 ± 0.0 a	20.9 ± 0.1 a	20.8 ± 0.2 a
	c 18:2	65.7 ± 0.2 a	65.8 ± 0.1 a	65.9 ± 0.2 a
	c 18:3	0.7 ± 0.1 a	0.6 ± 0.0 a	0.7 ± 0.1 a
	c 20:0	nd	0.1 ± 0.0 a	0.1 ± 0.0 a
	SFA	12.3 ± 0.2 a	12.2 ± 0.2 a	12.1 ± 0.1 a
	MUFA	21.0 ± 0.0 a	21.0 ± 0.1 a	20.9 ± 0.1 a
	PUFA	66.4 ± 0.3 a	66.4 ± 0.1 a	66.6 ± 0.1 a
Phenolic compounds content (mg gallic acid/g oil)		32.3 ± 0.4 c	41.6 ± 0.1 b	68.8 ± 0.2 a
DPPH (mM Trolox/g oil)		11.9 ± 1.1 b	17.3 ± 2.1 b	44.1 ± 1.7 a
FRAP (mM Trolox/g oil)		27.3 ± 1.4 b	37.3 ± 0.3 a	16.9 ± 0.5 c
Peroxide index (meq/kg oil)		27.0 ± 0.6 a	22.5 ± 0.1 b	4.6 ± 0.2 c

Mean ± standard deviation. Values followed by the same letter did not differ statistically according to the Tukey test at 5%. nd = undetectable, defined as ≤0.05%. GA = gum Arabic; GA/MD = gum Arabic and maltodextrin (50:50). SFA: saturated fatty acids; MUFA: monounsaturated fatty acids; PUFA: polyunsaturated fatty acids.

these substances present a large diversity of structures, including phenols and phenolic acids, hydroxycinnamic acid derivatives and flavonoids (Ho, 1992). In grape seed oil, the main phenolic compounds identified are gallic acid, epicatechin and epicatechin gallate (Zhao et al., 2017). Although TPC content decreased after spray drying process, its composition could have been changed due to distinct encapsulation efficiency (Secolin et al., 2017) and thermal stability of each phenolic compound; and interconversion reactions to derived structures (Barcia et al., 2014; Cheynier, 2012).

Phenolic compounds are important for human nutrition, since plays the role of biological antioxidant. As consequence of the decrease of TPC content in grape seed oil encapsulated, a reduction in the DPPH radical scavenging activity was observed (Table 2). Some authors have also reported this negative effect of spray drying on the antioxidant activity of oils due to thermal and oxidative degradation of antioxidant substances (Calva-Estrada et al., 2018; Ferreira et al., 2016). On the other hand, although the oils encapsulated presented lower phenolic compound contents, they showed greater iron ion reducing capacity than the control sample (Table 2). This unexpected result was reported by Arana-Sánchez et al. (2010) for oregano oil microencapsulated by spray drying. The authors attributed this result to compositional changes in the oil during the microencapsulation process. Probably, in the current work, spray drying process could have favored the formation of some compounds possessing iron reducing capacity.

With respect to the wall material, higher antioxidant activity was observed when maltodextrin was used in combination with gum Arabic (Table 2), probably due to greater TPC content (41.6 mg gallic acid/g oil) than in powder formulated with only GA (32.3 mg gallic acid/g oil). Moreover, since maltodextrin 10DE and gum Arabic are sources of reducing sugars (BeMiller & Huber, 2007) and protein (Comunian & Fávaro-Trindade, 2016), respectively, it is plausible that the Maillard reaction could take place during thermal processing, resulting in formation of Maillard reaction products (MRP) with antioxidant properties (Zhang et al., 2017).

3.5 Physicochemical properties of the powders

Moisture content and water activity

The microspheres with GA presented higher moisture contents than those with GA/MD combination (Table 1). Moisture content is an indicator of the adequacy of the drying process, since water acts as a plasticizer and depresses the glass transition temperature, causing stickiness and caking of powders and increasing molecular mobility (Bhandari & Roos, 2017). On the other hand, the GA/MD powder presented a greater Aw than the GA powder. These results showed that the powder composition could influence the intensity with which water associates with the non-aqueous constituents. Pérez-Alonso et al. (2006) demonstrated that GA had more active adsorption sites on its surface with higher binding energies than MD. Thus these findings could explain the fact that the GA powder showed lower Aw at higher moisture contents than the GA/MD samples.

Particle size distribution, surface area and bulk density

The microspheres formulated with GA and GA/MD presented a small mean particle size (Table 1), as expected for spray-dried powder. Such a result negatively affects the flowability and dissolution properties of a particulate system (Vissotto et al., 2014; Hogekamp & Schubert, 2003). In addition, smaller particles have a higher total surface area (Table 1); as a consequence, there are a great affinity for moisture, leading to powder caking (Hogekamp & Schubert, 2003), and amount of surface oil available for oxidation.

All the samples presented trimodal particle size distributions (Figure 1b), favoring compaction, in which small particles can penetrate into the spaces between the larger ones, and segregation of the system, in which larger particles remaining at the top and smaller particles at the bottom (Santana et al., 2013). This heterogeneity of particulate systems can also be evaluated from the span values (Table 1). Similar results were reported by Santos et al. (2005) for paprika oleoresin microcapsules obtained by spray drying.

For the oils encapsulated with GA and GA/MD, the bulk densities presented no significant differences (Table 1) and were larger than the microspheres of vanilla extract (Calva-Estrada et al., 2018) and grape skin extract (Kalušević et al., 2017). A higher powder bulk density is desirable, requiring a smaller package volume, thus reducing the costs of packaging material, transport and storage.

Powder morphology

Figure 2 depicts the various sizes and shapes of the microspheres, which are typical for spray dried particles.

The microspheres presented a continuous wall and the absence of cracks or collapse, which is desirable to effectively protect the oil against oxidation. On the other hand, there were several shriveled particles, which were resulted from slow film

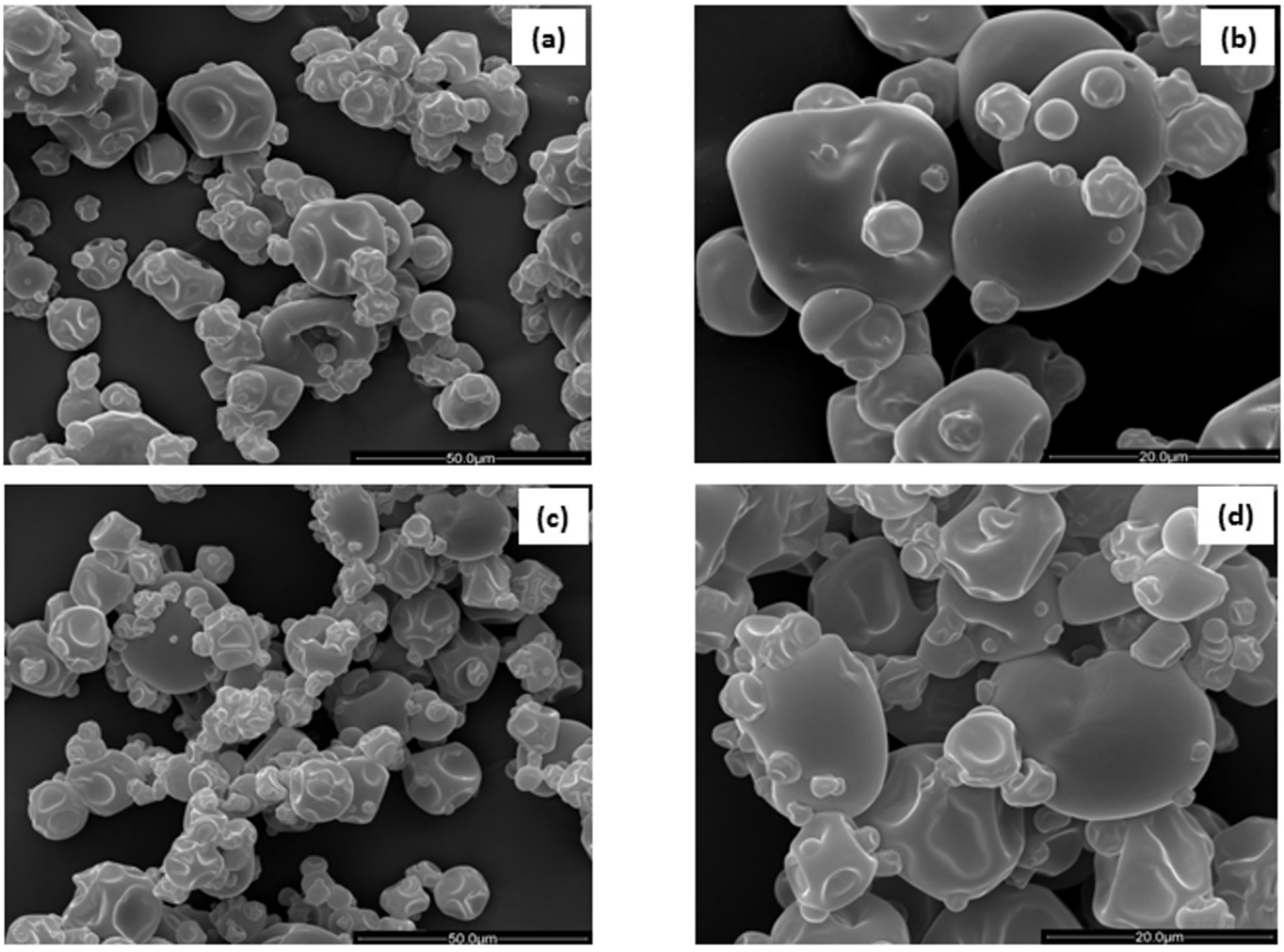

Figure 2. Morphology of the grape seed oil microspheres: (a) gum Arabic (magnification x2000), (b) gum Arabic (x5000), (c) gum Arabic/maltodextrin (x2000) and (d) gum Arabic/maltodextrin (x5000).

formation during drying of the atomized droplets, causing their shrinkage during the final stages of drying and cooling (Arana-Sánchez et al., 2010). The irregular shapes and presence of dents in the microspheres adversely affect the flow properties of the powders and increase the surface area, making the particles more susceptible to oxidation (Li et al., 2017).

4 Conclusions

Despite the microspheres with GA/MD had higher surface oil contents than those prepared with GA, the samples did not show any significant difference in encapsulation efficiency. Encapsulation with the GA/MD combination resulted in greater antioxidant activity according to FRAP and DPPH radical scavenging activity, and a lower peroxide value than the GA microspheres. However, the spray drying process significantly reduced the total phenolic compound content and DPPH radical scavenging activity, and favored lipid oxidation in the grape seed oil. On the other hand, the reducing capability of the iron ion increased after the microencapsulation process. The addition of maltodextrin to gum Arabic in the wall material at 50:50 ratio could be suggested as an alternative due to the increasing prices of gum Arabic and its oscillation in supply.

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