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Microencapsulation of green coffee oil: Encapsulation efficiency, morphology, and bioactive compound retention

Microencapsulación de aceite de café verde: Eficacia de la encapsulación, morfología y retención de compuestos bioactivos

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Abstract

The development of functional foods that promote health benefits is one of the most active areas of research in food processing. The supercritical fluid green coffee oil has proven to be a rich source of polyunsaturated fatty acids and chlorogenic acid, which is a potent antioxidant. This study investigated the physicochemical characteristics of green coffee oil microcapsules, including encapsulation efficiency, morphology, and retention of bioactive compounds produced by piezoelectric atomizer technology. First, green coffee oil emulsions were produced by ultrasonication using two wall material/oil ratios (1 and 3) and two ultrasonication times (5 and 20 min). Dynamic and electrophoretic light scattering were used to measure the size and zeta potential of the emulsions. The emulsions were dried using a piezoelectric atomizer with a 5 μm mesh at 110 and 120 °C. The encapsulation efficiency, drying yield, morphology, and 5-caffeoylquinic acid retention were evaluated. The encapsulation efficiency ranged from 55.7 to 87.5%, while the drying yield varied from 51.7 to 80.8%. According to the SEM images, a low wall-material/oil ratio produced agglomerated particles, while a high ratio produced spherical particles. The retention of the 5-caffeoylquinic acid ranged from 23.0 to 85.5%. Green coffee oil powders could be applied as functional ingredients.

Keywords: *green coffee oil, chlorogenic acid, microcapsules.*

Resumen

La promoción de beneficios para la salud mediante la incorporación de ingredientes bioactivos es un área de investigación activa en el procesado de alimentos. El aceite de café verde extraído con fluidos supercríticos es fuente de ácidos grasos poliinsaturados y ácido clorogénico, un potente antioxidante. Este estudio muestra las características de microcápsulas con aceite de café verde producidos con la tecnología de atomización piezoeléctrico, incluyendo eficiencia de encapsulación, morfología y retención de ácido 5-cafeilquínico. Se produjeron emulsiones de aceite de café verde por ultrasonicación variando el material de pared/aceite (1 y 3) y tiempos de ultrasonido (5 y 20 min). Las emulsiones se secaron utilizando un atomizador piezoeléctrico con malla de 5 μm a 110 y 120 °C. Se evaluaron la eficacia de la encapsulación, rendimiento del secado, morfología y retención del ácido 5-cafeilquínico. La eficacia de encapsulación osciló entre 55.7 y 87.5%, mientras que el rendimiento del secado varió entre 51.7 y 80.8%. Las imágenes SEM mostraron que una proporción baja de material de pared/aceite produjo partículas aglomeradas, mientras que una proporción alta produjo partículas esféricas. La retención del ácido 5-cafeoilquínico osciló entre el 23.0 y el 85.5%. Los polvos de aceite de café verde podrían aplicarse como ingrediente funcional.

Palabras clave: aceite de café verde, ácido clorogénico, microcápsulas.

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1 Introduction

Green coffee oil (GCO) from *Coffea arabica* is an ingredient used in the cosmetic and nutraceutical industries because its fatty acid, triacylglycerol, sterol, and diterpene contents can reduce UV damage to the skin and promote chemoprotective activity (Moeenfard and Alves, 2020; Nosari *et al*., 2015; Wagemaker *et al*., 2012). GCOs are extracted by mechanical pressing, solvent extraction, or supercritical fluid extraction (Barajas-Álvarez *et al*., 2021; Efthymiopoulos *et al*., 2019). However, unsaturated fatty acids in GCOs are susceptible to chemical degradation, producing undesirable odors and metabolites that decrease their biological activities and shelf life (Granados-Vallejo *et al*., 2019; Nallamuthu *et al*., 2015). Recently, Barajas-Álvarez *et al*. (2021) reported that green coffee supercritical extracts contain significant amounts of caffeine and 5-caffeoylquinic acid, which may increase the health benefits. Among the reported multiple health benefits of 5-caffeoylquinic acid are anti-inflammatory, neuroprotective, anticancer, and antioxidant effects (Jeszka-Skowron *et al*., 2016).

Encapsulation technologies improve the stability of oils by allowing them to form shells around oil droplets, which enhances their stability, handling, and incorporation into different nutraceutical products (de Oliveira *et al*., 2020; Granados-Vallejo *et al*., 2019). Spray drying is the most widely used encapsulation technology for producing powdered ingredients. Spray drying involves atomizing a colloidal solution inside a chamber with hot air that instantly dehydrates the droplets to produce particles of different sizes (approximately 200 nm to 250 μm) (Cortés-Rodríguez *et al*., 2022). The feed properties (composition, solid concentration, and viscosity) and spray-drying process (equipment configuration, atomizer selection, and operating conditions) impact the yield, efficiency, size distribution, solubility, flowability, morphology, and stability of the spray-dried powders. In addition, fine droplets (approximately 3 to 15 μm) can be produced through a fine mesh from 4.0 to 7.0 μm using a piezoelectric spray system known as NanoSpray-drier®, which vibrates from 60 to 140 kHz, helping to produce powders of smaller particle size distributions (< 10 μm) than rotary atomizers and pressure nozzles (X. Li *et al*., 2010). The droplet sizes of the sprayed liquid are related to the frequency of vibration, the mesh size, the solution viscosity, and the superficial tension (Piñón-Balderrama *et al*., 2020). Several studies have encapsulated bioactive compounds, including carotenoids, curcumin, rutin, and eugenol, to enhance the stability and biological activity of these plants (Díaz *et al*., 2019; Hu *et al*., 2016; Mahalakshmi *et al*., 2020; Pedrozo *et al*., 2020; Prasad Reddy *et al*., 2019). Encapsulation using the NanoSpray-drier is a recent approach to protect oils, and additional studies are necessary to establish whether tiny powders can preserve the stability of encapsulated oils. In this study microcapsules of green coffee oil produced by spray drying technology were produced and characterized, after which the encapsulation efficiency, drying yield, and chlorogenic acid retention ability of the green coffee oil powders were evaluated.

2 Materials

Green coffee oil (GCO) was extracted from green coffee beans (*Coffee arabica*) by supercritical extraction, as described by Barajas-Álvarez *et al*. (2021). The fatty acid profile of GCO was 47.63% linoleic, 42.24% palmitic, 3.41% stearic, 3.07% oleic, and 1.12% linolenic acid. GCO contained 0.60 mg 5-caffeoylquinic acid/g. Gum arabic (*Acacia senegal*) was purchased from Reasol (CDMX, Mexico). The ethanol, methanol, and phosphoric acid used were HPLC grade. A 5-caffeoylquinic acid standard was purchased from Sigma-Aldrich (State of Mexico, Mexico).

2.1 *Fourier transform infrared spectroscopy (FTIR)*

Fourier transform infrared spectroscopy was used to characterize the GCOs using a Cary 630 FTIR instrument (Agilent Technologies, Santa Clara, CA, USA) equipped with an attenuated total reflectance (ATR). FTIR spectra were obtained in 32 scans from 4000 to 800 cm^{-1} with a resolution of 2 cm^{-1} using the MicroLab FTIR software (Agilent Technologies, Santa Clara, CA, USA).

2.2 *Preparation of GCO emulsions*

Gum arabic teardrops (approximately 25 g) were dispersed in distilled hot water (~90 °C) (approximately 100 g) for 1 h. Centrifugation of the gum arabic dispersion at 4,000 rpm for 20 min helped to remove impurities (Thermo Scientific SL 16, West Columbia, SC, USA). The gum arabic was adjusted to 20 wt%. To guarantee hydration, the solution was refrigerated overnight at 4 °C. A two-step method produced the GCO emulsions. First, a high shear disperser was used to produce a coarse emulsion at 13,500 rpm for 5 min (T25 Ultra-Turrax, IKA-Werke, Staufen, Germany). Two wall-material/GCO mass ratios (1 and 3) were used to encapsulate a constant oil volume fraction (ϕ = 0.20). Then, each emulsion was processed with an FB-50 ultrasonic device with a 1/8' diameter probe (Fisher Scientific, Waltham, MA, USA) for 5 and 25 min. The amplitude of the sonication probe was set at 90%. A constant volume of 20 mL of each emulsion was produced in a 30 mL beaker. The experiments were performed in duplicate. The sample temperature during sonication was preserved below 20 °C using a water-cooled jacket.

2.3 *Droplet size and zeta-potential measurements*

The droplet size, polydispersity index, and zeta-potential of the emulsions were measured using a Zetasizer Nano ZS90 (Malvern Instruments Ltd., Worcestershire, UK) according to the methods described by García-Márquez *et al*. (2017). The emulsions were diluted 100-fold with distilled water before measurement. The droplet size and zeta-potential value were reported as averages of measured three measurements for each sample.

2.4 Emulsion viscosity

The viscosity of the emulsions was determined with a continuous flow ramp using an AR 1000 rheometer (TA Instruments, DE, USA) at 25 °C. A cone geometry of 60 mm in diameter with a 1° of inclination and 29 μm truncation gap was used. The measurements were made with a shear rate from 0.1 to 120 s^{-1}. If the emulsion did not meet the viscosity requirement (≤ 0.010 Pa·s), it was diluted with water and its viscosity was recalculated.

2.5 Spray drying process

The GCO emulsions were spray-dried using a Nano Spray Dryer B-90HP with a spray mesh of 5.5 μm (Büchi Labortechnik, Flawil, Switzerland). The drying inlet temperatures were set at 110 or 120 °C at an airflow rate of 116 L/min. The powders were collected from the electrostatic particle collector using a rubber scraper and stored in a hermetic bag at 4°C.

2.6 Physicochemical characterization

2.6.1 Spray drying yield recovery

The yield of the NanoSpray-drier process was calculated using the following equation (1):

$$\text{yield (\%)} = \frac{\text{mass of collecting particles (g)}}{\text{mass of solids feeded (g)}} \times 100\% \quad (1)$$

2.6.2 Encapsulation efficiency

The encapsulation efficiency (EE) was determined by the fraction of encapsulated oil divided by the total amount of oil multiplied by 100% using equation (2):

$$\text{EE (\%)} = \frac{\text{Total oil (g)} - \text{Surface oil (g)}}{\text{Total oil (g)}} \times 100\% \quad (2)$$

The surface oil content was calculated according to the method described by Silva *et al*. (2014).

2.6.3 Morphology of spray-dried particles

The surface morphology of the GCO particles was examined using a scanning electronic microscopy (SEM, Mira3 LMU, Tescan, Brno-Kohoutovice, Czech Republic). Before imaging, the samples were coated with a gold layer by vacuum sputtering (SPI Module Sputter Coater, West Chester, PA, USA).

2.6.4 Chlorogenic acid concentration

CGA was estimated by HPLC (2998, Waters Corp., Milford, MA, USA) according to the procedure reported by Ruiz-Palomino *et al*. (2019). The separation was performed using a Kromasil-C18 column (250 × 4.6 mm, 5 μm; AkzoNobel, Bohus, Sweden) maintained at 25°C. Mobile phase A was 5 mM phosphoric acid, and mobile phase B was methanol at a flow rate of 1 mL min^{-1}. The gradient was initially set at an A/B ratio of 85/15 from 0 to 5 min; then 80/20 from 6 to 10 min; 60/40 from 11 to 20 min; 70/30 from 21 to 25; 80/20 from 26 to 30; and finally, 85/15 from 31 to 35 min. Absorbance was measured at 325 nm. The injection volume was 20 μL. A standard curve of 5-caffeoylquinic acid was used to quantify the chlorogenic acid concentration in the sample. The samples and standards were centrifugated at 10,000 rpm for 10 min. The supernatants were filtered in two steps, first, using a Sep-Pak C18 cartridge (55-105 μm, Waters Corp., Milford, MA, USA) and, second, using a 0.45 μm syringe filter (Millipore, Burlington, MA, USA). The CGA concentration was calculated as the retention of the particles over the initial CGA concentration in the oil (equation 3).

$$\text{CGA retention (\%)} = \frac{\text{GCA concentration in powder}}{\text{GCA concentration in oil}} \times 100\% \quad (3)$$

2.7 Statistical analysis

Analysis of variance (ANOVA) was performed to determine the differences between treatment means according to Tukey's test ($p < 0.05$) using Statgraphics Centurion XVII software (version 17.0.16 Statpoint Technologies, Inc., Warrenton, VA, USA). The data are expressed as the means ± standard deviations.

3 Results and discussion

3.1 Green coffee oil characterization

The FTIR spectrum of GCO showed several bands characteristic of edible oils, caffeine, and chlorogenic acids (Figure 1). The absorption band observed at 3008 cm^{-1} corresponds to the C-H stretching vibration of the *cis*-double bonds (HC=CH) of polyunsaturated fatty acids (Guillén and Cabo, 1997; Raba *et al*., 2015). The methyl and methylene groups of the aliphatic chain C-H bonds were observed at 2920 cm^{-1} and 2851 cm^{-1} bands, respectively, and are typically associated with lipids and caffeine in coffee extracts (Topala and Tataru, 2015). The band found at 1741 cm^{-1} is related to the carbonyl vibration (C=O) of aliphatic esters in triglycerides, a typical band for lipids. An unsaturated fatty bond (C=C) was observed at 1666 cm^{-1}. According to Guillén and Cabo (1997), this band was attributed to the disubstituted *cis* C=C of the acyl groups of oleic and linoleic acids. The bands corresponding to the aliphatic groups (CH_2 and CH_3) appeared at 1461 cm^{-1} and 1375 cm^{-1}, respectively (Guillén *et al*., 2003). The caffeic and chlorogenic acid bands were found at 1236, 1159, and 1045 cm^{-1}. Topala and Tataru (2015) observed FTIR spectra of green coffee oil with and without caffeine, identifying several bands associated with caffeine at 2920, 2851, 1650, 1153, and 716 cm^{-1}.

3.2 Emulsion characterization

Gum arabic (*Acacia senegal*) is a natural emulsifier, stabilizing, and film-forming agent (Loan *et al*., 2023). The droplet size of the emulsions depends on the energy supplied during homogenization and the properties of the emulsifier agent. In general, if the gum arabic/GCO ratio and sonication time increased, the droplet size of the emulsions decreased

Table 1. Physicochemical properties of the emulsions at different sonication times and gum arabic/green coffee oil ratios.

Code	Biopolymer/oil ratio	Sonication time (min)	Mean droplet size (nm)	*PI*	Zeta potential (mV)
F1	1	5	595.1 ± 16.7^{ab}	0.23 ± 0.02^{a}	-44.2 ± 1.6^{a}
F2	1	25	604.2 ± 8.7^{ab}	0.13 ± 0.04^{bc}	-42.4 ± 2.1^{a}
F3	3	5	653.9 ± 10.2^{b}	0.18 ± 0.05^{ab}	-37.6 ± 1.3^{b}
F4	3	25	554.6 ± 9.1^{a}	0.11 ± 0.03^{c}	-40.3 ± 1.0^{ab}

Tukey's test indicates significant differences between samples indicated by lowercase letters ($p < 0.05$).

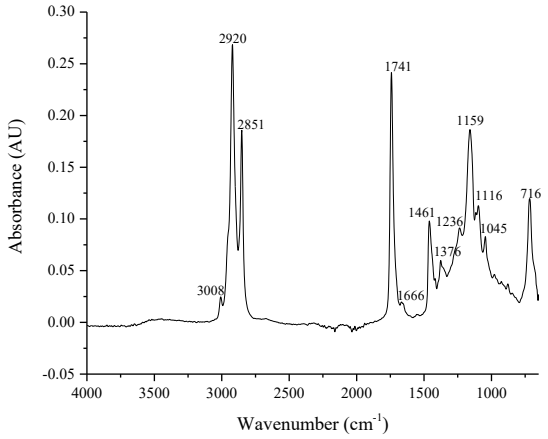

Figure 1: FTIR spectrum of GCO.

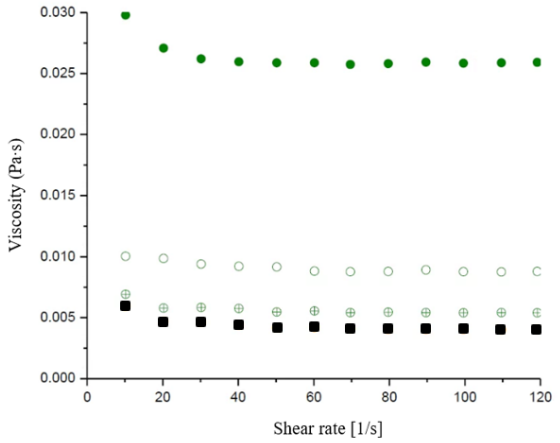

Figure 2: Viscosity of green coffee oil emulsions. F3 emulsion (Black squares), F4 emulsion (green filled circle), diluted F4 emulsion (1:1) (green circle) and diluted F4 emulsion (2:1) (green circle with plus).

(Table 1). No differences were observed between samples produced with a low gum arabic/GCO ratio and those produced with different sonication times. As expected, long sonication times and a high gum arabic/GCO ratio reduced the average particle size and *PI* values. According to Bhattacharjee (2016), *PI* values describe the distribution of emulsion droplets and can be used to describe emulsion the quality of an emulsion. If the *PI* is less than 0.10, the emulsion is considered highly monodisperse; if the *PI* is between 0.10 and 0.40, the emulsion is moderately polydisperse; and if the *PI* is greater than 0.40, the emulsion is highly polydisperse. The *PI* values were less than 0.3, indicating moderately polydisperse emulsions. Espinosa-Andrews and Páez-Hernández (2020) reported that prolonged sonication times could reduce the *PI* value of an emulsion, resulting in emulsions with increased stability.

In addition, the zeta-potentials of the emulsions reached -37 mV. The increased zeta-potential values suggest the formation of electrically stable systems, avoiding processes such as aggregation, flocculation, and sedimentation (Li *et al*., 2020). According to the DLVO model, a positive primary maximum to avoid an irreversible flocculation energy barrier should be >1.5kT (>37.5 mV) at room temperature, preventing interparticle contact and producing a metastable system (Jayme *et al*., 1999). Based on these findings, F2 and F4 emulsions were selected to produce powders due to their smaller particle size and lower *PI* values.

3.3 Nanospray drying yield

Both emulsions were pumped to the piezoelectric atomizing nozzle of the Nano Spray Dryer B-90 to produce fine powders. However, sample F4 could not be sprayed by the piezoelectric nozzle because its high solids content increased the emulsion viscosity to approximately 25 cP (Figure 2), reducing its ability to be sprayed through the piezoelectric atomizing nozzle. Wong and John (2016) reported that the piezoelectric atomizing nozzle may be able to spray liquids with low solids content or low viscosity, *i.e.*, the feed viscosity of the liquid should be lower than 10 cP. According to this, sample F4 was diluted (one part of emulsion with two parts of water) until approximately 5 cP before drying to ensure to be below the limits set by the nanospray dryer. This condition did not change the droplet size or the zeta potential of the emulsion droplets.

The yield of the nanospray-dried samples ranged between 51.7% and 80.8%. Figure 3a shows that the drying yield depends on the temperature. The drying yields values were low at a temperature of 110 °C, but increasing the temperature increased the drying yield. The highest yield values were observed at an inlet temperature of 120 °C. No significant differences ($p < 0.05$) were detected in samples with different gum arabic/green coffee oil ratios. The dying yield values are similar to those reported by Li *et al*. (2010) for spray-dried gum arabic powder. When the microdroplets come into contact with hot air, they are instantly dehydrated, and an inadequate temperature cannot remove the water from the surface of the droplets, impacting the spraying yield. Desai *et al*. (2020) reported that an aqueous extract of green coffee encapsulated with maltodextrin produced a high yield upon spray drying using a polymer:extract ratio of 2:1 at a temperature of 125°C. According to Gu *et al*. (2015), an increase in the inlet temperature positively impacts the spray drying yield. The encapsulation efficiency ranged from

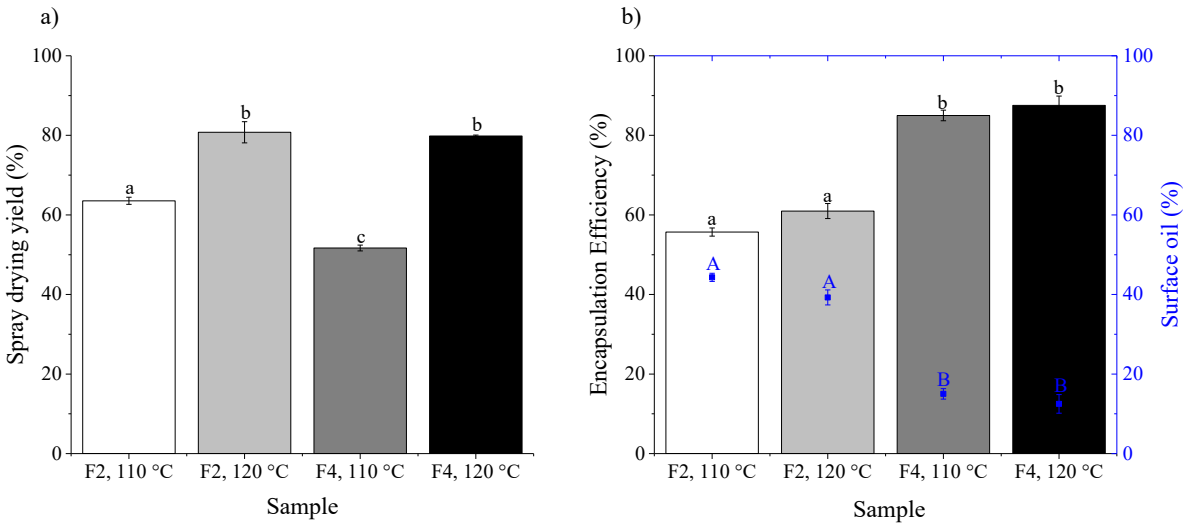

Figure 3: Physicochemical properties of the green coffee oil microparticles: a) spray drying yield recovery and b) encapsulation efficiency and surface oil content. Different lowercase letters indicate significant differences according to Tukey's test at $p < 0.05$.

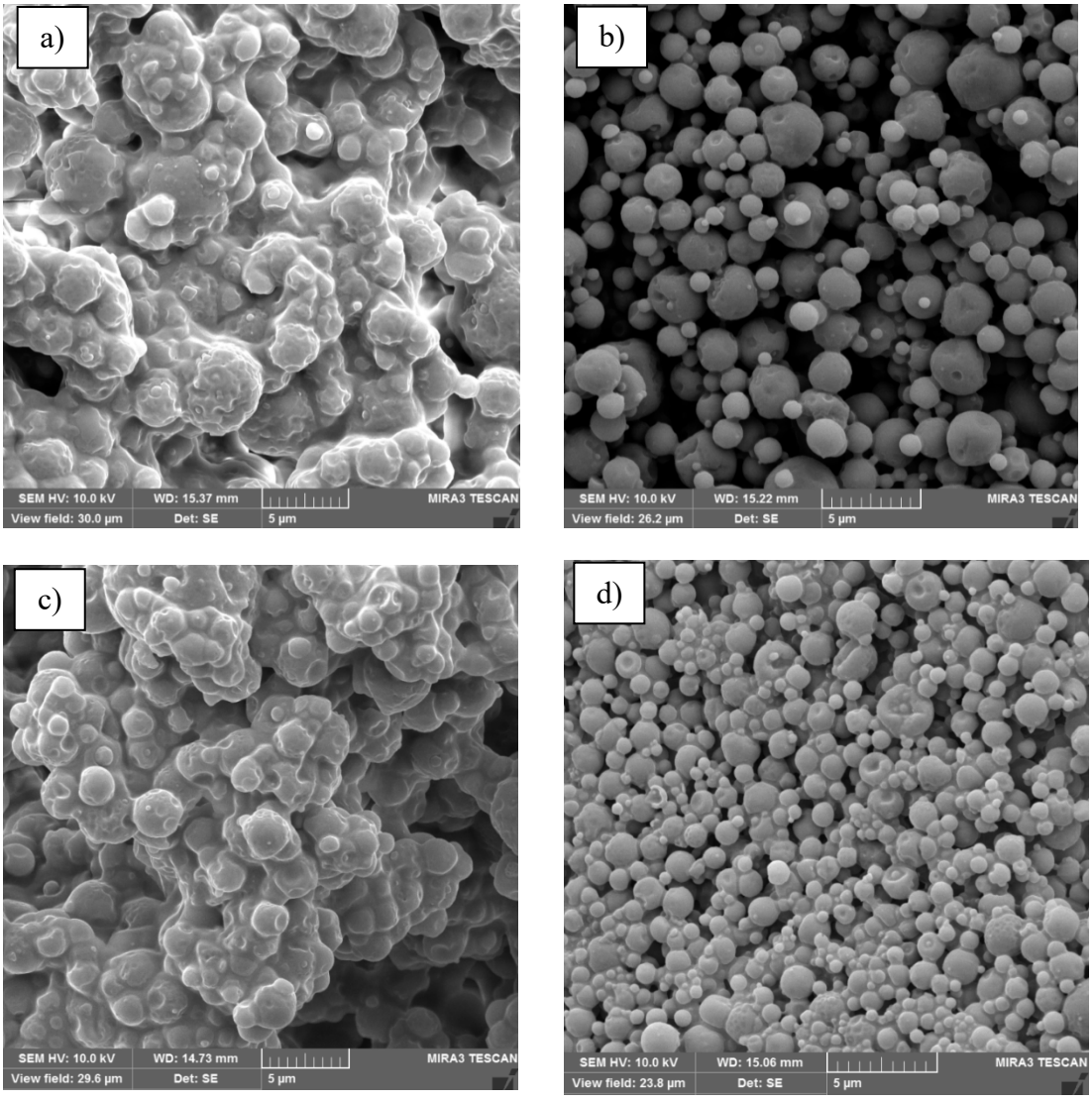

Figure 4: Micrographics of green coffee oil microparticles obtained by nanospray drying: a) F2, 110 °C, b) F4, 110 °C, c) F2, 120 °C and d) F4, 120 °C.

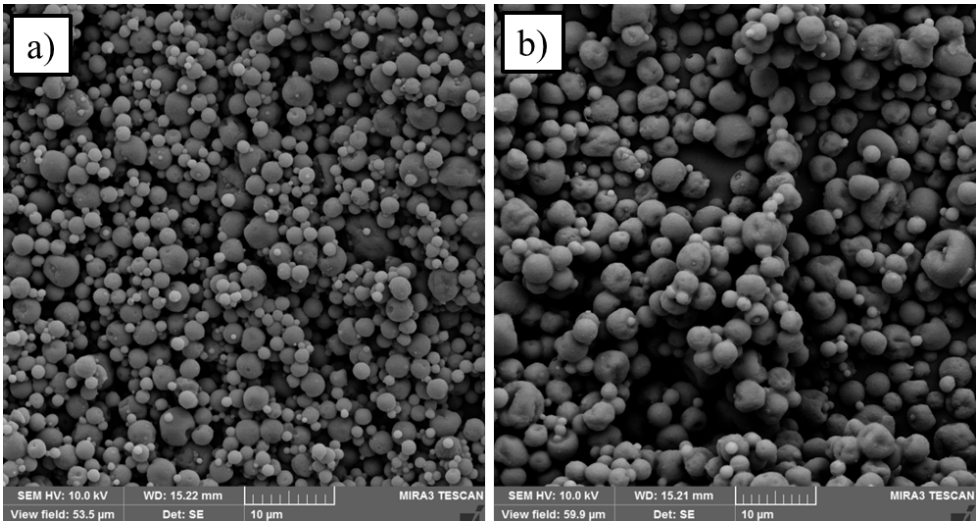

Figure 5. Micrographics of green coffee oil microparticles obtained by nanospray drying using two spray meshes: a) F2, 120 °C and 5.5 μm mesh and b) F2, 120 °C and 7 μm mesh.

55.7% to 87.5% (Figure 3b). The encapsulation efficiency increased with increasing solid concentration in the emulsions, *i.e.*, the highest encapsulation efficiency was obtained using a gum arabic/green coffee oil ratio equal to 3. The presence of a higher concentration of wall material enhanced the encapsulation efficiency (Gharsallaoui *et al*., 2007). Frascareli *et al*. (2012) reported that as the concentration of gum arabic on the surface of the oil droplets increased, the encapsulation efficiency of coffee oil increased, offering a protective structural matrix. Microcapsules F1 and F2 (with a biopolymer/oil ratio of 1) had more surface oil than did F3 and F4 (with a biopolymer/oil ratio of 3), resulting in lower encapsulation efficiency. The surface oil content is a critical parameter related to oil oxidation. When the surface oil is exposed to oxygen, the oxidation rate could increase (Granados-Vallejo *et al*., 2019; Silva *et al*., 2014).

3.4 Microcapsule morphology

The removal of moisture from spray droplets is associated with the morphology of the final product (Figure 4). Samples with a low wall material ratio showed agglomerated particles recovered from presenting an oily appearance, regardless of the drying temperature (Figures 4a and 4c). This behavior explains the results obtained for the encapsulation efficiency and for the surface oil outside the microparticles. The results showed that a gum arabic/GCO ratio of 1 was inadequate for achieving a total GCO coating during drying. Wang *et al*. (2016) observed that particles containing 40% oil were agglomerated particles and sticky due to droplet fragmentation during the spray drying process.

In contrast, samples with a high wall material/oil ratio exhibited spherical shapes with no cracks or pores on their surface. The presence of discrete individual particles suggested that the GCO oil was encapsulated, and some particles had dents caused by a few bumps between them. This type of morphology reduces gas permeability, improving oil protection (de Barros Fernandes *et al*., 2016). The particle size obtained (< 2 μm) was smaller than the powder obtained by conventional spray dryers (from 50 to 400 μm) (Granados-Vallejo *et al*., 2019; Hu *et al*., 2016).

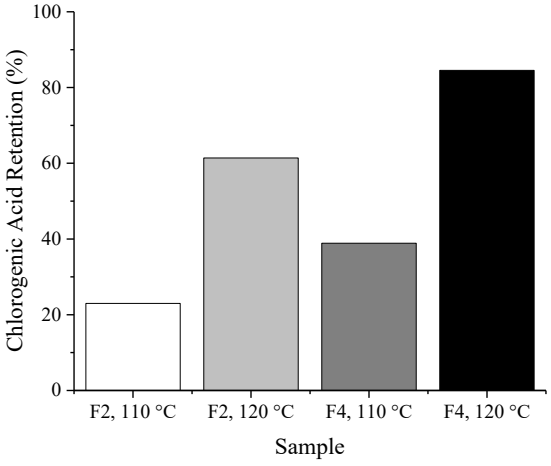

Figure 6: Chlorogenic acid retention in the green coffee oil microparticles.

The pore size of the spray mesh changes the particle size distribution (Lee *et al*., 2011). Figure 5 shows microphotographs of GCO microcapsules produced with two different nanospray meshes (5.5 and 7.0 μm) at 120°C. The mesh size positively influenced the particle size, resulting in spherical shapes without fissures or pores. Particles ranging from 500 nm to 2 μm in size were obtained using a 5.5 μm spray mesh size, whereas a 7 μm mesh size produced particles between 2 and 5 μm in size. Gu *et al*. (2015) and Wang *et al*. (2016) reported that particles ranging from 2 to 10 μm were obtained with 5.5 or 7.0 μm meshes.

3.5 Evaluation of chlorogenic acid retention

According to Mills *et al*. (2013), the concentration of CGA can be reduced by processing stages of coffee products, such as fermentation, roasting, freeze-drying, or spray-drying. The concentration of the CGA supercritical extract was 0.597 mg/g GCO. The concentration of CGA in the particles

decreased by 23.0% to 85.5% after spray drying (Figure 6). The amount of CGA was high when the wall material/oil ratio was 3 and the drying temperature was 120 °C. Desai *et al*. (2020) reported that increasing the inlet temperature from 100 to 125 °C and adding a high amount of wall material resulted in more phenolic compounds being found in the powders. Pettinato *et al*. 2017) reported a 30% reduction in phenols in the aqueous extract of spent ground coffee after spray drying.

Conclusions

Piezoelectric atomizer technology (NanoSpray drier) produced microcapsules of green coffee oil enriched with chlorogenic acids. Gum arabic acted as an excellent emulsifier and stabilizer of green coffee oil. A high gum arabic/green coffee oil ratio and extended sonication time reduced the droplet size and polydispersity index of the emulsions. The zeta potential of the emulsions suggested that high colloidal stability was favored by repulsion energy. Different drying temperatures and emulsion formulations influenced the spray drying yield, encapsulation efficiency, morphology, and retention of chlorogenic acids in the microparticles. A high wall-material/oil ratio was required to enhance the complete encapsulation of green coffee oil and prevent the degradation of antioxidant compounds. Nanospray drying technology allowed us to obtain microparticles that contained a high amount of green coffee oil enriched with chlorogenic acids. Green coffee oil microparticles can be potentially incorporated into foods or cosmetic products.

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