

Hydrocolloids and partially defatted cake on encapsulation of baru oil (*Dipteryx alata* Vogel): a study on emulsion, particle, and oxidative stability

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Abstract

The baru (*Dipteryx alata* Vogel) is a fruit of the Brazilian Cerrado, composed of a pleasant-tasting almond with a high oil content. The consumption of vegetable oils is suggested by the World Health Organization, but vegetable oils lack protection when extracted from their food matrix. Thus, the hydrocolloids Arabic gum (AG) and maltodextrin (MD) as well as the partially defatted cake (PDC), a co-product of the baru cold-pressed oil, were used to produce baru oil microparticles by spray drying. Fatty acid profile, emulsion stability, microparticle quality, and oxidative stability were evaluated. Baru oil is rich in omega-9 (47.3%). AG/MD (25% maltodextrin) showed better emulsion stability after 1h of preparation, higher zeta potential ($-28.87 \text{ mV} \pm 0.026$) and encapsulation efficiency ($76.72\% \pm 0.002$), and lower peroxide content after 4 weeks at 60°C (28.38 meq/kg). The addition of PDC improved the efficiency parameters in relation to the standard formulation (AG). The replacement of 25% of AG by MD improved the emulsion and microparticles' characteristics and better protected the baru oil against oxidation. Through a well-known technique, this study found technological use for a co-product of the extraction of baru oil and made good use of maltodextrin, a hydrocolloid with a lower relative cost.

1. Introduction

The barueiro (*Dipteryx alata* Vogel) is a native tree of the Brazilian Cerrado. This tree produces the baru, a fruit with light brown bark, sweet pulp, and a single edible almond. The baru almond (BA) has high added value due to its rich nutritional composition, mainly in lipids, followed by proteins, carbohydrates, minerals, fibers, and bioactive compounds (Carrazza & Avila, 2010; Lima et al., 2022). Several studies have shown antioxidant activity of the baru almond (and consequently of its oil) against oxidative stress, cardiovascular diseases risk, cancer, atherogenic indices, gastrointestinal diseases, abdominal adiposity, and wound healing. In addition, it helps to improve the lipid profile in overweight individuals (Bailão et al., 2015; Bento et al., 2014; Coco et al., 2021; de Souza et al., 2018; Fetzer et al., 2018; Ragassi Fiorini et al., 2017). Furthermore, the increase in Cerrado deforestation and therefore the extinction of its species has been discussed. (Colman et al., 2022; Lopes dos Santos et al., 2021; Parente et al., 2021; Resende et al., 2021). So, this research also serves as an alert for it.

From the almond, it is possible to extract the baru oil (BO), rich in unsaturated fatty acids (about 77%), being oleic acid the major component (48%), followed by linoleic (25%) and linolenic acid (0,2%), which have important biological functions (Sá et al., 2020). After the cold pressing the oil, the partially defatted cake of baru (PDC) is obtained, a co-product rich in proteins and bioactive compounds, which is usually wasted. Some authors have used PDC for nutritional purposes (L. de L. de O. Pineli et al., 2015a, 2015b; Rojas et al., 2019), but so far, its technological potential has not been evaluated. In the plant kingdom, finding a single source of quality protein is rare, so the combined consumption (such as cereals and pulses) of plant protein sources is suggested. Surprisingly, the baru almond presents all the essential amino acids within the reference standards (WHO/FAO/UNU) (De Oliveira Sousa et al., 2011; Sá et al., 2020). Technologically, due to the presence of legumins (proteins with emulsifying capacity) in baru almond, it is possible to use it in emulsions such as oil-in-water systems (Cruz et al., 2011).

After extracting vegetable oils from their matrices, it is proposed to apply technological processes that focus on protecting them against light, heat, and oxygen, to avoid nutritional and sensorial oxidative alterations. Microencapsulation is widely cited in the literature as one of the most efficient techniques for protecting compounds. The technique protects the material from environmental factors, reducing the progress of lipid oxidation reactions. Besides, it is possible to change the physical state of the oil, in order to enable its incorporation into various food matrices. (Annamalai et al., 2020; Lai et al., 2021; L. Pineli et al., 2015; L. de L. de O. Pineli et al., 2015a; Rojas et al., 2019; Vera et al., 2009). The selection of encapsulating material is fundamental for the process efficiency. Polysaccharides are widely used to encapsulate oils, while their combination with proteins (protein-polysaccharide complex) can improve the emulsion stability (Benito-Román et al., 2020; Murali et al., 2016).

Among different microencapsulation techniques, the spray dryer is widely used in the encapsulation of oils due to the versatility, low operational costs, and high equipment availability (Gharsallaoui et al., 2007; Jafari et al., 2008; Lai et al., 2021; Mohammed et al., 2020; Rojas et al., 2019; Shahidi & Han, 1993). Research based on encapsulation of baru oil are rare and therefore this work aimed to evaluate the impacts of the combination of hydrocolloids (Arabic gum – AG and maltodextrin - MD) and PDC before and after obtaining baru oil particles through spray drying. The focus is on the characterization of the emulsion, the particles, and the oxidative stability. For the first time, the co-product of the cold-pressed almond is used in the microencapsulation area.

2. Material And Methods

2.1. Materials

Baru almonds and Arabic gum were acquired from Armazém São Vito – Comércio de Produtos Alimentícios Ltda® (São Paulo, Brazil) and Sinergia Científica® (Campinas, São Paulo), respectively. Maltodextrin Mor-Rex® 1910 (DE 10) was donated by Ingredion Brasil Ingredientes Ltda® (Mogi Guaçu, São Paulo) and the partially defatted cake was obtained from the baru oil cold pressing process. Other materials and reagents used were all analytical grades.

2.2. Cold pressing baru oil and partially defatted cake of baru (PDC)

The oil was cold pressing (pressure of 60 ton force), after grinding the almonds in a laboratory Blender Waring Commercial® (Torrington, Connecticut, USA). After the pressing, the PDC was gathered, re-crushed, and sieved (0.125 mm) to improve their dispersion in water. The oil fatty acids composition was determined by GC/MS: (C16:0–6.5%; C18:0–5.4%; C20:0–1.6%; C22:0–5.2%; C24:0–6.3%; C18:1–47.3%; C18:2–21.4%; C18:3 – 3.6%; C20:1–2.6%).

2.3. Preparation of emulsions

Emulsions were prepared using the phase inversion technique, after preliminary tests to define the best process conditions. Firstly, the encapsulating materials were mixed with distilled water plus Tween 80® and left under stirring in a shaker for 12 h (aqueous phase). The Span 80® was added to the baru oil (oily phase). Then, the aqueous phase (heated to 75°C on a hot plate) was slowly poured onto the oily phase (70°C). The emulsions were homogenized in Ultra Turrax® T5 Basic IKA at 22000 rpm for 3 min. The amount of each constituent of the emulsions was defined in preliminary tests (not shown), and the composition of the emulsions is shown in Table 1.

2.4. Emulsion and stability

2.4.1. Dynamic turbidimetric and zeta potential (ζ)

The dynamic turbidimetric was measured at 25°C in Turbiscan LAB® Expert Formulation (L'Union, France). The stability was monitored at 15 min intervals during 1 h, a time considered adequate for the applied volume for the atomization process (100 mL of emulsion). Zeta potential was determined by Zetasizer® Nano-ZS equipment, Malvern Instruments (UK). The emulsions were previously dilute in deionized water, at a concentration of 2% (v/v).

2.4.2. Droplet size distribution and mean droplet size

Oil droplet size distribution and mean droplet size, expressed as Sauter mean diameter ($D_{3,2}$), were measured in Mastersizer® 2000 (Malvern, United Kingdom) equipment (Noguera et al., 2022). The analysis was carried out immediately after the emulsion preparation.

2.5. Microencapsulation process

After emulsification step, the emulsions were atomized in a Mini Spray Dryer B-290 Buchi® (Flawil, Switzerland) to obtain microparticles of baru oil, using double fluid atomizer with 0.5 mm of nozzle diameter, inlet air temperature of $170 \pm 5^\circ\text{C}$, outlet air temperature of $110 \pm 5^\circ\text{C}$, feed flow of 5 mL/min, air flow for atomization of 600 L/h, and drying air flow of 35 m³/h. The microparticles produced were collected and stored in plastic packaging in darkness. Three independent batches were developed for each treatment.

2.6. Characterization of microparticles

2.6.1. Encapsulation efficiency (EE) and oil loaded (OL)

The encapsulation efficiency was evaluated based on relation between the mass of inner oil and the mass of initially added oil to the emulsion (theoretical oil, TEO), considering that there were losses of the baru oil before, during, and after atomization. To determine the inner oil, the surface oil (SO) and total oil (TO) were also measured. For SO, 30 mL of n-hexane were mixed with 3 g of particles in a Falcon tube and vortexed for 5 min, at room temperature (25°C). The mixture was vacuum filtered using a Buchner funnel and Whatman paper n° 1, and the collected powder was washed twice with 10 mL of n-hexane.

The particles were taken to the oven at 40°C to remove the residual n-hexane. Thus, the mass of surface oil was determined by a gravimetric measure.

On the other hand, 3 g of particles were mixed with 25 mL of distilled water (60°C) and magnetically stirred for 10 min aiming to disrupt the particles and to release inner oil. The mixture was added to 120 mL of n-hexane:isopropanol (3:1, v/v) and stirred again for 15 min to promote the migration of the oil from the microparticles to the organic phase. Finally, phase separation was observed, and the organic supernatant (baru oil plus solvent) was collected. Finally, this phase was rotoevaporated at 45°C (under vacuum) to remove the solvents and the remainder was considered as total oil (TO). Equations 2 and 3 were used to calculate the encapsulation efficiency (EE, % w/w) and oil loading (OL, %w/w), respectively.

$$EE(\%w/w) = \frac{TO - SO}{TEO} \cdot 100(2)$$

$$OL(\%w/w) = \frac{TO}{TEO} \cdot 100(3)$$

Where, TO, SO and TEO represents total oil, surface oil, and theoretical oil, respectively.

2.6.2. Quality parameters directly influenced by water

Several quality parameters are affected by the presence and interactions of water with solutes (from a free to fully bound state). Water activity (A_w) and moisture were determined in an Aqualab® Model 3TE digital hygrometer (25°C) and direct desiccation in oven (105°C), respectively. The moisture was calculated by weight difference. Hygroscopicity was determined through a Tecnal® TE4001 Climatic Chamber with temperature and humidity controlled (25°C and 75% RH) for one week while wettability by static wetting test (Cai & Corke, 2000; Fuchs et al., 2006). The hygroscopicity was expressed in percentage on a dry basis (g of adsorbed water / 100 g of particles) and wettability was express in time (min).

The bulk density (g/cm³) and the packed bulk density (g/cm³) were measured by the methodologies of A. M. Goula & Adamopoulos, (2004) and H. C. F. et al Carneiro (2013), respectively. Both densities were calculated by the relation between the particles mass and the volume occupied by them. The real density (g/cm³) was determined, at 25 °C, in an AccuPyc® 1330 V2.02 2399 automatic helium gas pycnometer (Norcross, USA). The particle porosity (%) was calculated by the relation between the packed bulk density and the real density of the particles (Noguera et al., 2022).

2.6.3. Particle size distribution and mean particle size

The particle size distribution and mean particle size, expressed as the Brouckere mean diameter ($D_{4,3}$), were measured in Horiba® LV 950-V2 (Kyoto, Japan) equipment. The span value (polydispersity index) was also calculated as the difference between the cumulative distribution of 90% and 10% of the total particles divided by the cumulative distribution of 50% of the total particles (Noguera et al., 2022).

2.6.4. Scanning Electron Microscopy

The particles were observed using a JEOL® JSM 5800LV microscope (Massachusetts, USA) operating under a voltage of 10 kV and a current of 60 pA, after undergoing vacuum deposition of gold ions (Sputter BAL-TEC® Balzers SCD 050 equipment,) with coating for 150 s.

2.7. Oxidative stability

The auto-oxidation phenomenon was evaluated by two distinct but complementary methods – peroxide production and hexanal detection. The samples of free baru oil (control) and particles were stored in airtight glass jars and left in an oven for four weeks at 60°C.

2.7.1. Peroxide index

The extraction of oil from microparticles was based on the methodology described in 2.6.1. The peroxide index was determined according to the AMERICAN OIL CHEMISTS' SOCIETY (AOCS Official method Cd 8–53) methodology.

2.7.2. Hexanal detection

For hexanal detection, headspace/gas chromatography was used. The headspaces of the vials (1 mL) containing the free baru oil or particles were injected into an Agilent® model HP6890 gas chromatograph with the assistance of a gas tight syringe. The analysis was performed during four weeks of storage. The gas chromatograph was equipped with an Agilent® model HP 5975 selective mass detector and capillary column HP-5MS (30 m x 0.25 mm x 0.25 µm). Operating conditions: 1:40 split mode, injector: 220°C, detector: 250°C; column initial temperature 35°C; heating rate of 3°C/min to 150°C, 2 min. Helium gas was used as a carrier gas at a flow rate of 1 mL/min.

2.8. Statistical analysis

The results were submitted to analysis of variance (ANOVA), with help of the software MiniTab® (version 19), to verify the existence of a significant difference among treatments and to compare means two by two (Tukey's test, $p < 0.05$). All experiments were carried out in triplicate ($n = 3$).

3. Results And Discussion

3.1. Dynamic turbidimetric and zeta potential (ζ)

About dynamic turbidimetry, the Fig. 1 (A-D) shows light backscatter curves as a function of the height on 0, 15, 30, 45, and 60 min. It was possible to observe the occurrence of creaming, sedimentation, clarification, flocculation, and coalescence phenomena in emulsions. The AG/MD emulsion was more stable than the AG, a fact observed by a minimal displacement of the backscatter curves with time (Fig. 1B). The AG emulsion presented flocculation and coalescence phenomena (Fig. 1A).

The emulsion MD/AG (Fig. 1C) showed an increase in light backscatter in the upper part of the cell, typical of the creaming process (cloudy or opaque region) after coalescence of oil droplets, and a reduction in light backscattering was noticed on the lower part of this emulsion, typical from the clarification process (less cloudy or transparent to light). The use of PDC in O/W emulsion resulted in destabilization phenomena (Fig. 1D). However, this material may have contributed to the greater stability when compared to the MD/AG emulsion, which may indicate the adsorption of the cake proteins on the surface of the oil droplets.

When comparing the global stability of the emulsions in relation to time (Turbiscan Stability Index, TSI) (graph not shown), it was observed that the AG/MD emulsion was the most stable over the 1 hour of the experiment. However, an interesting fact observed is that the AG/MD/PDC emulsion was the 2nd most stable (very similar to the AG/MD behavior) up to 30 min, which may indicate the use of PDC for short-time atomization purposes. The MD/GA emulsion was the most unstable during the analysis.

Consoli et al. (2017) reported that emulsions containing zeta potential greater than + 30 mV or lower than - 30 mV tend to be more stable, due to greater electrostatic repulsion between the oil droplets, preventing aggregation and coalescence. According to M. B. Silva et al. (2018), negative zeta potential values indicate that polymers have negative residual charges, resulting from their molecular conformations. Potential values of all emulsions were higher than - 30.0 mV (Table 1). MD/AG formulation presented the value of - 7,14 mV. This observation is evidenced by the low contribution of maltodextrin to the stability of the emulsions mentioned in the dynamic turbidimetry analysis. In the AG/MD/PDC formulation, the presence of PDC, even at a concentration of 1g/100 mL may have contributed to the increase in the zeta potential, possibly by negatively charged amino acid residues in the cake proteins adsorbed on the surface of the droplets (Gomes & Kurozawa, 2020).

Table 1

Composition of oil-in-water emulsions produced by phase inversion method and results on emulsion stability - zeta potential and mean droplet diameter (n = 3)

Formulations				
Composition (g/100 mL)	AG	AG/MD	MD/AG	AG/MD/PDC
Baru oil	5	5	5	5
Arabic gum	25	18,75	6,25	12
Maltodextrin	-	6,25	18,75	12
PDC of baru	-	-	-	1
Emulsion stability parameters				
Zeta potential (mV)	- 13,36 ^b ± 0,025	- 28,87 ^a ± 0,026	-7,14 ^c ± 0,052	- 26,76 ^a ± 0,052
D _{3,2} (µm)	3,18 ^c ± 0,045	3,76 ^b ± 0,098	5,85 ^a ± 0,015	5,96 ^a ± 0,036
AG = Arabic Gum, AG/MD = Arabic Gum and Maltodextrin, MD/AG = Maltodextrin and Arabic Gum, AG/MD/PDC = Arabic Gum, Maltodextrin, and Partially Defatted Cake. All formulations contain 0.6 g/100 mL of Tween 80® and 0.4 g/100 mL of Span 80®. The final volume of each emulsion was adjusted with water to 100 mL. The emulsions were produced by phase inversion method. The zeta potential and the mean diameter of the droplets were performed immediately after the preparation of the emulsions. Total of 3 measurements (n = 3). Mean value ± standard deviation (statistical analysis one-way ANOVA). Different letters in the same row indicate significant difference between samples by Tukey's test (p < 0.05).				

3.2. Droplet size distribution and mean droplet size

Emulsions exhibited bi/trimodal distribution: two droplet populations for AG, AG/MD, and MD/AG and three for AG/MD/PDC (figure not shown) and the mean droplets size is presented in Table 1. Emulsion AG presented droplets of smaller average diameter with the mostly varying between 3 and 20 µm, reflecting the high emulsifying capacity of Arabic gum. The emulsion tends to become more stable when smaller droplet sizes are obtained. In addition, smaller droplets can contribute to greater encapsulation efficiency and lower surface oil of the particles (Noguera et al., 2022; Taheri & Jafari, 2019).

The higher average droplet diameters of AG/MD and MD/AG emulsions in relation to AG may indicate that the presence of maltodextrin promoted an increase in droplet size, so that maltodextrin and gum arabic, when used in these proportions, may not contribute to the formation of emulsion. A likely explanation is the lack of emulsifying capacity of maltodextrin in relation to Arabic gum, a material that has a surface-active property (protein fraction) capable of almost entirely coating the oil droplets (Gharsallaoui et al., 2007). A closer look, however, demonstrates that although AG has a smaller droplet size, it should be noted that it was the AG/MD emulsion that exhibited better stability after 1 h of preparation, demonstrating better stabilization capacity (Shishir et al., 2018). On the other hand, higher

proportions of maltodextrin in relation to the Arabic gum (MD/GA) must have promoted a greater reduction in viscosity, facilitating the coalescence and destabilization of the emulsion.

The AG/MD/PDC emulsion apparently presented droplets with a larger average diameter. However, there is a hypothesis that the insoluble particles present in the PDC have interfered with the $D_{3.2}$ of this formulation. It is suggested that the third population, observed in the droplet size distribution profile of this emulsion, refers to PDC particles. In view of the above, both the wall material and its concentration influence the average diameter and size distribution.

3.3. Characterization of microparticles

All the results of particle characterization are exhibited in Table 2.

Table 2

Results of the characterization of microparticles loaded with baru almond oil - parameters of efficiency, quality, and size distribution of the particle obtained by spray drying (n = 3)

Characterization of particles				
	AG	AG/MD	MD/AG	AG/MD/PDC
EE (% w/w)	62,57 ^b ± 0,006	76,72 ^a ± 0,002	57,45 ^c ± 0,007	67,88 ^d ± 0,001
OL (% w/w)	85,69 ^d ± 0,001	91,59 ^a ± 0,002	83,91 ^c ± 0,004	87,66 ^b ± 0,002
Water activity	0,123 ^c ± 0,002	0,137 ^b ± 0,001	0,141 ^a ± 0,001	0,134 ^b ± 0,001
Moisture (% w/w)	3,34 ^c ± 0,051	3,64 ^{bc} ± 0,062	4,17 ^a ± 0,028	3,93 ^{ab} ± 0,096
Wettability (min)	6,56 ^a ± 0,038	5,08 ^b ± 0,053	3,92 ^c ± 0,051	4,77 ^b ± 0,049
Hygroscopicity (g.100g ⁻¹)	12,06 ^a ± 0,202	9,82 ^b ± 0,332	7,65 ^c ± 0,242	8,09 ^c ± 0,233
Bulk Density (g/cm ³)	0,20 ^a ± 0,009	0,20 ^a ± 0,009	0,24 ^b ± 0,004	0,22 ^b ± 0,009
Packed Bulk Density (g/cm ³)	0,37 ^a ± 0,032	0,36 ^a ± 0,047	0,46 ^b ± 0,048	0,46 ^b ± 0,056
Real Density (g/cm ³)	1,18 ^b ± 0,001	1,23 ^c ± 0,009	1,29 ^a ± 0,009	1,21 ^d ± 0,010
Porosity (%)	83,10 ^a ± 0,024	83,80 ^a ± 0,016	81,40 ^b ± 0,026	81,90 ^b ± 0,014
D _{4,3} (µm)	16,51 ^a ± 0,557	9,80 ^b ± 0,178	9,04 ^b ± 0,350	16,50 ^a ± 0,490
Span values	2,34 ^a ± 0,054	1,50 ^b ± 0,017	1,29 ^b ± 0,051	4,80 ^c ± 0,192
AG = Arabic Gum, AG/MD = Arabic Gum and Maltodextrin, MD/AG = Maltodextrin and Arabic Gum, AG/MD/PDC = Arabic Gum, Maltodextrin, and Partially Defatted Cake. EE (particle encapsulation efficiency, % w/w), SO (particle surface oil, % w/w), TO (particle total oil, % w/w), OL (particle oil loading, % w/w), D _{4,3} (mean particle size, µm) span values (polydispersity index of the particles). Total of 3 measurements (n = 3). Mean value ± standard deviation (statistical analysis one-way ANOVA). Different letters in the same row indicate significant difference between treatments by Tukey's test (p < 0.05).				

3.3.1. Encapsulation efficiency (EE) and oil loaded (OL)

The percentage of total oil recovered in all formulations was lower than the percentage of TEO value (15.38% w/w), confirming the occurrence of oil losses during the process (section 2.6.1). AG/MD particles stood out for encapsulation efficiency when comparing to standard (AG). This result was to be expected,

since the AG/MD emulsion showed the best stability, as observed in the zeta potential and dynamic turbidimetry results. Thus, the replacement of a higher cost polymer (Arabic gum) for a lower cost one (maltodextrin) and with improvements in the technological quality of the particles deserves to be highlighted.

However, further increasing the concentration of maltodextrin caused a reduction in the efficiency parameters (MD/AG). Possibly, the larger droplet size observed in this emulsion, discussed in topic 3.2, impaired the encapsulation process and, therefore, resulted in greater amount of oil on the particle surface (Tan et al., 2005). On the other hand, the use of PDC together with AG and MD resulted in greater encapsulation efficiency when compared to standard. This result is notable and encourages the use of PDC, as a potential encapsulating material.

AG/MD carried the highest amount of baru oil, meaning that losses occurred during processing were smaller. MD/AG particles, in turn, showed lower oil carrying probably due to the absence of the emulsifying property of maltodextrin. Thus, it was observed that the type of wall material and its concentration significantly effect encapsulation efficiency and oil loaded parameters.

3.3.2. Particle quality parameters

Presence or absorption of water by microparticles can favor deterioration reactions during storage, contributing to lipid oxidation and microbiological deterioration. Thus, it is recommended that powders has values of $A_w < 0.3$ and moisture between 3 and 4% w/w (Klaypradit & Huang, 2008). When the maltodextrin was added or Arabic gum was reduced, both water activity and moisture increased. Among the formulations, MD/AG presented the highest values for these two parameters. Pérez-Alonso et al. (2006) points out that Arabic gum contains more active adsorption sites on the molecule surface (strong binding energy). In other words, this material is capable of quickly binding the water molecules, so that the free water content is usually lower in powders consisting of Arabic gum as an encapsulating agent. Maltodextrins from highly hydrolyzed starches (dextrose equivalent-DE of 20) tend to absorb more moisture, due to greater exposure of hydrophilic groups that easily bind to water (Silva et al., 2014). In this study, maltodextrin DE 10 (low degree of hydrolysis) was used.

AG particles presented a greater hygroscopicity, which is undesirable when referring to shelf life, as this phenomenon facilitates the occurrence of reactions that can lead to deterioration. A study developed by Goula et al. (2004) about tomato pulp atomization, points out that the hygroscopicity increases as moisture content decreases. This report is valid for the present work, in which formulations with lower moisture (AG and AG/MD) were the most hygroscopic. Regarding those containing maltodextrin, it is noteworthy that hygroscopicity has a strong influence of the DE, so that maltodextrins with lower DE (higher molecular mass) have a lower tendency to absorb moisture (Srinivasan & Parkin, 2019). For wettability, it is noted that increase in the concentration of maltodextrin is inversely proportional to the wettability time of the particles. This fact may be associated with the high hygroscopicity of this material.

Assessing density and porosity of powders helps to understand the conditions of storage, processing, packaging, and distribution. These parameters relate mass and volume occupied by particles as well as the presence of empty spaces (Barbosa-Cánovas & Juliano, 2005; Beristain et al., 2001; Mahdi et al., 2020). Bulk density considers empty spaces between particles. A higher concentration of maltodextrin (MD/AG) promoted a significant increase in bulk density when compared to the AG formulation. Larger amounts of added maltodextrin possibly contributed to high agglomeration and/or structural collapse of the particles, causing a decrease in the occupied volume (Bae & Lee, 2008). On the other hand, MD/AG particles presented more surface oil, which may contribute to particle agglomeration, resulting in increased density.

Packed bulk density minimizes gaps between particles. Higher packed bulk densities allow particles to be better stored and transported, using smaller packages because the smaller volumes. Furthermore, there will be a lower tendency for oxidation during storage, due to the lower concentration of occluded air in the empty spaces (Mahdi et al., 2020; Tonon et al., 2011). Real density values show that the formulations differed statistically from each other, ranging between 1.18 and 1.29 g.cm⁻³. It is possible to link this density to particle size so that particles of smaller sizes generally exhibit higher real densities, resulting from lower air volume filling the particles. AG and AG/MD particles presented the higher porosities, differed statistically of MD/AG and AG/MD/PDC. Higher porosity indicates the greater presence of oxygen in the bed: this fact can accelerate lipid oxidation reactions.

3.3.3. Particle size distribution and mean particle size

As shown in Table 2, the largest mean diameter refers to the AG formulation. One of the explanations may be the relationship between emulsion viscosity and particle size, so that more viscous emulsions take longer to dry, resulting in larger particles. The same principle applies to formulations with lower viscosity, such as those composed of maltodextrin. The span values (polydispersity index), in turn, ranged between 1.29 and 4.80. The AG/MD/PDC particles had a higher span value, probably due to the interference of the PDC components in the polydispersity of the particle size. AG/MD and MD/AG presented unimodal distribution, demonstrating greater homogeneity of powders, while AG and AG/MD/PDC presented bimodal distributions, represented by two populations of particles. Specifically for AG/MD/PDC, the presence of the PDC may have influenced this behavior, and the second “population” of particles observed possibly refers to the insoluble fraction (fibers) of PDC of baru.

Figure 2 refers to the cumulative distribution curves, from which it is possible to infer the frequency of each particle size range. Approximately 50% of AG particles had sizes between 1 and 10 µm. The addition of maltodextrin, in turn, increased the frequency of particles with sizes equal to or less than 10 µm, reaching almost 70% in the MD/AG particles. On the other hand, it can be noted that the addition of PDC resulted in a considerable increase in size (AG/MD/PDC), so that the vast majority had sizes greater than or equal to 100 µm. The hypothesis is based on the presence of PDC in the formulation, which can present larger sizes than 100 µm. The low viscosity of maltodextrin, even at high concentrations, may explain the formation of smaller particles of AG/MD and MD/AG, since less viscous emulsions tend to original smaller particles. Other authors have reported the relationship between particle size and texture

when the objective is the incorporation into food products. Larger particles than 30 µm are already detected on the palate, promoting a sensation of sandiness (Frascareli et al., 2012; Paucar et al., 2016). Thus, AG, AG/MD, and MD/AG formulations can certainly be incorporated in foods without causing negative effects on the sensory perception of consumers.

3.3.4. Scanning Electronic Microscopy

The microstructure of particles (Fig. 3) was observed in magnification of 1500x.

No relevant morphological differences were found among the particles. Some particles had a rough surface, characteristic considered undesirable for the flow and fluidity of the powders. In addition, this feature allows greater access of oxygen, facilitating the occurrence of oxidation, especially if particle has a high concentration of surface oil. Roughness or depressions found on surfaces may originate from the shrinkage of particles during drying and subsequent cooling, particularly in spray drying. Bae & Lee (2008) and Shamaei et al. (2017) reported that particles that contained maltodextrin in the composition were more likely to show cracks and/or fissures on the surface. However, Tolun et al. (2016) state that when only maltodextrin was used as a wall material, the particles are more uniform structures and smooth surfaces.

In general, the particles showed tendency to agglomeration, especially in the formulation that exhibited more surface oil (MD/AG). Regarding the AG/MD/PDC particles, no regions or structures that characterize the presence of PDC were identified. This fact may be related to the small amount of cake added, making it difficult to visualize and identify its components. Particles of different sizes were found so that the polydispersity discussed above was confirmed. Some authors attribute this characteristic to the spray drying technique, which produces particles of different sizes (Tonon et al., 2011).

3.4. Oxidative stability

The peroxide values of free and encapsulated baru oil (AG, AG/MD, MD/AG, and AG/MD/PDC) stored at 60°C for four weeks is shown in Table 3a.

Table 3

a Results of peroxide values of free and encapsulated baru oil at time zero and during the four weeks of storage in an oven at 60°C* (n = 3); **3b** Hexanal detection intensity of free baru oil and of the two formulations with lower peroxide values**

Time (weeks)	a. Peroxide values (meq peroxide/kg oil)				
	Free oil	AG	AG/MD	MD/AG	AG/MD/PDC
0	6,77 ^e ± 0,567	14,50 ^b ± 0,772	12,17 ^c ± 0,841	20,49 ^a ± 1,059	16,84 ^d ± 1,055
1	9,68 ^e ± 1,338	22,55 ^b ± 1,385	16,27 ^c ± 1,350	24,70 ^a ± 1,980	19,88 ^d ± 0,590
2	20,27 ^d ± 1,041	26,46 ^b ± 1,072	19,44 ^d ± 1,589	29,47 ^a ± 0,980	24,80 ^c ± 0,845
3	38,72 ^a ± 1,134	32,45 ^b ± 1,213	22,44 ^d ± 0,367	35,21 ^c ± 0,770	33,27 ^b ± 0,880
4	43,85 ^a ± 0,910	36,27 ^b ± 1,068	28,38 ^d ± 0,903	40,27 ^c ± 0,670	38,82 ^c ± 1,080
Time (weeks)	b. Hexanal detection intensity				
	Free oil	GA		GA/MD	
1	Low	Low		Low	
2	Medium	High		High	
3	Low	Medium		Medium	
4	Medium	Low		Low	
Free oil = baru oil non-encapsulated, AG = Arabic Gum, AG/MD = Arabic Gum and Maltodextrin. Total of 3 measurements (n = 3). Mean value ± standard deviation (statistical analysis one-way ANOVA). Different letters in the same row indicate significant difference between samples by Tukey's test (p < 0.05). *Time 0: right after oil extraction and microparticles production. Samples were stored in airtight glass vials. **Peak relative area: 5x10 ⁶ < Low < 10x10 ⁶ , 10x10 ⁶ < Medium < 20x10 ⁶ , High < 20x10 ⁶ .					

It was observed that week-by-week, treatments differed statistically from each other, except for AG and AG/MD/PDC treatments at the third week. At the initial time (week 0), the encapsulated oil presented a statistically higher peroxide index when compared to the free oil (control). As the particles were being formed, the contact on the heated surfaces of the equipment during the drying process, due to stickiness behavior of the oil surface, may have contributed to accelerate the oxidation reaction of the oil prematurely. Only in the third week it was observed the free oil (38.72 meq/Kg) exceeded the peroxide content of the spray-dried powders. After four weeks, the free oil was even more oxidized (43.85 meq/kg) compared to the encapsulated oil (all the treatments).

In general, vegetable oils have natural antioxidant compounds in their composition, such as tocopherols, sterols, carotenoids, and phenolic compounds that can provide oxidative stability. However, the roasting process of the commercial baru almonds acquired may have destroyed these compounds, causing the oil to oxidize after being extracted. In this context, microencapsulation can be applied to protect the oil

obtained from roasted almonds. Most likely, the physical barrier formed by the encapsulating material made it difficult for oxygen to get to the oil quickly. AG/MD treatment is noteworthy for its lower mean peroxide index at the end of the study (28.38 meq/kg), ensuring a greater protective effect of baru almond oil against oxidation. This fact was expected since this emulsion was the most stable and had the highest encapsulation efficiency. Thus, with a higher internalized oil content, oxygen access to the matrix is more difficult.

The process of oxidation of vegetable oil begins with the production of peroxides and ends in a pathway that produces different compounds, such as hexanal. Because this chemical compound is easy to volatility, it is possible to identify it by the GC/headspace and by its off flavor. It was decided to monitor the hexanal in the samples with the lowest peroxide index (AG and AG/MD) and for the free oil. The analysis was performed semi-analytically, comparing the peak areas of this compound for the different treatments. Table 3b shows hexanal detection intensity for the free baru oil, AG, and AG/MD samples week by week for 28 days. Using the GC/MS software and the NIST- 11 library, hexanal was identified with more than 90% of confidence level. As part of the interpretation of the results, the intensity level of hexanal detection was established in the samples (low, medium, and high intensity).

In the 2nd week, hexanal detection was more intense for the AG and AG/MD samples (high detection), reflecting the intense degradation of the initial peroxides (1st and 2nd weeks), as part of the final stage of auto-oxidation. For the free oil, cycles of medium and low detection of hexanal were observed, characterizing the absence of protection. From the 3rd week onwards, a possible protection of microparticles (lower peroxide levels and decay of hexanal detection) was observed, when compared to free oil. This behavior can be explained by the barrier created by the polymers, making it difficult for oxygen to interact with the internal oxidized oil, delaying the oxidation process. Another possibility is the occurrence of hexanal degradation (H. C. F. Carneiro et al., 2011). Aldehydes can react with oxygen present in the environment, resulting in hexanoic acid. Hexanoic acid was detected in the samples in the third week, which coincided with the decay of hexanal (at a retention time of 12.5 min).

4. Conclusion

The emulsions were suitable for the encapsulation process, despite instabilities over time. The particles showed good technological quality, with humidity and water activity lower than 5% and 0.2, respectively, in addition to micro size and high porosity, characteristic of the production of particles by spray drying. The microparticles helped to protect the baru oil against oxidation, especially when the main co-product (partially defatted cake) and a low-cost polymer (maltodextrin) were also used. Finally, this study helps in the knowledge and in the preservation of a native species of the Cerrado, a hotspot area, that has remarkable potential of application in several areas.

Declarations

Data availability statement: The datasets generated during and/or analysed during the current study are available from the corresponding author on reasonable request.

Author's contribution: Orientation: Rodney Alexandre Ferreira Rodrigues; Supervision: Angela del Pilar Flores Granados; Idea conception: Nathan Hargreaves Noguera; Development: Dyana Carla Lima.

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Figures

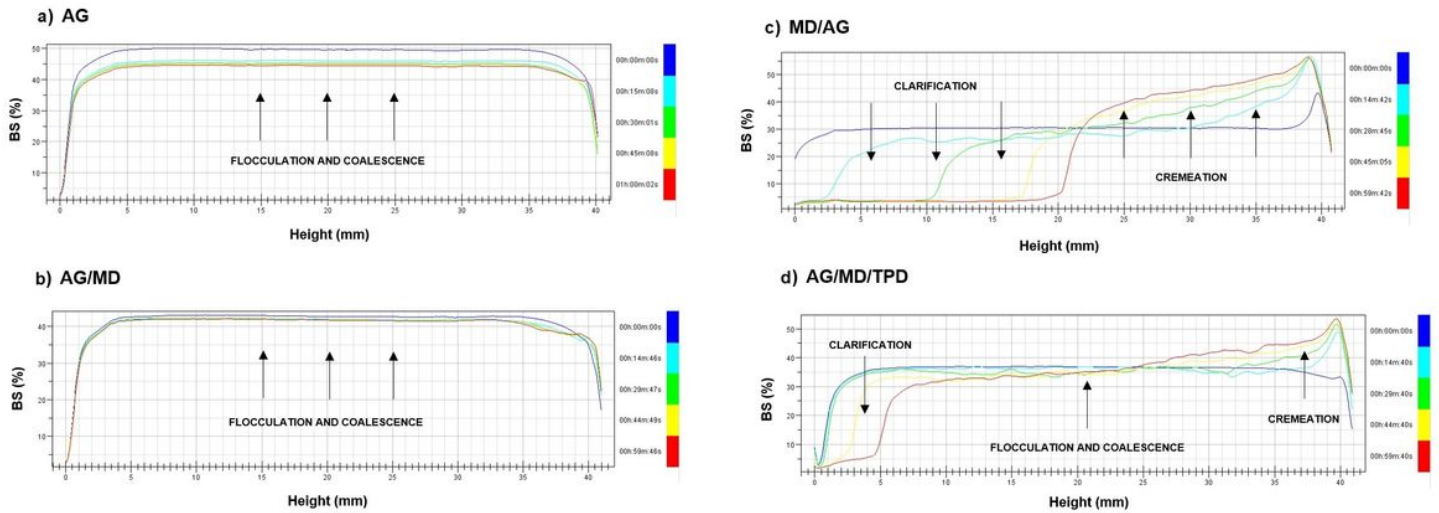


Figure 1

(a-d) Dynamic turbidity of emulsions containing baru almond oil measured by backscattering curves (%) as a function of time (15 in 15 minutes up to 1 hour) and cell height (mm): a study of stability

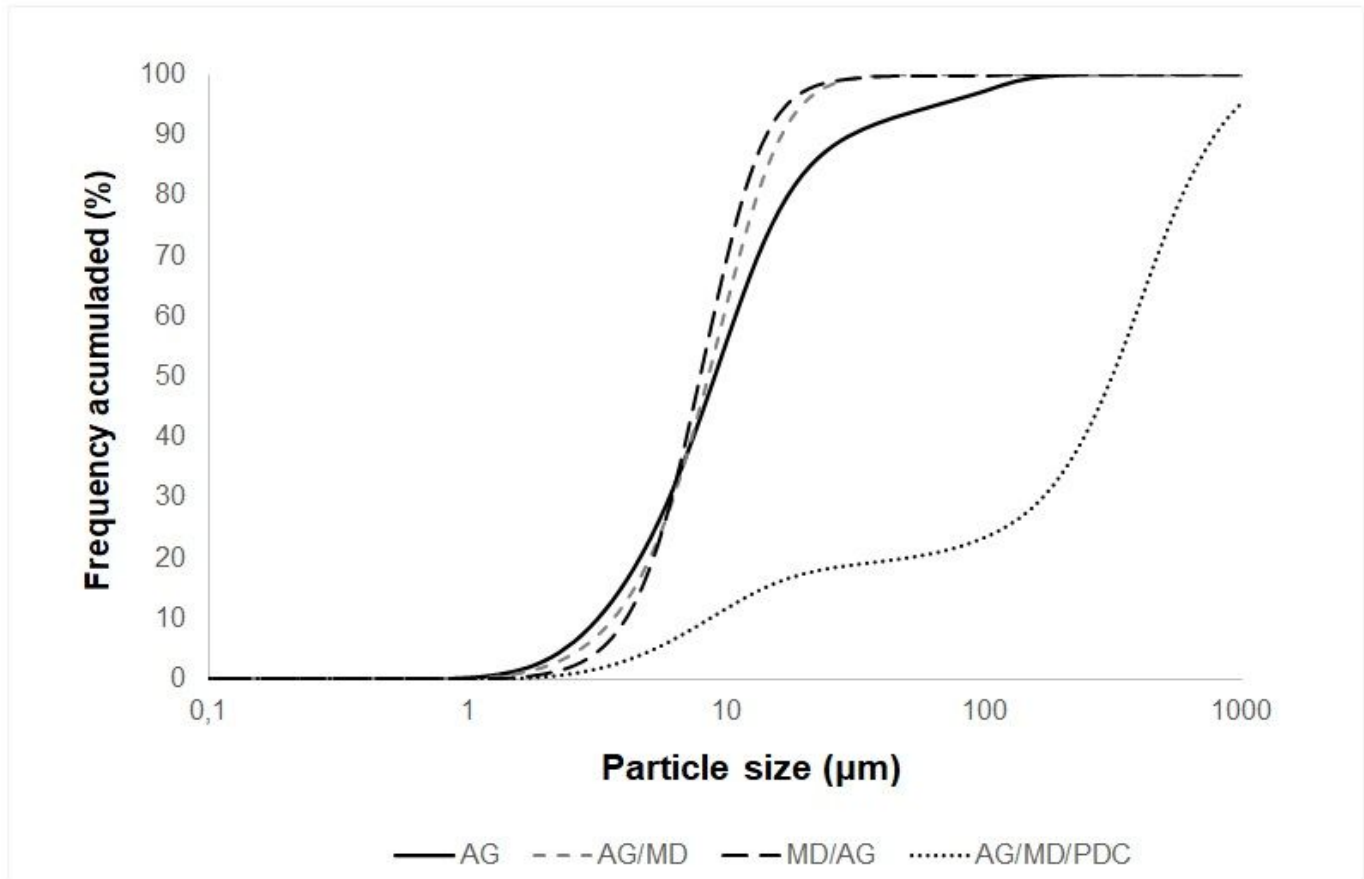


Figure 2

Cumulative distribution curves related to particle size (μm) produced by spray drying as a function of their frequencies (%): the addition of partially defatted cake increased the frequency of larger particles.

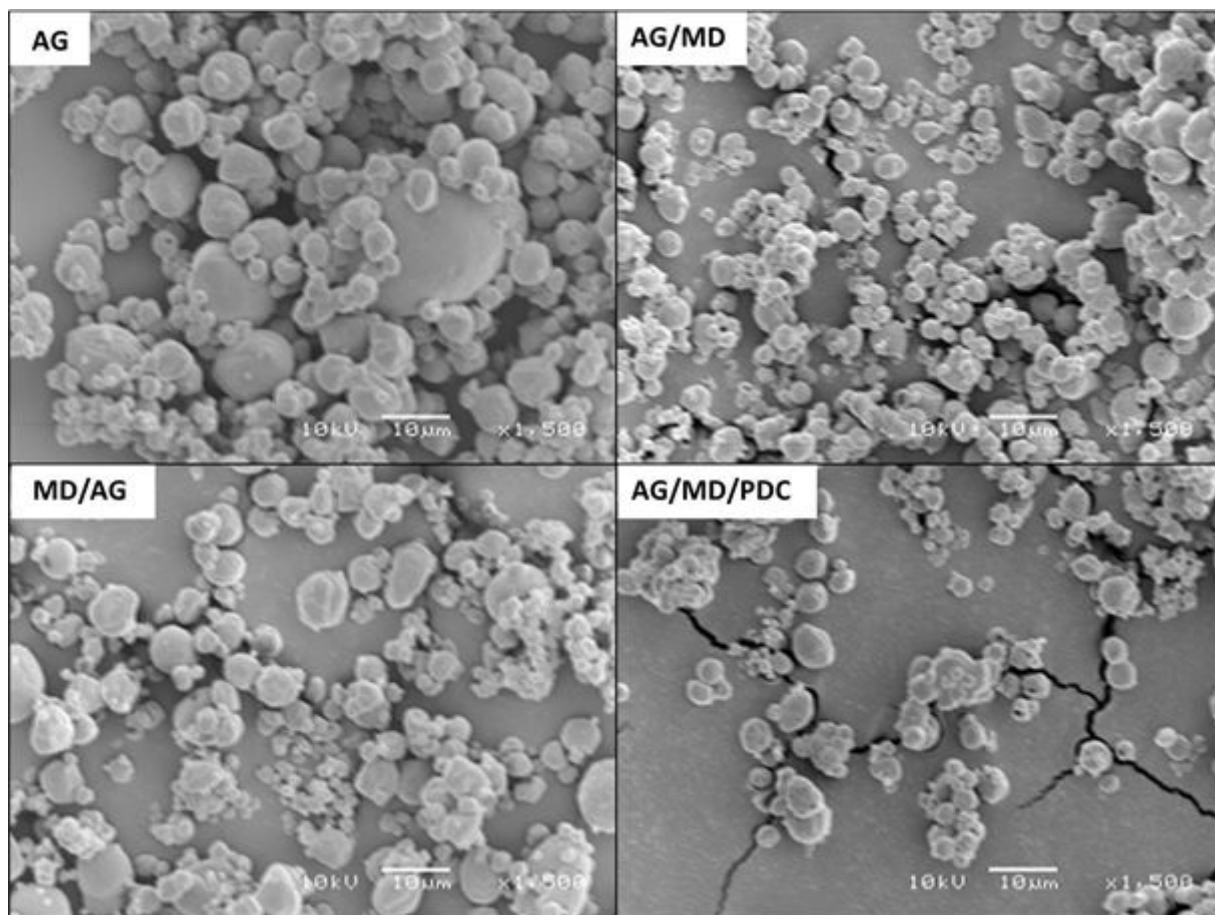


Figure 3

Scanning electron microscopy of microparticles containing baru almond oil at 1500x magnification: roughness and spherical shape are intrinsic characteristics of spray particles]