Food Research 8 (4) : 206 - 216 (August 2024)
Journal homepage: https://www.myfoodresearch.com

FOOD RESEARCH

RESEARCH PAPER

Chia seed oil microencapsulated by spray-drying: optimization and microcapsules characteristics

[1]Cuevas-Glory, L., [1]Rosel-Sosa, D.G., [2,3]Ramírez-Ortiz, M.E., [1]Gallegos-Tintoré, S., [4]Chim-Chi, Y.A., [2]Chel-Guerrero, L. and [2,*]Betancur-Ancona, D.

[1]Instituto Tecnológico de Mérida. Av. Tecnológico s/n, Mérida, Yucatán, México
[2]Universidad Autónoma de Yucatán, Facultad de Ingeniería Química. Campus de Ciencias Exactas e Ingenierías, Periférico Norte Km 33.5, Chuburná de Hidalgo Inn. Mérida, Yucatán, México
[3]Universidad Nacional Autónoma de México, Facultad de Estudios Superiores Cuautitlán, Departamento de Ingeniería y Tecnología, Av. 1° de Mayo S/N, Sta. María las Torres, Cuautitlán Izcalli, Edo. de México, México
[4]Tecnológico Nacional de México. Instituto Tecnológico Superior de Calkiní, Av. Ah-Canul, C.P. 24900 Calkiní, Campeche, México

Article history:
Received: 12 August 2022
Received in revised form: 21 September 2022
Accepted: 3 October 2023
Available Online: 28 July 2024

Keywords:
Chia seed oil,
Microencapsulation,
Omega 3-fatty acid,
Spray drying

DOI:
https://doi.org/10.26656/fr.2017.8(4).428

Abstract

A current trend in food research is the development of beneficial health products that could help to decrease certain risks of diseases. The chia seed (*Salvia hispanica* L.) oil is a proven source of essential fatty acids (omega-3), which can help to prevent cardiovascular diseases. In this study, chia seed oil was characterized and fatty acids were identified before and after the spray-drying process. A 3^2 factorial design with response surface methodology was utilized for chia seed oil spray drying experiments. Statistic design factors and levels were as follows: gum arabic: maltodextrin mixture (GA:MD) (1:1, 1:2 and 1:3 w/w) and drying temperatures (DT) (140, 160, and 180°C). A total of 67.9% of unsaturated fatty acids were found in raw chia seed oil and 65.7% in microencapsulated oil. The optimal parameters for the spray drying process were as follows: GA:MD 51:1 and DT, 180°C; powder yield rate, 49.5% (w/w), mean size of particles 129 µm, and sphericity coefficient of 0.11. Microcapsules tended to demean over time, changing their color, and producing agglomerate, which decreased their flowing capacity. On the other hand, the product was easier to handle, the oil was protected from heat and moisture conditions, and the microencapsulation process provided stability to the oil's active substances, such as omega-type fatty acids.

1. Introduction

Chia plant (*Salvia hispanica* L.) is native to México and has become popular in recent years due to the health benefits of consuming its seeds. Chia seeds contain vitamin B, calcium, potassium, zinc, and copper; it's rich in fiber and proteins, and no antinutrients have been detected (Capitani *et al.,* 2012). The three most significant components of chia seeds are oil, fiber, and antioxidant compounds. Chia seeds provide health benefits to the human body, like cholesterolemia reduction, modification to glycemic and insulinemic responses, and changes in intestinal function; it also has been reported its antioxidant capacity, which helps to delay cell degradation (Capitani *et al.,* 2012).

The essential fatty acid content in chia seed oil makes it a good edible oil alternative for commercial production. Oil content in chia seeds ranges from 32 to 39%, where the most significant constituents are triglycerides and polyunsaturated fatty acids (PUFAs) (Ayerza, 2011). Usually, α-linolenic (omega-3) and linoleic (omega-6) fatty acids account for 60% and 20%, respectively, of the total, with small amounts of palmitic and stearic fatty acids (Mosquera-Quelal *et al.,* 2017). Omega-3 fatty acids play an essential role in human physiology, especially during fetal and infant growth and for the prevention of cardiovascular diseases. These fatty acids also act as antithrombotic, anti-inflammatory, and antiarrhythmic agents, favoring plaque stabilization (Da Silva-Marineli *et al.,* 2014). However, successful incorporation of n-3 PUFAs in processed foods is limited due to their low solubility in most food matrices and high susceptibility to oxidation (Aghbashlo *et al.,* 2013). The oxidative degradation chain reaction results in a loss of

*Corresponding author.
Email: *bancona@correo.uady.mx*

eISSN: 2550-2166
Published by Rynnye Lyan Resources

nutritional quality and the development of undesired flavors due to reaction products, affecting the shelf stability and sensory properties of the oil (Berto *et al*., 2020). Microencapsulation is a good alternative to preserve the beneficial properties of highly unsaturated oils keeping their properties from environmental factors. Capsules are characterized by size (less than 1 mm) and are used to protect biologically active substances from environmental factors, such as pH, temperature, salts and oxidation. Also, they are used as a way of separating, packaging, and storing micro-scaled products; they have the advantage of releasing the encapsulated material under controlled conditions (Zhang *et al.,* 2014). Wall material choice for spray-drying microencapsulation is crucial for efficiency and stability. The criteria for the selection of wall material are mainly based on physicochemical properties such as solubility, molecular weight, glass/melting transition, crystallinity, diffusibility, film forming, and emulsifying properties. Wall materials such as proteins, carbohydrates, lipids, gums, polysaccharides, and cellulose materials are commonly utilized for microencapsulating omega-3 fatty acids and oils. These materials can be used pure or combined to achieve certain functions, either during the process or in the final product. Hydrolyzed starch, maltodextrin, is commonly used as wall material in microencapsulating food ingredients; it provides good oxidative stability to the encapsulated oil but exhibits poor emulsifying capacity, emulsion stability, and low oil retention (Gharsallaoui *et al.,* 2007). However, combined with other encapsulating materials such as proteins or gums, the stability of the microencapsulated oils improves. Few studies have been undertaken to encapsulate chia seed oil using the spray drying process with different wall materials. Ixtaina *et al.* (2015) conducted research to determine the influence of operating conditions (the homogenization pressure for emulsifying and the spray-drying inlet/outlet temperatures) over the physicochemical properties of chia seed oil microencapsulated with sodium caseinate and lactose by spray-drying. Martínez *et al.* (2015), evaluated the use of maltodextrin in combination with hydroxypropyl methylcellulose as the microcapsule wall materials, as well as the effect of rosemary extract on the oxidative stability of the microcapsules. Finally, other authors (Fernandes *et al.,* 2021) found that the best conditions for microencapsulating chia seed oil by spray drying were at a temperature of 120°C and a feed rate of 0.1L/h, keeping the maltodextrin and gum arabic ratio constant, as well as all other parameters. Obtaining a yield of 50% and microcapsules of 3.01 um in diameter. This work aims to establish the influence of the inlet air temperature and the proportion of gum arabic: maltodextrin on the encapsulation process of chia seed oil by spray drying, and analyzing data using a response surface methodology.

2. Materials and methods

2.1 Materials

Chia seeds were obtained from the local market (San Benito) in Merida, Yucatan, Mexico. The chemicals (reagent grade) were purchased from J.T. Baker (Phillipsburg, NJ, USA) and Sigma (St. Louis, MO, USA). Gum arabic was purchased from Industria Ragar, S.A. de C.V. (Mexico City, Mexico), and maltodextrin (DE 10) was obtained from IMSA (Guadalajara, Mexico).

2.2 Extraction of chia seed oil by using screw press

Raw chia seed oil was obtained by pressing (Screw press, Monforts WDV 55/13 D101LA4, Germany) 6.97 kg of chia seeds following the method proposed by Ixtaina *et al.* (2011). The range of temperature utilized varied from 25 – 30°C, and a 5 mm restriction die and a screw press speed of 20 rpm, were utilized. The chia seed oil was centrifuged at 5,000 rpm (Thermo Scientific Megaguge 16R spin dryer, USA) for 25 mins at 10°C to remove suspended oil residues. Oil yield was determined gravimetrically and was expressed as a percentage on a dry basis (g oil/100 g chia seed). The obtained clean oil was stored under a nitrogen atmosphere, in hermetically sealed amber containers at 4°C for its preservation.

2.3 Chia seed oil analysis

The physicochemical properties of chia seed oil were determined by using official methods and recommended practices by the Mexican Official Standards (NMX), among others. Refractive index, relative density, and color were determined by following the methods described by NMX-F-75-2012, and NMX-F-116-2012, respectively. Acidity, saponification index, iodine, and peroxides index were evaluated by the following norms: NMX-F-101-1987, NMX-F-174-S-1981, NMX-F-152-S-2011, NMX-F-154-2010, respectively.

2.4 Experimental design for response surface methodology

The conditions of the spray-drying process were optimized by using RSM. A 3^2 factorial design including three replicates of the center point was performed. A total of 12 experimental runs were carried out (Montgomery, 2013). Experiments were randomized to minimize the effects of unexplained variability in the actual responses due to other factors. Factors and levels utilized were as follows: X1, gum arabic: maltodextrin ratio (1:1, 1:2 y 1:3 w/w), and X2, drying temperatures (DT) (140, 160 and 180°C). The oil encapsulation yield

https://doi.org/10.26656/fr.2017.8(4).428

(Y1) and the efficiency of oil encapsulation (Y2) were considered as the response variables.

Experimental data were fitted to a second-order polynomial model. The generalized second-order polynomial model used in the response surface analysis was as follows:

$$Y = \beta_0 + \sum_{j=1}^{k} \beta_j X_j + \sum_{j=1}^{k} \beta_{jj} X_j^2 + \sum \sum_{i<j} \beta_{ij} X_i X_j$$

Where Y is the response, β_0 is the constant coefficient, β_j is the linear coefficient, β_{ij} is the interaction coefficient, β_{jj} is the quadratic coefficient and Xi and Xj are the coded values of independent variables and k represents the number of variables (k = 2). The fitting of the model to the experimental data was given by the coefficient of determination (R^2), which accounts for the degree of variance of a modeled variable that can be explained by the model.

The variance analysis, the lack of fit test, regression coefficients, and the three-dimensional graphs were carried out by using Statgraphics Plus statistical package software (Statgraphics Plus 5.1, Manugistics Inc., USA).

2.5 Preparation of emulsions

Oil emulsion was obtained by blending (Ultra-Turrax T18, IKA, Werke GmbH, Staufen, Germany) a mixture of gum arabic and maltodextrin in water (encapsulating agent) and chia seed oil (30%) at 10,000 rpm for 60 s (Ixtaina *et al.,* 2015).

2.6 Spray drying

Spray drying was performed in a laboratory-scale spray dryer (LabPlant SD-05, Huddersfield, England). The feed flow rate was 1.5 mL/min keeping feed pressure constant (1 bar). After equipment stabilization, the oil emulsion (30 mL) plus 100 mL of mixed gum were fed by using a peristaltic pump to the injector nozzle (0.5 mm). Dry powder samples were recovered from the collecting device, then packed in polythene bags and stored in a desiccator at 20°C for subsequent characterization.

2.7 Oil encapsulation yield

Oil Encapsulation Yield (OEY) was determined by Soxhlet extraction procedure for 4 hrs using hexane as extractive solvent (200 mL). After extraction, the percentage of oil contained in the microcapsules was gravimetrically quantified (Equation 1) as follows:

$$\text{OEY}\,(\%) = \frac{\text{Encapsulated oil weight}}{\textit{Weight of the capsules}} \times 100 \quad (1)$$

2.8 Efficiency of oil encapsulation

The efficiency of oil encapsulation (EOE) (Equation 2) was determined according to Aberkane *et al.* (2014). Superficial oil was determined after stirring, for 2 mins at 25°C, a mix of 1 g of the powder in 20 mL of hexane. The formed suspension was slowly filtered through a Whatman filter paper n°1 and the solvent was evaporated in a rotary evaporator. Total oil was assumed to be equal to the initial oil. Preliminary tests revealed that all initial oil was retained. EOE (%) was calculated from Equation 2.

$$\text{EOE}(\%) = \frac{\text{Total oil} - \text{Superficial oil}}{\text{Total oil}} \times 100 \quad (2)$$

2.9 Characterization of microcapsules

Morphology: a configured microscope (Leica, 020-518.500 DM/LS, Germany) with magnifying lenses of 10× and 40× was utilized to determine the size and form of the microcapsules. The sphericity coefficient (Sc) of microcapsules from the best treatment was obtained according to (Lee *et al.,* 2013) equation 3, measuring the diameters of at least 30 particles.

$$\text{Sphericity coefficient} = (\text{Dmax-Dmin})/(\text{Dmax+Dmin}) \quad (3)$$

Where Dmax is the maximum diameter and Dmin is the minimum diameter

Superficial morphology: to determine microcapsules' superficial morphology, some samples of the powders from optimal treatment at different storage times (0, 4, 8, and 12 months) were covered with a thin gold coating and analyzed by using an electronic microscope (JEOL, JSM–6390LV, Japan). Voltage and magnification were 15 KV and 2000×, respectively (Romani *et al.,* 2017).

Flow capacity: powder flow capacity was determined by measuring the angle of repose following the method described by Swarbrick (1997). Chia seed oil powder (20 g) was passed through a horizontal funnel to form a mound of particles. Height (h) and diameter were measured by using a Vernier caliper. The radius (r) of the cone base was calculated (Equation 4) to determine the angle of repose (φ).

$$\varphi = \frac{1}{\tan}\left(\frac{h}{r}\right) \quad (4)$$

Moisture and water activity: moisture content was determined according to the AOAC 925.08 method (Association of the Official Analytical Collabortion (AOAC) International, 1997). Water activity (Aqualab 4TEV, Farmingdale, USA) of the microcapsules was measured at 25°C. The equipment calibration was performed by using LiCl (0.250 Aw) as the standard

https://doi.org/10.26656/fr.2017.8(4).428

compound.

Color: color differences (ΔE*) of powders from different storage times (0, 4, 8 and 12 months) were determined by using a Photoelectric colorimeter (Chroma-meter, Minolta, Tokyo, Japan) by following the CIELab Hunter classification system: L (white = 100, black = 0), a (red = positive, green = negative) and b (yellow = positive, blue = negative), and Equation 5.

$$\Delta E = \sqrt{((\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2)} \quad (5)$$

2.10 Composition of the fatty acids in chia seed oil

The fatty acids profile of Chia seed oil was obtained by gas chromatography (Ixtaina *et al.,* 2011). The fatty acids were chemically transformed into fatty acid methyl esters as follows: chia seed oil (100 mg) was mixed with 1 mL of KOH (0.5 N) in methanol and 1 mL of methanolic boron trifluoride. The obtained mixture was stirred and heated at 100°C in a convection oven (Thermo Scientific, Iowa, USA) for an hour and then cooled to room temperature. Sodium chloride solution (2 mL, 10% v/v) and hexane (2 mL, chromatographic grade) were then added to the mixture. Finally, the mixture was stirred vigorously and kept still until the phases separated. The upper phase was transferred to an amber vial and stored at freezing temperature. The analysis was performed on a gas chromatograph (Perkin Elmer XL, USA) configured with a flame ionization detector (FID) and an AT-5 (Alltech Associates, Australia) gas chromatograph column (30 m, 0.25 mm ID, and 0.25 µm thick). The Injector port and FID Temperatures were 260°C and 270°C, respectively. The carrier gas (N_2) flow rate was 2 mL/min. The oven program was as follows 100°C to 180°C at 5°C/min and then to 220°C at 0.8°C/min. Identification of the fatty acid compounds was made by comparing the retention time of each fatty acid with the retention time of the corresponding standard (FAME MIX 37, Supelco). The results were expressed as g of fatty acids in 100 g of chia seed oil.

3. Results and discussion

3.1 Chia seed oil extraction yield

A total of 1.62 kg of oil was obtained from 6.97 kg (d.b) of chia seed oil seed processed in a screw press. The yield of chia seed oil extraction (23.24%) was similar to that reported by Ixtaina *et al.* (2011) in chia seeds from Argentina (24.8%) and Guatemala (20.3%), under similar extraction conditions. On the other hand, Álvarez-Chávez *et al.* (2008) have reported higher extraction yields in chia seeds cultivated in Jalisco (29.7%) and Sinaloa (25.5%), as well as Segura-Campos *et al.* (2014) in chia seeds from Yucatan, Mexico (27.3%) by solvent extraction. Although solvent extraction is more efficient than mechanical extraction, it's not recommended as chia seed oil is utilized mainly for human consumption.

3.2 Physicochemical characterization of chia seed oil

Chia seed oil refractive index (1.48±0.001) was similar to those reported by Ixtaina *et al.* (2011), under the same extraction conditions by pressing (chia seed oil from Argentina: 1.4794; chia seed oil from Guatemala: 1.4794). This index was also similar to that reported by Segura-Campos *et al*. (2014) in chia seed oil obtained by solvent extraction (1.4761).

Chia seed oil relative density (0.92) was comparable (0.9241) to the one reported by Segura-Campos *et al.* (2014) in chia seed oil obtained by using solvent extraction. Regarding CIELAB parameters, such as color, lightness (L*, 58.45±0.01), green-red hue (a*, 0.897±0.01) and blue-yellow hue (b*, 59.08±0.02) of chia seed oil, the values obtained in this work were higher than those reported by Ixtaina *et al.* (2011) in chia seed oil from Argentina (L*,42.85, a*, -3.75 and b*, 25.90) and Guatemala (L*,39.72, a*, -2.07 and b*, 23.82). A higher value of L* reflects more brightness, while positive values of b* parameter are characteristic of yellow colors. The yellow-colored chia seed oil samples were located near the b* axis in the second quadrant (negative values of a* and positive values of b*). This allows us to consider that chia seed oil from Yucatan, Mexico, is brighter and yellower.

The chia seed oil showed a higher acidity index ((1.3±0.01 mg KOH/g oil) than those (0.91 and 0.70 mg KOH/g oil) obtained by Ixtaina *et al.* (2011). This may be due to the higher moisture content in chia seeds processed by Ixtaina *et al.* (2011) (10% compared to 6.59% obtained in the present study); moisture content increases seed plasticity and lubricating effect in the press barrel, causing slight hydrolysis.

On the other hand, high acidity values have also been reported for chia seed oil (1.64-2.05 mg KOH/g oil) obtained by solvent extraction (Ixtaina *et al.,* 2010; Ixtaina *et al.,* 2011; Segura-Campos *et al.,* 2014). Higher chemical hydrolysis may result in higher acidity due to the solvent and long extraction times. These results are similar to those reported by other authors (Brevedan *et al.,* 2000; Lafont *et al.,* 2011), who found that oils obtained by solvent extraction (hexane), presented higher acidity than oils obtained by pressing. Lower amounts (0.81 and 0.13±0.003 mg KOH/g oil) have also been reported in chia seed oil obtained by supercritical CO_2

https://doi.org/10.26656/fr.2017.8(4).428

extraction (Ixtaina *et al*. 2010). The lower acidity is due to the higher selectivity of the supercritical fluid to extract a lower amount of free fatty acids (Gómez and de la Ossa, 2002).

The saponification index of chia seed oil found in this work (198.3±2.09 mg KOH/g oil) was similar (193.12 and 192.99 mg KOH/g oil) to that reported for chia seed oil obtained by the pressing process (Ixtaina *et al.,* 2011). Other authors described similar results by solvent extraction (193.01-193.6 mg KOH/g oil) and supercritical CO_2 extraction (192.5 mg KOH/g oil) (Ixtaina *et al.* 2010, Ixtaina *et al.,* 2011). However, higher content has been reported by using hexane extraction (222.66 mg KOH/g oil) (Segura-Campos *et al.,* 2014) in chia seed oil from Yucatan, Mexico. This saponification value varies in reverse to the length of fatty acids, which suggests that the chia seed oil evaluated in this work contained more fatty acids of long -chain than the chia seed oil analyzed by Segura-Campos *et al.* (2014).

The iodine value of chia seed oil (107.22 g I_2/100 g oil) determined in the present study was lower than that obtained in chia seed oil from Argentina (208.50/100 g oil) and Guatemala (209.4 g I_2/100 g oil). The high values of the iodine index in chia seed oil are due to the high numbers of double bonds in the length of the fatty acids chain.

No peroxide value was detected in the samples analyzed in this work. Segura-Campos *et al.* (2014) reported 17.5 mEq peroxide/kg oil and Ixtaina *et al.* (2015) 2.0 mEq peroxide/kg oil. This parameter indicates oxidative rancidity. Storage conditions utilized in this work (atmosphere of N_2 at 4°C in the dark) could have favored the chia seed oil keeping free of peroxides. It is important to know the peroxide value, since the oxidative deterioration of n-3 fatty acids may occur, leading especially to hydroperoxides, the main product of lipid oxidation; which are considered toxic.

3.3 Microencapsulation by spray drying

Effect of spray drying conditions on oil encapsulation yield.

Statistical analysis demonstrated oil encapsulation yield (OEY) was significantly ($P<0.05$) affected only by the temperature (T) quadratic effect. Then, the temperature had a positive quadratic effect, and the behavior is explained by equation 6.

$$OEY\ (\%) = 79.165 - 0.677*(G:M) + 0.36*T + 0.119*(G:M)^2 - 1.485*(T)*(G:M) + 2.539*T^2 \quad (6)$$

Chia seed oil encapsulation yield, in this study, varied from 76.5 to 84.3%. A lower yield has been reported (39.7%) for chia seed oil encapsulating when maltodextrin in combination with hydroxypropyl methylcellulose as encapsulating wall material is used (Martínez *et al.,* 2015). The spray drying process requires a high inlet temperature to ensure full evaporation of water to prevent powder stick to the drying chamber wall. At high temperatures, inlet water evaporates easily since temperature favors the mass transfer between the chamber wall and the fluids in movement, which improves oil encapsulation yield. Similarly, the yield can also be improved if the outlet temperature is increased. Therefore, it is desirable to determine drying temperatures (inlet and outlet temperatures) to obtain a high oil encapsulation yield. In this work, the surface response graphic (Figure 1) showed the effect of the temperature and gum arabic: maltodextrin ratio (G:M ratio) on chia seed oil encapsulation yield. Yield increased with G:M ratio. Even though the G:M ratio had no significant influence ($p<0.05$), the maximum yield was observed at the lowest level G:M ratio, which corresponded to equal quantities of both gums, and the highest level of temperature near 180°C.

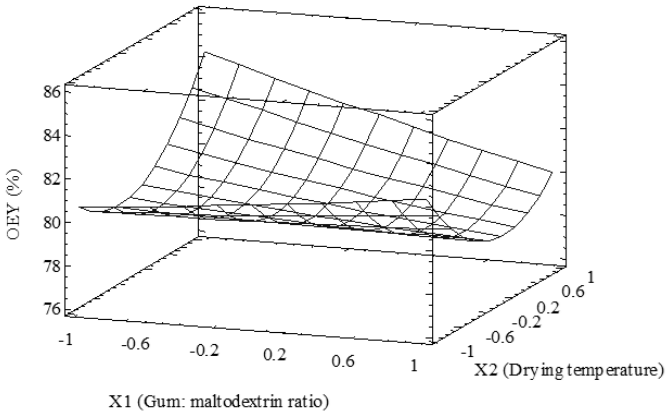

Figure 1. Effect of gum arabic: maltodextrin ratio and temperature on chia seed oil encapsulation yield (%).

3.4 Effect of process variables on encapsulation oil efficiency

Equation 7 shows a significant negative linear effect of gum: maltodextrin ratio on encapsulation oil efficiency (EOE) and a positive significant quadratic effect of this variable. The G:M ratio had no significant influence ($p<0.05$). A positive interaction on variables: G:M ratio and temperature, was observed.

$$EOE\ (\%) = 39.09 - 2.328*(G:M) - 1.105*(T) + 0.545*(G:M)^2 - 1.135*(G:M)*T + 5.245*T^2 \quad (7)$$

Response surface analysis (Figure 2) shows the effects of G:M ratio and temperature on the encapsulation oil efficiency. At a temperature of 180°C, when the maltodextrin ratio increases, a higher

https://doi.org/10.26656/fr.2017.8(4).428

encapsulation efficiency is obtained. Martínez *et al.* (2015) reported an average encapsulation efficiency of 73%, using maltodextrin 6% (DE 15) and hydroxypropyl methylcellulose 3%, resulting in higher than obtained in this work (37.7 – 49.9%). Dextrose Equivalent (DE) for maltodextrins has a significant influence on yield, when DE decreases yield increases because glass transition temperature increases (Otálora *et al.,* 2015). Contrarily, in this case, an opposite relationship was obtained, due to other factors such as, among others, the relationship of the wall material in relation to the active agent, the level of process scaling, and the presence and structural characteristics of surfactant compounds. As suggested by the results of Rincon-Duran and Arenas-Bustos (2020) who found efficiencies of 34 to 49% using maltodextrin alone and mixed with 25% soy protein as an emulsifying agent.

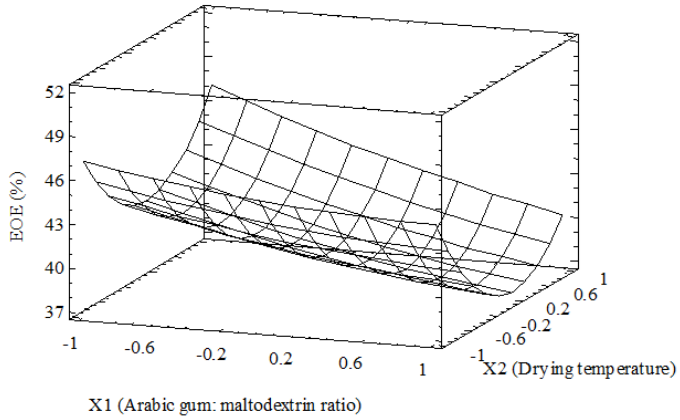

Figure 2. Effect of gum arabic: maltodextrin ratio and temperature on efficiency of chia seed oil encapsulation (%).

Using the equations obtained from the statistical design, it was possible to determine the optimal treatment (OT) at G: M ratio 1:1 and temperature 180°C to accomplish the best yield (49.5%) and to improve the efficiency of the spray drying process. In cases like the encapsulation of rich polyunsaturated fatty acid oils, a non-encapsulated nucleus can affect microcapsule stability by oxidation during storage, due to no microcapsule protection, which incises product acceptability. Produced hydroperoxide radicals can decompose easily and produce a bad flavor (Anwar and Kunz, 2011). In this work, no statistical differences ($P>0.05$) in superficial oil content (range: 15.7 - 20.4%) between treatments were detected.

3.5 Characterization of the microcapsules

3.5.1 Morphology of microcapsules

Figure 3 shows the micrographs of chia seed oil microcapsules taken in an optic microscope with a 40× lens. The sizes of microcapsules obtained (129±16 µm) were like those of fish oil microcapsules that were of the order from between 50-150 µm obtained by the spray drying process reported by Anwar and Kunz (2011). These authors found that for fish oil, there is an encapsulation of multiple nuclei and thus, larger final particle sizes (500-600 µm) due to the mechanism of layer growth and/or agglomeration which forms raspberry-like particles, a mechanism that may have occurred in the microcapsules of this work

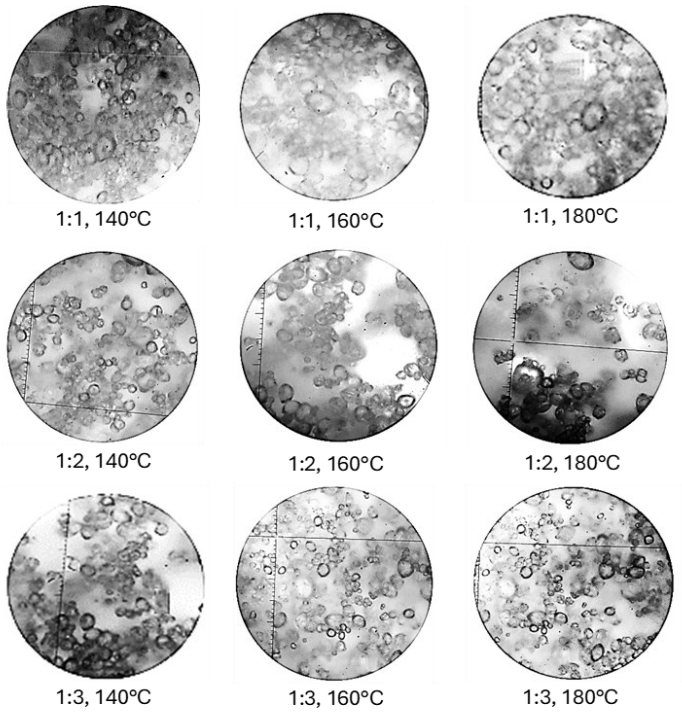

Figure 3. Micrographs of microcapsules obtained from different encapsulation conditions (40×).

The sphericity coefficient (0.11±0.07) indicated that in general, the particles tended to be spherical since a zero value of this parameter indicates a perfect sphere (Lee *et al.,* 2013). No size homogeneity was detected in all treatments, resulting in highly heterogeneous sizes at 180°C and a higher content of maltodextrin (1:3) than in the OT (1:1, 180°C). In this OT when maltodextrin content equated to gum arabic content, largest particles were obtained probably due to high viscosity related to the high content of gum in the solution. Rocha-Selmi *et al.* (2013) reported that increased viscosity caused by a high concentration of polymer might interfere with forming microcapsules since macromolecule mobility can be reduced and consequently, increase competition for solvent. Aggregation tendency can give the powders an irregular morphology. Vandana *et al.* (2014) reported the formation of amorphous regions (that may lead to poor stability in powders) when utilizing technologies such as milling, drying by crushing, and SCF (supercritical fluids).

Figure 4 shows the size change of microcapsules in two ways: the bunching of particles (probably due to superficial oil) and wall deterioration that causes agglomeration. Figures 3c (8^{th} month of storage) and 3d (12^{th} month of storage) show the fusion between particles and the fracture of their walls. The irregular

https://doi.org/10.26656/fr.2017.8(4).428

shapes of the particles could be detected from the beginning of storage. However, these irregularities increased with time, an occurrence expected in natural products (Tonon *et al.,* 2009). Excessive evaporation due to high temperatures can yield cracks in the membrane, causing premature liberation of bioactive compounds and their degradation. In addition, the high drying temperatures could also have damaged the powder.

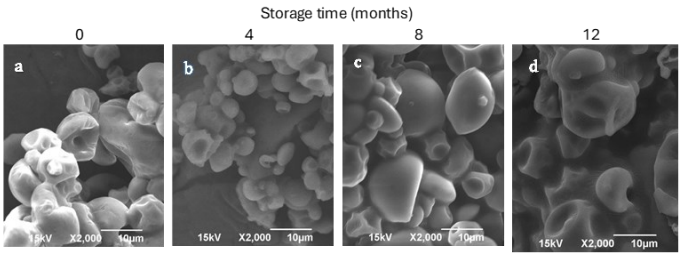

Figure 4. Micrographs of the microcapsules obtained from optimal treatment by scanning electronic microscopy (SEM) at different storage times.

3.5.2 Flow properties of microcapsules

Flow properties are directly related to storage, handling, and powder processing (Gallo *et al.,* 2011), and an index associated with the flow is the angle of repose of the dry microcapsules in powder form, which ranged between 39.21 and 49.08. The microcapsules' flow capacity varied from regular to poor when compared to results obtained by Swarbrick (1997). However, Buanz (2021) indicates that a range of angles between 25 and 45 is still acceptable. The angle of repose tends towards acceptable values when the gum arabic: maltodextrin ratio and temperature decreased as follows: 46.1 at 1:3 G:M and 180°C and 39.21 at 1:1 G:M and 140°C and for optimal treatment (1:1 G: M and 180°C) it was 42.52. Vandana *et al.* (2014) mentioned that using stabilizing agents can produce a significant effect on particles' morphology, therefore the flow and micronization are produced by spray drying. Stabilizing agents can cover the hydrophobic surface and produce low adhesiveness, lower agglomeration, and lower electrostatic effect on microparticles, which improves powder flowability despite the increase of fine particles (Vandana *et al.,* 2014) as in the present case, better flow properties were found at the higher G: MD ratio (1:1). Gallo *et al.* (2011) reported that superficial characteristics on *Rhamnus purshiana*'s microparticles influenced less on the flow angle than in the mean size of particles; therefore, large particles (129 mm) cause poor flow, but smaller particle sizes allow greater cohesiveness and therefore less flowability.

3.5.3 Moisture and water activity

Microcapsule moisture varied from 2.5% to 4%, no influence due to G: M ratio and DT was found in this characteristic. And is within the maximum limit suggested for food (Ixtaina *et al.,* 2015), this result was similar to those obtained by Porras-Saavedra *et al.* (2015) in microcapsules of soya isolate mixtures with maltodextrin/gum arabic (2.21% to 6.79%). A low moisture value is desirable since it extends microcapsules' shelf life.

Water activity (a_w) values increased (from 0.338 to 0.434) as the G: M ratio ($P<0.05$) and temperature ($P<0.05$) augmented. Thus, to obtain a lower a_w, it would be necessary to work at the lowest conditions of both factors to get an a_w value of 0.34 or under OT conditions, an a_w of 0.385. These low a_w values could make microcapsules very resistant to microbial since the a_w needed for most bacteria, yeasts and fungi are 0.90, 0.87, and 0.80, respectively. However, for critical values of Aw (0.35 to 0.45), physical changes such as adhesiveness of powder and amorphous recrystallization of sugars due to irreversible agglomerations could occur. These changes depend on glass transition temperature and storage conditions. Adhesivity of powder and agglomeration of microcapsules can turn noticeable on small bunches and chains after the eighth month of storage (Figures 4C and 4D).

Ixtaina *et al.* (2015) reported a_w values (0.20 to 0.32) for chia microcapsules, lower than those obtained in this study, by using sodium caseinate and lactose that have different abilities to interact with water. Turchiuli *et al.* (2014) pointed out that hygroscopic components, such as gum arabic or inulin, can yield a_w values higher than 0.4. Powders obtained with maltodextrin (DE = 12) only were the least hygroscopic. To preserve powders from agglomeration during storage, an atmosphere of less than 40% relative humidity is recommended which is fulfilled in the present case.

3.5.4 Color changes over storage time

Luminosity values corresponded to a clear, near-white sample (89.93 to 87.54). Positive b* values fell into the yellow color of the scale (13.15 to 17.99) and a* values to the center of the scale. Green color slightly prevailed in samples up to 8 months (-0.29), and red color in samples up to 12 months (0.93). ΔE* parameter (CIELab color difference) quantifies the difference in color between two samples, as perceived by the human eye. In this study, ΔE* varied from 1.93 (4 months) to 5.40 (12 months). ΔE* values ranging from 0.0 to 1.5 are considered small, indicating the two samples are almost identical visually. When ΔE* goes from 1.5 to 5.0, the color difference in the two samples is perceptible, and when larger than 5.0, plainly evident (Obón *et al.,* 2009). The authors mentioned that the average observer perceives differences above 5.0 or 6.0 and only a trained

https://doi.org/10.26656/fr.2017.8(4).428

eye perceives differences between 3.0 to 4.0. Nonetheless, the human eye is more sensitive to changes at levels of grey and medium tones, therefore a difference down to 0.5 units is possible to perceive in this case. The initial color loss was observed in microcapsules since the powder turned dark (decreasing L) and yellow (increasing b*) over time due to the oxidation of superficial oil and leaked oil from microcapsules.

3.6 Composition of the fatty acids in chia seed oil and microencapsulate

The content of fatty acids from raw and microencapsulated chia seed oil was determined, finding in the former 67.9% of unsaturated fatty acids (PUFA) that were identified, 16 of which corresponded to those PUFAs and 17 to saturated fatty acids (SFA). Fatty acids considered important by their concentration in chia seed oil, were as follows: linoleic acid [C18:2 (omega-6) (3.050 g/100 g oil) (UFA)], α-linolenic acid [C18:3 (omega-3) (10.731 g/100 g oil) (UFA)] and palmitic acid (SFA). Some authors (Álvarez-Chávez *et al.,* 2008; Da Silva-Marineli *et al.,* 2014) have reported these same kinds of fatty acids in chia seed oil as well. The unsaturated fatty acids linoleic acid C18:2 (ω-6) and α-linolenic acid C18:3 (ω-3) (10.731 g/100 g oil), have been pointed out as beneficial for human health, as they reduce bad cholesterol and triglycerides (Ayerza, 2011).

The linoleic and linolenic fatty acids concentration ratio was 0.81. This calculated value was probably due to the origin, the variety of chia seeds, and the extraction process utilized. Da Silva-Marineli *et al.* (2014) and Martínez *et al.* (2015) reported values of 0.29 and 0.33, respectively, for the same fatty acids ratios. The content of PUFAs in microencapsulated chia seed oil remained practically unchanged at 65.7%. The most important compounds identified, based on their concentration, were the unsaturated fatty acids: linoleic and α-linolenic.

An increase in the concentration in the chia seed oil powders of fatty acids was detected as follows: heptadecanoic (from 1.08 to 20.94 g/100 g oil), linoleic (from 3.05 to 11.88 g/100 g oil), and cis-11,14 eicosadienoic (from 0.11 to 6.02 g/100 g oil), butyric (from 0.37 to 5.54 g/100 g oil) and caproic (from 0.04 to 2.74 g/100 g oil). Yoshida and Tagaki (1997) suggested that temperature favors triglyceride hydrolysis, which increases the proportion of short-chain saturated fatty acids in vegetable oils. Lin *et al.* (2016) found that high temperature (200°C) and extended time (10 or 20 min) in the toasting process of almond kernels, can yield decomposition of fatty acids, particularly unsaturated fatty acids.

On the other hand, the chia powder fatty acid ratio (ω-6/ω-3) decreased to 0.66, and for safe human consumption, this ratio should round near one. However, by consensus, this limit should not exceed the value of 0.2. Omega-3 and omega-6 fatty acids are stored in cell membranes during their metabolism and from there released into the circulatory system. The benign effect of ω-3 fatty acids is opposite to the effect of ω-6 fatty acids on the health of the cardiovascular system, hence the importance of taking this relationship into account (Högberg *et al.,* 2003).

4. Conclusion

The yield of chia seed oil cold press extraction was 21.59%. Chia seed oil quality corresponded to an unadulterated oil with a high content of unsaturated fatty acids [α-linolenic (10.73 g/100 g oil) and linoleic (3.05 g/100 g oil) acids]. Spray drying optimal conditions for yield and efficiency were as follows: ratio of gum arabic/ maltodextrin, 1:1 (w/w) and temperature, 180°C. The yield of encapsulated chia seed oil was 49.5%. Chia seed oil microcapsules tended to be spherical by a coefficient of sphericity of 0.11 with an average size of 129 um. The gum arabic and maltodextrin ratio influenced the size of the microcapsules, causing microcapsules agglomeration. Moisture from microcapsules had a maximum value of 4% and an Aw of 0.385 obtained from optimal treatment and the ΔE* reached 5.40 value up to a year of storage. No changes in the chemical composition of microencapsulated chia seed oil were observed, preserving its content of unsaturated fatty acids. The spray drying method helped to preserve most of the physicochemical characteristics of the microencapsulated chia seed oil. The product obtained by this method could be used as a food ingredient.

References

Aberkane, L., Roudaut, G. and Saurel, R. (2014). Encapsulation and oxidative stability of PUFA-rich oil microencapsulated by spray drying using pea protein and pectin. *Food Bioprocess Technology*, 7, 1505–1517. https://doi.org/10.1007/s11947-013-1202-9

Aghbashlo, M., Mobli, H., Madadlou, A. and Rafiee, S. (2013). Influence of wall material and inlet drying air temperature on the microencapsulation of fish oil by spray drying. *Food and Bioprocess Technology*, 6 (2), 1561–1569. https://doi.org/10.1007/s11947-012-0796-7

Álvarez-Chávez, L.M., Valdivia-López, M.A., Aburto-Juárez, M.L. and Tecante, A. (2008). Chemical characterization of the lipid fraction of Mexican chia seed (*Salvia hispanica* L.). *International Journal of*

Food Properties, 11(3), 687–697. https://doi.org/10.1080/10942910701622656

Anwar, S.H. and Kunz, B. (2011). The influence of drying methods on the stabilization of fish oil microcapsules: Comparison of spray granulation, spray drying, and freeze drying. *Journal of Food Engineering*, 105(2), 367–378. https://doi.org/10.1016/j.jfoodeng.2011.02.047

Association of the Official Analytical Collaboration (AOAC) International. (1997). Official Methods of Analysis. 17th ed. Washington D.C., U.S.A.: AOAC International.

Ayerza, R. (2011). Contenido de aceite y composición de ácidos grasos de semillas de chia (*Salvia hispanica* L.) Var. Iztac 1 cultivadas en seis ecosistemas tropicales diferentes. Interciencia. *Revista de ciencia y tecnología de América*, 36(8), 620-624. [In Spanish].

Berto, B.M., Garcia, R.K.A., Fernandes, G.D., Barrera-Arellano, D. and Pereira, G.G. (2020). Linseed oil: Characterization and study of its oxidative degradation. *Grasas y Aceites*, 71(1), e337. https://doi.org/10.3989/gya.1059182

Brevedan, M.I.V., Carelli, A.A. and Crapiste, G.H. (2000). Changes in composition and quality of sunflower oils during extraction and degumming. *Grasas y Aceites*, 51(6), 417–423. https://doi.org/10.3989/gya.2000.v51.i6.460

Buanz, A. (2021). Chapter 16 - Powder characterization. In Adejare, A. (Ed.). Remington. The Science and Practice of Pharmacy. 23rd ed., p. 295–305. USA: Academic Press. https://doi.org/10.1016/B978-0-12-820007-0.00016-7

Capitani, M.I., Spotorno, V., Nolasco, S.M. and Tomás, M.C. (2012). Physicochemical and functional characterization of by-products from chia (*Salvia hispanica* L.) seeds of Argentina. *LWT - Food Science and Technology*, 45(1), 94-102. https://doi.org/10.1016/j.lwt.2011.07.012

Da Silva-Marineli, R., Aguiar Moraes, E., Alves Lenquiste, S., Teixeira Godoy, A., Nogueira Eberlin, M. and Maróstica, M.R. (2014). Chemical characterization and antioxidant potential of Chilean chia seeds and oil *(Salvia hispanica* L.). *LWT- Food Science and Technology,* 59(2), 1304-1310. https://doi.org/10.1016/j.lwt.2014.04.014

Fernandes, S.S., Greque, L., Santos, M.F.C., De Novais, L.M.R., D'oca, C.D.R.M., Prentice, C. and Salas-Mellado, M.M. (2021). Effect of the spray drying conditions on the physicochemical and structural characteristics and the stability of chia seed oil microparticles. *Journal of Applied Polymers*, 138 (39), e51015. https://doi.org/10.1002/app.51015

Gallo, L., Llabot, J.M., Allemandi, D., Bucalá, V. and Piña, J. (2011). Influence of spray-drying operating conditions on *Rhamnus purshiana* (*Cáscara sagrada*) extract powder physical properties. *Powder Technology*, 208(1), 205–214. https://doi.org/10.1016/j.powtec.2010.12.021

Gharsallaoui, A., Roudaut, G., Chambin, O., Voilley, A. and Saurel, R. (2007). Applications of spray drying in microencapsulation of food ingredients: an overview. *Food Research International*, 40(9), 1107–1121. https://doi.org/10.1016/j.foodres.2007.07.004

Gómez, A.M. and de la Ossa, E.M. (2002). Quality of borage seed oil extracted by liquid and supercritical carbon dioxide. *Chemical Engineering Journal*, 88 (1), 103-109. https://doi.org/10.1016/S1385-8947(01)00260-1

Högberg, A., Pickova, J., Andersson, K. and Lundström, K. (2003). Fatty acid composition and tocopherol content of muscle in pigs fed organic and conventional feed with different n6/n3 ratios, respectively. *Food Chemistry*, 80(2), 177–186. https://doi.org/10.1016/S0308-8146(02)00252-2

Ixtaina, V.Y., Julio, L.M., Wagner, J.R., Nolasco, S.M. and Tomás, M.C. (2015). Physicochemical characterization and stability of chia seed oil microencapsulated with sodium caseinate and lactose by spray-drying. *Powder Technology*, 271, 26–34. https://doi.org/10.1016/j.powtec.2014.11.006

Ixtaina, V.Y., Martínez, M., Spotorno, V., Mateo, C., Maestri, D., Diehl, B., Nolasco, S. and Tomás, M. (2011). Characterization of chia seed oils obtained by pressing and solvent extraction. *Journal of Food Composition and Analysis*, 24(2), 166-174. https://doi.org/10.1016/j.jfca.2010.08.006

Ixtaina, V.Y., Vega, A., Nolasco, S.M., Tomás, M.C., Gimeno, M. and Bárzana, E. (2010) Supercritical carbon dioxide extraction of oil from Mexican chia seed (*Salvia hispanica* L.); characterization and process optimization. *Journal of Supercritical Fluids*, 55(1), 192-199. https://doi.org/10.1016/j.supflu.2010.06.003

Lafont, J.J., Páez, M.S. and Portacio, A.A. (2011). Extracción y caracterización fisicoquímica del aceite de la semilla (Almendra) del marañón (*Anacardium occidentale* L). *Informacion Tecnologica*, 22(1), 51–58. https://doi.org/10.4067/S0718-07642011000100007 [In Spanish].

Lee, B.B., Raviddra, P. and Chan, E-S. (2013). Size and shape of calcium alginate beads produced by extrusion dripping. *Chemical Engineering Technology*, 36(10), 1627–1642. https://

doi.org/10.1002/ceat.201300230

Lin, J.-T., Liu, S.-C., Hu, C.-C., Shyu, Y.-S., Hsu, C.-Y. and Yang, D.-J. (2016). Effects of roasting temperature and duration on fatty acid composition, phenolic composition, Maillard reaction degree and antioxidant attribute of almond (*Prunus dulcis*) kernel. *Food Chemistry*, 190(1), 520-528. https://doi.org/10.1016/j.foodchem.2015.06.004

Martínez, M.L., Curti, M.I., Roccia, P., Llabot, J.M., Penci, M.C., Bodoira, R.M. and Ribotta, P.D. (2015). Oxidative stability of walnut (*Juglans regia L.*) and chia (*Salvia hispanica L.*) oils microencapsulated by spray drying. *Powder Technology*, 270(Part A), 271–277. https://doi.org/10.1016/j.powtec.2014.10.031

Mexican Official Standards. (1981). Alimentos para humanos. Determinación del índice de saponificación en aceites y grasas vegetales o animales (NMX-F-174-S-1981). Retrieved from website: https://www.scribd.com/presentation/348566119/Norma-Mexicana-Nmx-f-174-s-1981 [In Spanish].

Mexican Official Standards. (1987). Alimentos. Aceites y grasas vegetales o animales. Determinación del índice de acidez (NMX-F-101-1987). Retrieved from website: https://www.scribd.com/doc/223251348/NMX-F-101-1987 [In Spanish].

Mexican Official Standards. (2010). Alimentos - Aceites y grasas vegetales o animales - Determinación del índice de peróxido- Método de prueba (NMX-F-154-SCFI-2010). Retrieved from website: http://www.economia-nmx.gob.mx/normas/nmx/2010/nmx-f-154-scfi-2010.pdf [In Spanish].

Mexican Official Standards. (2011). Alimentos para humanos - Aceites y grasas vegetales o animales - Determinación del índice de yodo por el método de Wijs (NMX-F-152-S-2011). Retrieved from website: https://www.scribd.com/document/721803530/NMX-F-152-SCFI-2011-INDICE-DE-YODO [In Spanish].

Mexican Official Standards. (2012). Alimentos - Aceites y grasas vegetales o animales - Determinación de la densidad relativa - Método de prueba (NMX-F-075-SCFI-2012) Retrieved from website: https://www.scribd.com/document/633032422/NORMA-MEXICANA-NMX-F-075-SCFI-2012 [In Spanish].

Mexican Official Standards. (2012). Alimentos - Aceites y grasas vegetales o animales - Determinación de color - Método de prueba (NMX-F-116-SCFI-2012). Retrieved from website: https://www.scribd.com/document/347624055/Color-Nmx-f-116-Scfi-2012

Montgomery, D.C. (2013). Design and Analysis of Experiments. México. 8th ed, Massachusetts, USA: John Wiley and Sons, Inc.

Mosquera-Quelal, M., Reinoso-García, M. and Vásquez Castillo, W. (2017). Composición lipídicac caracterización de los ácidos grasos de la semilla de Chía (*Salvia hispánica* L.). *Industrial Data*, 20(2), 15-20. https://doi.org/10.15381/idata.v20i2.13952 [In Spanish].

Obón, J.M., Castellar, M.R., Alacid, M. and Fernández-López, J.A. (2009). Production of a red-purple food colorant from *Opuntia stricta* fruits by spray drying and its application in food model systems. *Journal of Food Engineering*, 90(4), 471–479. https://doi.org/10.1016/j.jfoodeng.2008.07.013

Otálora, M.C., Carriazo, J.G., Iturriaga, L., Nazareno, M.A. and Osorio, C. (2015) Microencapsulation of betalains obtained from cactus fruit (*Opuntia ficus-indica*) by spray drying using cactus cladode mucilage and maltodextrin as encapsulating agents, *Food Chemistry*, 187, 174–181. https://doi.org/10.1016/j.foodchem.2015.04.090

Porras-Saavedra, J., Palacios-González, E., Lartundo-Rojas, L., Garibay-Febles, V., Yáñez-Fernández, J., Hernández-Sánchez, H., Gutiérrez-López, G. and Alamilla-Beltrán, L. (2015). Microstructural properties and distribution of components in microparticles obtained by spray-drying. *Journal of Food Engineering*, 152, 105–112. https://doi.org/10.1016/j.jfoodeng.2014.11.014

Rincon-Duran, M.C. and Arenas-Bustos, I. (2020). Formulacion y evaluación de la eficiencia de cápsulas de aceite de chía por medio del método de secado por purverización (Spray-Drying). *Bogota, Colombia:* University of the Andes, BSc. Thesis. [In Spanish].

Rocha-Selmi, G.A., Theodoro, A.C., Thomazini M., Bolini, H.M.A. and Favaro-Trindade, C.S. (2013). Double emulsion stage prior to complex coacervation process for microencapsulation of sweetener sucralose. *Journal of Food Engineering*, 119(1), 28–32. https://doi.org/10.1016/j.jfoodeng.2013.05.002

Romani, V.P., Prentice-Hernández, C. and Martins, V.G. (2017). Active and sustainable materials from rice starch, fish protein and oregano essential oil for food packaging. *Industrial Crops and Products*, 97, 268–274. https://doi.org/10.1016/j.indcrop.2016.12.026

Segura-Campos, M.R., Ciau-Solís, N., Rosado-Rubio, G., Chel-Guerrero, L. and Betancur-Ancona, D. (2014). Physicochemical characterization of chia (*Salvia hispanica*) seed oil from Yucatán, México. *Agricultural Sciences*, 5, 220-226. https://doi.org/10.4236/as.2014.53025

Swarbrick, J. (1997). Encyclopedia of Pharmaceutical

Technology. Vol. 7, p. 165. New York, USA: Marcel Dekker INC.

Tonon, R.V., Brabet, C., Pallet, D., Brat, P. and Hubinger, M.D. (2009). Physicochemical and morphological characterization of açai (*Euterpe oleraceae Mart.*) powder produced with different carrier agents. *International Journal of Food Science and Technology*, 44(10), 1950–1958. https://doi.org/10.1111/j.1365-2621.2009.02012.x

Turchiuli, C., Jimenez Munguia, M.T., Hernandez Sanchez, M., Cortes Ferre, H. and Dumoulin, E. (2014). Use of different supports for oil encapsulation in powder by spray drying. *Powder Technology*, 255, 103–108. https://doi.org/10.1016/j.powtec.2013.08.026

Vandana, K.R., Prasanna Raju, Y., Harini Chowdary, V., Sushma, M. and Vijay Kumar, N. (2014.) An overview on *in situ* micronization technique - An emerging novel concept in advanced drug delivery. *Saudi Pharmaceutical Journal*, 22(4), 283–289. https://doi.org/10.1016/j.jsps.2013.05.004

Yoshida, H. and Takagi, S. (1997). Effects of seed roasting temperature and time on the quality characteristics of sesame (*Sesamum indicum*) oil. *Journal of the Science of Food Agriculture*, 75(1), 19-26. https://doi.org/10.1002/(SICI)1097-0010(199709)75:1<19::AID-JSFA830>3.0.CO;2-C

Zhang, Y., Tan, C., Abbas, S., Eric, K., Zhang, X., Xia, S. and Jia, C. (2014). The effect of soy protein structural modification on emulsion properties and oxidative stability of fish oil microcapsules. *Colloids and Surfaces. B. Biointerfaces*, 120, 63–70. https://doi.org/10.1016/j.colsurfb.2014.05.006

https://doi.org/10.26656/fr.2017.8(4).428