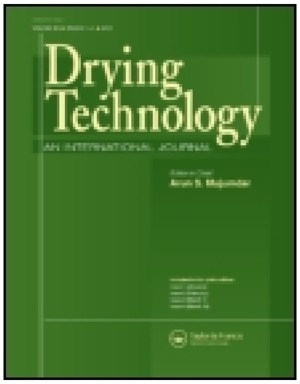

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Lorena Costa Garcia [a b], Renata Valeriano Tonon [c] & Miriam Dupas Hubinger [a]
[a] Faculty of Food Engineering, University of Campinas, Campinas, Brazil
[b] Embrapa Agroenergy, Parque Estação Biológica, Brasília, Brazil
[c] Embrapa Food Technology, Rio de Janeiro, Brazil

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Effect of Homogenization Pressure and Oil Load on the Emulsion Properties and the Oil Retention of Microencapsulated Basil Essential Oil (*Ocimum basilicum* L.)

Lorena Costa Garcia,[1,2] Renata Valeriano Tonon,[3] and Miriam Dupas Hubinger[1]
[1]*Faculty of Food Engineering, University of Campinas, Campinas, Brazil*
[2]*Embrapa Agroenergy, Parque Estação Biológica, Brasília, Brazil*
[3]*Embrapa Food Technology, Rio de Janeiro, Brazil*

The objective of this work was to evaluate the influence of oil concentration and homogenization pressure on the emulsion and particle properties during the microencapsulation of basil essential oil by spray drying, using gum arabic as the wall material. Experiments were planned according a 2^2 rotational central composite design. The independent variables were oil concentration with respect to total solids (10–25%) and homogenization pressure (0–100 MPa). Emulsions were analyzed for droplet mean diameter, stability, and viscosity, and particles were analyzed for oil retention, moisture content, particle size, and morphology. Emulsion viscosity was not affected by any of the independent variables. The increase in the homogenization pressure from 0 to 100 MPa resulted in smaller emulsion droplet size (down to 0.40 μm) and, consequently, higher oil retention (up to 95%). On the other hand, higher oil loads (25%) resulted in poorer oil retention (51.22%). Microencapsulation of basil essential oil using gum arabic as the wall material proved to be a suitable process to obtain powdered basil essential oil, presenting great oil retention with the use of lower oil concentration and higher homogenization pressure.

Keywords Basil essential oil; Emulsion properties; Gum arabic; Microencapsulation; Spray drying

INTRODUCTION

Essential oils are natural liquid products obtained from plants. They are commonly extracted by hydro or steam distillation, even though there are several extraction methods, such as supercritical CO_2, maceration, among others. Their components are substances that are sensitive to oxygen, light, moisture, and heat.[1] The stability of essential oils can be increased by microencapsulation, which consists of the entrapment or coating of those substances within another material or system.[2]

Microencapsulation is of great importance in the flavoring and food industries; using this technique, materials in the liquid form are entrapped in a carrier matrix in order to obtain a dry powder. Depending on the product use, it is better to work with it in a powder form due to the greater facility in handling and incorporation into matrices, in particular solid matrices. The advantages of this technology include protection against degradation reactions and prevention of flavor loss as well as promoting the controlled release of the core material during food processing and storage.[3]

Among the encapsulation methods, spray drying is the most popular.[3,4] It consists of three steps: atomization, dehydration, and powder collection. Basically, the solution feed is sprayed by an atomizer into a drying chamber and the atomized droplets are dehydrated as they fall down inside the chamber. The dried particles are then carried into the cyclone and settle in the product collector.[5]

Spray-drying microencapsulation relies on achieving high core material retention during processing.[2] Emulsion stability is an important factor in the encapsulation of oils and flavors, because these substances are generally insoluble in water.[6] The goal of emulsification is to produce droplets as small as possible, and various techniques can be used for this purpose. High-pressure homogenization is widely used to emulsify, disperse, homogenize and to reduce the average droplets size in order to obtain more stable emulsions.[7]

Selection of an adequate coating material for the microencapsulation of essential oils depends on its capacity to protect the oil from degradation and avoid moisture adsorption and loss of core material during processing and storage conditions. The choice of the wall material is an important step for a successful microencapsulation process.

Gums are generally used as wall material in the microencapsulation process, because they have a film-forming capacity and are able to stabilize emulsions. Among all gums, gum arabic stands out due to its excellent emulsification properties and low viscosity, even at high concentrations. The emulsification properties of gum arabic are

Correspondence: Miriam Dupas Hubinger, Faculty of Food Engineering, University of Campinas, P.O. Box 6121, 13083-862, Campinas, SP, Brazil; E-mail: mhub@fea.unicamp.br

attributed to the presence of a small amount of protein fraction in its composition, about 2%.[8,9] This wall material has been widely used in microencapsulation of oils and flavors by spray drying, such as citral and cinnamaldehyde,[10] D-limonene,[4,10] and *Lippia sidoides* essential oil.[2]

Several works can be found in literature on microencapsulation of essential oils and flavors, such as D-limonene,[4,11,12] lemon myrtle,[13] cinnamaldehyde,[10] and L-menthol.[14] However, very few articles are available on the encapsulation of basil essential oil.[15,16] Basil is a popular herb and its essential oils have been extensively used in food, perfumery, and oral and dental products.[17] The interest in this oil relies on its linalool concentration, which is not only a chemical aroma but is also a precursor for other fragrance compounds. Linalool was extensively extracted from the wood of Brazilian Amazon rosewood and has been used since the 1930s in Chanel perfume.[18,19] However, the destructive harvesting of wild trees is a threat not only to the survival of these species but also to the maintenance of biodiversity in the region.[18] Therefore, basil essential oil provides a less expensive and more abundant substitute for rosewood oil.

The main reason to encapsulate basil oil is to convert the liquid extract into a solid form in order to favor its incorporation in final food, pharmaceutical, or cosmetic formulations. Another reason is to protect the volatile constituents of the oil. In this context, the objective of this work was to evaluate the influence of emulsion composition (oil concentration with respect to total solids) and homogenization pressure on emulsion properties and microencapsulation of basil essential oil by spray drying using gum arabic as the wall material.

MATERIALS

Basil essential oil was obtained from Linax Essential Oil Extraction (Votuporanga, Brazil). The physical and compositional characteristics (major volatiles components) of the studied oil are presented in Table 1. The wall material used was the gum arabic Instantgum BA supplied by Colloides Naturels Brazil (São Paulo, Brazil).

TABLE 1
Basil essential oil properties

Botanical name	*Ocimum basilicum* L.
Extraction	Steam distillation of leaves
Color	Pale yellow transparent liquid
Odor	Herbal spicy
Relative density (kg/L)	0.8986
Polarization	−6.50 to −16
Refraction number	1.465–1.480
Boiling point (°C)	212–215
Linalool concentration (%)	39.56
1-8 Cineol concentration (%)	25.76
Camphor concentration (%)	12.67

Note: Information provided by Linax Essential Oil Extraction.

METHODS

Experimental Design

A rotatable central composite design was used to evaluate the effect of the oil concentration with respect to total solids (10–25%) and homogenization pressure (0–100 MPa) on the emulsion properties and particle characteristics. The emulsion stability coefficient, droplet size, and viscosity as well as particle oil retention were analyzed as responses. Five levels of each variable were chosen for the trials, including four axial points and three repetitions of the central point, resulting in a total of 11 studied conditions (Table 2). The following polynomial equation was fitted to data:

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_{11} x_1^2 + \beta_{22} x_2^2 + \beta_{12} x_1 x_2 \qquad (1)$$

where β_n are constant regression coefficients, y is the response, and x_1, and x_2 are the coded independent variables (oil concentration with respect to total solids and homogenization pressure, respectively).

Emulsion Preparation

The carrier solution was prepared by dispersing gum arabic in warm distilled water at 40°C until it was completely dissolved. This solution was cooled down to room temperature and stored under refrigeration (5°C) until the next day. The total solid content (gum arabic + basil essential oil) in all emulsions was fixed at 30% (w/w) and the gum arabic concentration varied from 22.5 to 27% of the total weight (w/w) or from 75 to 90% (w/w) of the total solids content. Homogenization can be divided into two categories: creation of an emulsion directly from two separate liquids, defined as primary homogenization, and reduction of the size of the droplets in an already existing emulsion, defined as secondary homogenization.[6] In the present work, primary emulsions were formed by mixing the basil essential oil with the gum arabic solution using a rotor-stator homogenizer (Ika model T18, Ultra Turrax, Staufen, Germany) operating at 14,000 rpm for 5 min. Then, a secondary emulsion was formed using a high-pressure homogenizer (PANDA2 K, Niro Soave S.P.A., Parma, Italy) in two stages. The pressure in the first stage ranged from 15 to 100 MPa and in the second stage it was fixed in 5 MPa.

TABLE 2
Experimental design, trials, responses, and particle mean diameter on axial and central points of the experimental design

Tests	Oil concentration with respect to total solids (%)	Homogenization pressure (MPa)	Droplet mean diameter D_{32} (μm)	Stability coefficient ($D_{0.5\,h}/D_{24\,h}$)	Viscosity ($\times 10^2$ Pa · s)	Particle oil retention (%)	Particle mean diameter, D_{43} (μm)[a]	Particle moisture content (%)
1	12 (−1)	15 (−1)	0.608 ± 0.014	0.830	7.37 ± 0.09	76.31 ± 4.23	—	0.34 ± 0.04
2	23 (1)	15 (−1)	0.826 ± 0.007	0.856	7.12 ± 0.28	65.00 ± 2.43	—	1.26 ± 0.04
3	12 (−1)	85 (1)	0.465 ± 0.034	0.668	9.18 ± 0.64	89.01 ± 0.08	—	0.79 ± 0.09
4	23 (1)	85 (1)	0.698 ± 0.006	0.978	6.60 ± 0.09	62.34 ± 6.15	—	1.32 ± 0.03
5	10 (−1.41)	50 (0)	0.559 ± 0.017	0.776	8.69 ± 0.47	91.36 ± 0.34	15.564 ± 0.319^{A}	1.99 ± 0.15
6	25 (+1.41)	50 (0)	0.799 ± 0.023	0.861	7.18 ± 0.02	58.25 ± 0.75	12.886 ± 0.160^{C}	1.44 ± 0.13
7	17.5 (0)	0 (−1.41)	0.910 ± 0.012	0.888	11.36 ± 0.36	56.90 ± 7.28	14.924 ± 0.270^{a}	0.86 ± 0.08
8	17.5 (0)	100 (+1.41)	0.532 ± 0.025	0.682	6.18 ± 0.21	79.43 ± 2.96	14.316 ± 0.246^{b}	1.03 ± 0.06
9	17.5 (0)	50 (0)	0.691 ± 0.008	1.017	7.45 ± 0.26	73.33 ± 0.23	14.335 ± 0.029^{Bb}	2.03 ± 0.06
10	17.5 (0)	50 (0)	0.718 ± 0.007	1.017	8.15 ± 0.02	81.44 ± 3.02	—	1.34 ± 0.10
11	17.5 (0)	50 (0)	0.658 ± 0.028	0.922	7.31 ± 0.04	75.96 ± 2.95	—	0.98 ± 0.06

[a]Particle mean diameter was not a variable studied in the experimental design for response surface methodology. Means with the same lowercase letter did not differ significantly at $P \le 0.05$. Means with the same uppercase letter did not differ significantly at $P \le 0.05$.

Emulsion Droplet Size Analysis

The emulsion droplet size was determined using a laser light-scattering method using a Mastersizer (model 2000 S, Malvern Instruments Ltd., UK). Distilled water was used as the dispersant. Measurements were reported either as the full droplet size distribution or as the mean diameter. In this study, the droplet size was expressed as the Sauter mean diameter (D_{32}; Eq. (2)), which represents the surface average diameter and is commonly used for particles with small surfaces.[13]

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

where z_i is the number of droplets with diameter D_i.

Each sample was analyzed in triplicate and three determinations were made for each repetition.

Emulsion Stability

Emulsion stability was evaluated by observing the occurrence of gravitational separation and by calculating the relationship between emulsion droplet size measured 0.5 and 24 h after the homogenization process. Because the essential oil has lower density than water, gravitational separation can be visualized by the formation of an oily phase above the emulsion, resulting from the creaming effect.[20] In spray drying, it is important to have a stable emulsion. The relationship between emulsion droplet size measured 0.5 and 24 h after the homogenization process ($D_{0.5\,h}/D_{24\,h}$), called the *stability coefficient*, was used as an indicator of the emulsion's stability, because it measures the increase in droplet size caused by droplet coalescence. The closer this coefficient is to1.0, the more stable the emulsion is.

Rheological Measurements

Emulsion viscosity was measured through the determination of steady-shear flow curves using a controlled stress Physica MCR301 rheometer (Anton Paar, Graz, Austria) with stainless steel plate–plate geometry with a diameter of 75 mm and a gap of 0.5 mm. Three flow ramps (up, down, and up cycles) were obtained in a range of shear stress corresponding to shear rates from 0 to 300 s^{-1} in order to eliminate any possible thixotropy effect. Trials were performed in triplicate, using a new sample for each repetition. Rheograms were analyzed according to empirical models and viscosity was calculated as the relationship between the shear stress and shear rate.

Spray Drying

Emulsions were spray dried in a bench-top spray dryer (model MSD 1.0, Labmaq, Ribeirão Preto, Brazil) with a concurrent flow and equipped with a dual-fluid nozzle of 1.2 mm in diameter. The drying chamber had a diameter of 180 mm and a height of 520 mm. The emulsion was fed into the drying chamber through a peristaltic pump with a feed flow rate of 0.7 L/h^{-1}. The inlet and outlet air temperatures were 180 and 110°C ± 5°C, respectively, and the compressed and drying air flow rates were fixed at 2.4 m^3 h^{-1} and 0.0197 kg s^{-1}. The dried product was collected in a stainless steel cyclone with a diameter of 80 mm and a cut diameter of 4.48 μm. The cyclone cut diameter was estimated by the semi-empirical relationship proposed by Lapple.[21] The powders were produced in a laboratory at a temperature of 25 ± 2°C.

Oil Retention on Particles

The total oil retained in the microencapsulated powders was determined by hydrodistillation in a Clevenger apparatus, in triplicate, according to the method described by Bhandari et al.,[22] with some modifications. Five grams of powder was dissolved in 150 mL of distilled water in a 500-mL round-bottomed flask. About 0.5 mL of antifoam (Samy, Cajamar, Brazil) was added to the solution. Distillation was performed for 40 min and the volume of distilled essential oil was directly read in the Clevenger apparatus. The mass of oil in the microcapsules was obtained by multiplying the volume of oil read on the graduate region of the Clevenger apparatus ($V_{distilled\_oil}$) by the density (ρ) of basil essential oil (0.8986 g cm^{-3}). The total oil retained in the microcapsules was calculated according to Eq. (3):

$$\text{Oil retention (\%)} = \frac{(V_{\text{distilled\_oil}} \times \rho)}{M_{\text{initial\_oil}}} \times 100 \tag{3}$$

Moisture Content

The particle moisture content was determined gravimetrically by drying a 2-g sample in an oven at 105°C for 3 h.[23] Assays were carried out in triplicate. The results were expressed as percentage wet basis.

Powder Particle Size Analysis

Powder particle size was determined using a laser light-scattering method using a Mastersizer (model 505, Malvern Instruments Ltd.). Ethanol (99.5%) was used as the dispersant. Measurements were reported as the full particle size distribution and as the mean diameter. In this study, the powder size was expressed as the Brouckere diameter (D_{43}; Eq. (4)), which represents the volume mean diameter and, according to Huynh et al.,[13] is associated with larger surfaces.

$$D_{43} = \frac{\sum_{i=1}^{n} z_i d_i^4}{\sum_{i=1}^{n} z_i d_i^3} \tag{4}$$

where z_i is the number of droplets with diameter d_i.

A size distribution histogram is presented that associates the volume of particles (%) with the powder particle diameter (μm). Each sample was analyzed in triplicate and three determinations were made on each repetition.

Morphological Characteristics

Particle morphology was evaluated by scanning electron microscopy (SEM). Powders were attached to a double-sided adhesive tape mounted on SEM stubs, coated with 3–5 mA gold/palladium under vacuum, and examined with a Leo 440i scanning electron microscope (Leica Electron Microscopy Ltd., Cambridge, UK). The SEM was operated at 5 kV with magnifications of 5,000 ×.

Statistical Analysis

Analysis of variance (ANOVA), test for the lack of fit, determination of R^2, and the generation of three-dimensional graphs were carried out using Statistica 7.0 software (StatSoft, Tulsa, OK).

RESULTS AND DISCUSSION

Response Surface Analysis

The trials performed for the central composite design and the results obtained for the stability coefficient, droplet

TABLE 3

Coded second-order regression coefficients for droplet mean size, viscosity, and oil retention

Coefficient	D_{32} (μm)	Stability coefficient	Viscosity (× 10^2 Pa · s)	Oil retention (%)	Moisture content
β_0	0.678	0.984	0.076	73.57	0.727
β_1	0.099	0.114	NS	−10.61	NS
β_2	−0.10064	−0.831	NS	5.24	NS
β_{11}	NS	−0.151	NS	NS	NS
β_{22}	NS	−0.185	NS	NS	NS
β_{12}	NS	0.142	NS	NS	NS
R^2	0.890	0.849	—	0.81198	—

NS = Nonsignificant.

mean diameter, viscosity, particle oil retention, and moisture content are shown in Table 2.

Table 3 shows the regression coefficients for the coded second-order polynomial equation and the coefficient of determination (R^2). Some nonsignificant terms were eliminated and the resulting equations were tested for adequacy and fitness by ANOVA. The fitted models were suitable, showing significant regression, low residual values, no lack of fit, and satisfactory coefficients of determination.

Emulsion Droplet Mean Diameter

The influence of oil concentration in relation to the total solids and homogenization pressure on the droplet mean diameter is presented in Fig. 1.

The droplet mean diameter varied from 0.46 to 0.91 μm and was significantly affected by both oil concentration and homogenization pressure. The oil droplet size decreased with the increase in homogenization pressure, due to the high energy input by high-pressure homogenization, which promoted the disruption of the primary emulsion droplets. Soottitantawat et al.[4] and Nuchuchua et al.[24] also observed a reduction in droplet size with an increase in the homogenization pressure used for emulsification of D-limonene; and citronella, hairy basil, and vetiver oil, respectively.

Higher oil concentrations led to higher mean diameters. An increase in oil concentration implies a lower gum arabic content for the same total solids content. Because gum arabic has emulsifying properties, the lower concentration of this wall material may have resulted in a less efficient emulsification. Similar results were obtained by Soottitantawat et al.[14] in the microencapsulation of L-menthol using gum arabic and modified starches as wall materials and by Floury et al.[25] in the microencapsulation of sunflower oil using whey protein concentrate as the wall material. Beristan et al.[26] also observed an increase in emulsion droplet size when the initial cardamom essential oil concentration increased from 1:5 to 1:3 (oil: mesquite gum).

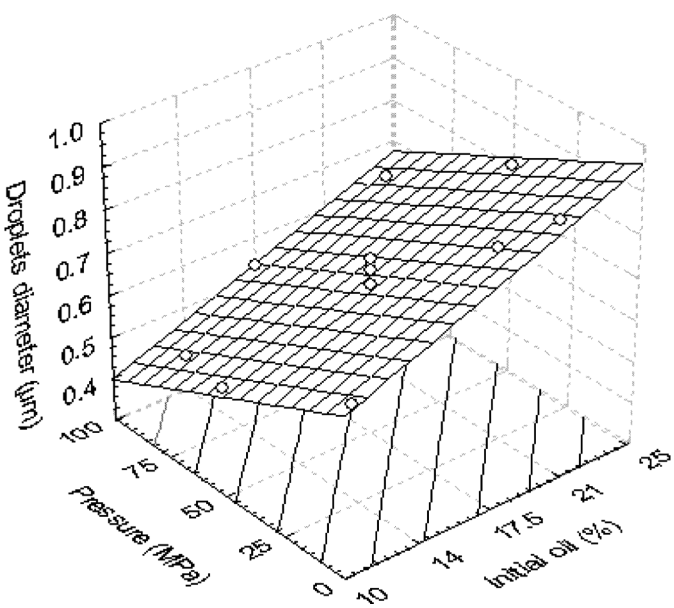

FIG. 1. Response surface for emulsion droplets mean diameter.

Emulsion Stability

No gravitational separation was observed 24 h after emulsification; thus, all of the studied emulsions were considered kinetically stable. The encapsulation efficiency of oils and flavors is expected to be influenced by the stability of initial emulsion such that better stability leads to higher efficiency.[27] According to McClements,[6] the stability of an emulsion to gravitational separation can be enhanced by reducing the size of the droplets it contains. Even though the evaluation of emulsion stability by observation of phase separation is a macroscopic and qualitative analysis, it is of great importance, especially in the encapsulation of oils, because the observation of an oily layer on the emulsion can be related to poor encapsulation efficiency of the wall material or ineffective emulsion homogenization.

Primary homogenization is defined as the production of an emulsion directly from two immiscible liquids, and secondary homogenization (in the present work, high-pressure homogenization) consists of the application of a high amount of energy in order to reduce the droplets size of a primary emulsion.[6] However, all of the emulsions tend to destabilize, resulting in separated phases. In the case of oil-in-water emulsions, there are different destabilizing mechanisms, including gravitational creaming, sedimentation, flocculation, and coalescence.[28]

Therefore, although no phase separation was observed in any of the emulsions, droplets may have coalesced during the storage period, which can also be an indicator of emulsion stability. Thus, in order to quantify the emulsion stability, the relationship between the droplet mean diameter measured 0.5 and 24 h after homogenization ($D_{0.5\,h}/D_{24\,h}$) was calculated. As previously mentioned, the closer this coefficient is to 1.0, the more stable the emulsion is in the time period considered.

As shown in Table 2, the stability coefficient varied from 0.668 to 1.017 and was significantly affected by both the oil concentration and homogenization pressure. Figure 2 shows the influence of these variables on the stability coefficient of the different emulsions.

The factor that most affected this response was homogenization pressure, which had a negative effect on the stability coefficient. When the droplet mean diameter is measured shortly after the homogenization process, the result is a small droplet size, either because the homogenization process was effective and the wall material has good emulsification properties or due to the high amount of energy retained in the emulsion. However, when the

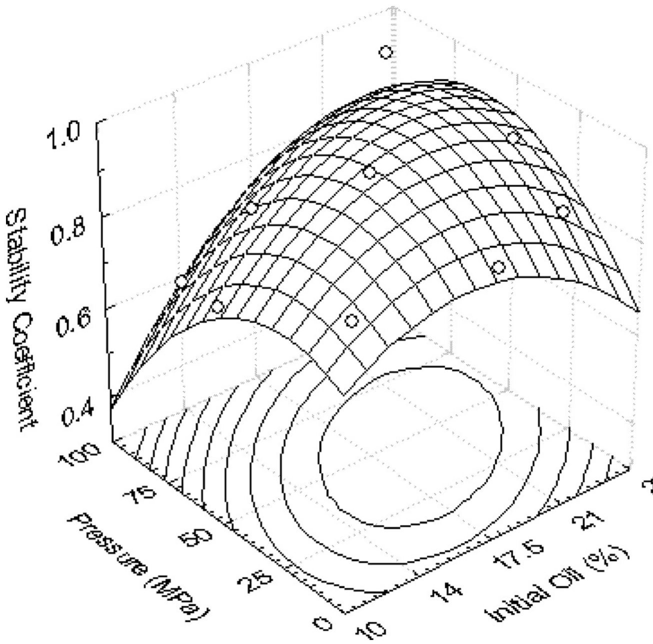

FIG. 2. Response surface for stability coefficient of emulsions.

emulsion stays at rest for a certain period of time after homogenization, the energy is dissipated and it is possible to observe the formation of larger droplets, indicating droplet coalescence.

In the present work, although the increase in the homogenization pressure reduced the droplet size at both 0.5 and 24 h after homogenization, the emulsions produced under higher homogenization pressures were less stable, showing higher coalescence levels.

The initial oil concentration showed a slight effect on the stability coefficient and was more pronounced when high homogenization pressures were applied. As previously discussed, a lower oil content resulted in smaller droplets diameters, which represents greater surface area, making coalescence easier and thus resulting in lower stability coefficients.

Finally, according to Fig. 2, the most stable emulsions (stability coefficients closest to 1) were obtained when the initial oil concentration was greater than 14% and homogenization pressures between 25 and 75 MPa were used.

Viscosity

All of the emulsions presented a Newtonian behavior, according to which viscosity is constant with the shear rate. Neither homogenization pressure nor oil concentration had an effect on the emulsion viscosity. Because the use of gum arabic as a wall material results in the formation of emulsions with low viscosity, even at high concentrations, no significant changes in the emulsion viscosity were observed with the use of different homogenization pressures and oil concentrations. Thus, because none of the independent variables had a statistically significant effect on viscosity, no model was obtained for this response.

According to Floury et al.,[25] passing an emulsion though the homogenizer at a very high pressure reduces its viscosity until a limiting value. This means that in emulsions with low viscosity, such as gum arabic–based emulsions, the effect of high-pressure homogenization on viscosity will probably not be significant.

Particle Oil Retention

Oil retention varied from 56.90 to 91.36% and was positively affected by homogenization pressure and negatively affected by the oil content, as shown in Fig. 3. Higher flavor loads resulted in poorer flavor retention because higher oil loads led to a greater proportion of volatiles close to the drying surface, thereby shortening the diffusion path length to air and favoring flavor loss. Moreover, the increase in oil concentration implies a decrease in the amount of wall material (for a fixed total solids content), which may be not enough to cover the oil droplets, making the loss of volatile compounds easier. Adamiec and Kalemba[1] also reported a decrease in the encapsulated oil with an increase in oil concentration when encapsulating elemi and peppermint oil with maltodextrin as the wall material. Beristain et al.[26] observed a reduction from 83 to 74% of cardamom oil retained in particles when the oil concentration increased from 20 to 25%.

On the other hand, the increase in oil retention resulting from the increase in homogenization pressure is related to

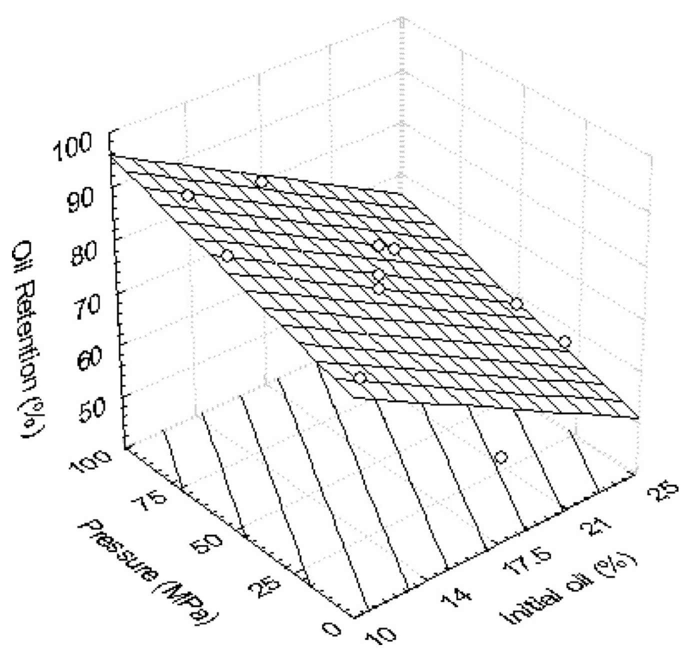

FIG. 3. Response surface for oil retention in particles of basil essential oil.

the emulsion characteristics, because higher homogenization pressures resulted in emulsions with smaller droplet diameters and viscosity. Similar results were obtained by Soottitantawat et al.,[4] who observed higher oil retention for smaller emulsion droplets in the microencapsulation of D-limonene by spray drying. Evaporation of flavor during atomization seems to be easier for emulsions with larger droplets.[3]

Particle Size Distribution and Mean Diameter

The powder size was expressed in terms of De Brouckere mean diameter (D_{43}), which represents the diameter of a sphere with equivalent volume. The size of particles obtained from emulsions with different oil concentrations and homogenized at different pressures is presented in Table 2. These conditions are relative to the axial and central points of the experimental design, in which one variable stays fixed and the other varies on three levels.

A slight but significant reduction in particle mean diameter was observed with the increase in oil concentration when the homogenization pressure was fixed at 50 MPa. On the other hand, particles produced from emulsions homogenized at different pressures with a constant oil concentration (17.5%) presented similar sizes. Studying the microencapsulation of D-limonene essential oil with gum arabic, Soottitantawat et al.[4] also reported that particles produced from emulsions homogenized at different pressures showed similar droplet diameters.

Particle size is dependent on the physical properties of the matrix to be dried (such as viscosity and solids concentration), drying temperature, and operating parameters chosen for atomization. In the present work, a linear relation between particle mean diameter (D_{43}) and emulsion viscosity was observed. For a fixed homogenization pressure (50 MPa), a reduction in emulsion viscosity and particle mean diameter as the initial oil concentration increased was observed. For instance, if we maintained the initial oil concentration fixed at 17.5%, an increase in homogenization pressure resulted in emulsions with reduced viscosity and particles with lower mean diameter. According to Jafari et al.,[27] the influence of particle size on the encapsulation efficiency of flavors and essential oils is still not clear. Soottitantawat et al.[4] reported that is not possible to make conclusions with respect to flavor retention, evaluating only powder size. According to these authors, a larger powder size leads to greater stability and less release of encapsulated flavor if the initial emulsion size is small. However, Finney et al.[11] concluded that particle size does not have a significant effect on oil retention if high in-feed solids are used.

Figure 4 shows the particle size distribution for the powders produced at different oil concentrations with respect to total solids (%) and homogenization pressure. Particles showed a bimodal distribution, with two distinctive peaks,

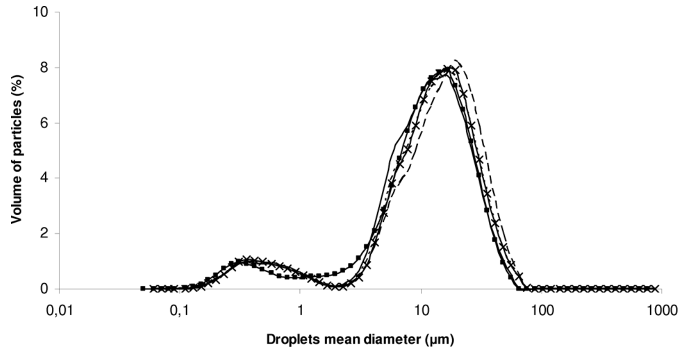

FIG. 4. Particle size distribution. –––10% oil; 50 Mpa; —■—17.5% oil; 0 Mpa; - - - - 10% oil; 50 Mpa; —— 25% opl; 50 Mpa; —*— 17.5% oil; 100 Mpa.

each one representing a predominant size. In the case of powders, this is particularly interesting because the small particles can penetrate into the spaces between the larges ones, occupying less space. The presence of larger particles may be attributed to the beginning of the agglomeration process, where the formation of irreversible bridges leads to the production of larger particles.[29]

Particle Moisture Content

As observed for emulsion viscosity, homogenization pressure and oil concentration did not influence the particle moisture content. One explanation may be the low particle moisture content obtained. Soottitantawat et al.[3] did not observe an effect of the emulsion droplet size and type of wall material on water content of the powder and suggested that this property is directly related to the drying conditions.

Because there were no effects of the studied variables on moisture content, it was not possible to obtain a predictive model or, consequently, a response surface.

Particle Morphology

The microstructure of the obtained powders was observed through SEM photographs. SEM is a technique that allows the evaluation of the outer and inner structures of particles. Figure 5 shows the surface and Fig. 6 shows the inner structure of basil essential oil powder encapsulated with gum arabic.

The morphology of the particles obtained by spray drying affects the flow properties of the obtained powder and, therefore, this characteristic is important. In addition, the encapsulation ability of various polymers can be evaluated based on the degree of integrity and porosity of the particles.

Particles with a spherical shape and an irregular, dented surface were observed in all of the studied conditions (Fig. 5). Dents are commonly observed in spray-dried particles and, according to Rosenberg et al.,[30] they are related

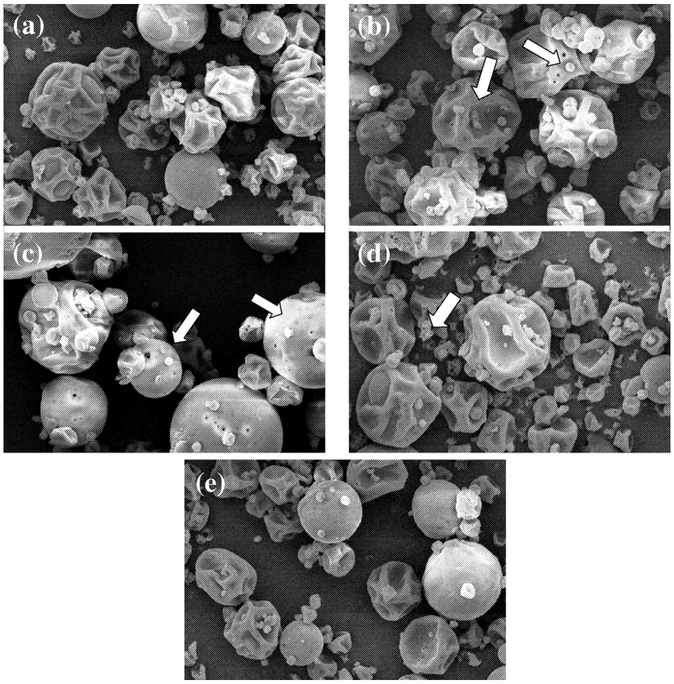

FIG. 5. Microstructure of encapsulated basil essential oil, using different initial oil concentration and homogenization pressures. Magnification: 5,000x. a) 10% oil/solids, 50 MPa; b) 25% oil/solids, 50 MPa; c) 17.5% oil/solids, 0 MPa; d) 17.5% oil/solids, 50 MPa; e) 17.5% oil/solids, 100 MPa.

to the shrinkage of the particles during the spray-drying process. Soottitantawat et al.[4,14] and Fernandes et al.[2] reported similar morphology for particles of D-limonene, L-menthol, and *L. sidoides* essential oil encapsulated using spray drying.

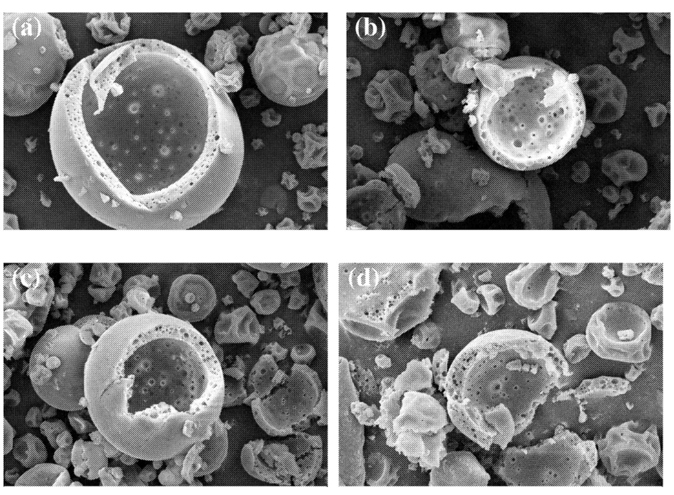

FIG. 6. Internal structure basil essential oil encapsulated with gum Arabic. Magnification: 5,000x. a) 10% oil/solids, 50 MPa; b) 25% oil/solids, 50 MPa; c) 17.5% oil/solids, 100 MPa; d) 17.50% oil/solids, 50 MPa.

According to Figs. 5a, 5b, and 5d, it is possible to observe an increase in holes on the particle surface as the oil concentration increased with respect to the total solids for the same homogenization pressure. The surfaces of particles with less basil essential oil (10% w/w) presented high integrity and the essential oil droplets were dispersed on the shell of the wall matrix. This can explain the high essential oil retention (~90%) observed in the particles formed with this oil concentration, which seems to favor retention of flavor compounds. More holes and cracks were observed on particles with higher oil uploads (17.5 and 25% w/w), favoring loss of core material and leading to lower oil retention.

The cracks and holes observed on some particles probably occurred during the final drying stages and are related to the poor viscoelastic properties of the wall material. They may also be related to the emulsification capacity of the wall material. For a given initial oil concentration (17.5%), the incidence of pores and cracks decreased with an increase in homogenization pressure (Figs. 5c, 5d, and 5e). At low homogenization pressure, the wall material was not able to effectively emulsify the core material under the process conditions used in this work, which explains the lower amount of oil retained in particles produced with low homogenization pressure.

The small holes observed on the internal surface of particles are droplets of basil essential oil embedded on the shell of the wall matrix (Fig. 6). A large void is observed in the center of the particle that occupies most of the particle volume. The spray-dried droplets in contact with the hot air were dehydrated and randomly repeated the inflation and shrinking processes. As a result, voids were present in the center of most of the particles.[3,31]

CONCLUSIONS

Microencapsulation of basil essential oil using gum arabic as the wall material is a suitable process to obtain powdered flavors. The study of the emulsion properties is of great importance in the production of particles with high oil retention. Particles produced from emulsions with a small droplet size resulting from the application of high homogenization pressure presented higher oil retention. In addition, the use of low initial oil concentration in relation to total solids resulted in particles with higher oil upload. Therefore, the best conditions for basil oil encapsulation to achieve the highest oil retention in the range of independent variables studied were oil concentration between 10 and 14% and homogenization pressures greater than 50 MPa.

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