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Research Article

Effect of emulsifying conditions on the microencapsulation quality of orange (*Citrus sinensis)* essential oil by spray drying

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Abstract

The effect of emulsification conditions on the encapsulation quality of spray-dried orange essential oil (OEsO) using gum Arabic as wall material was evaluated. Encapsulation quality was determined by encapsulation (EnE) and retention (ReE) efficiencies, as well as moisture and bulk density of the microcapsules. According to the analysis of variance, oil concentration, emulsification temperature and ultrasound exposure time of the emulsion, as well as their double and triple interactions, had a significant effect ($p < 0.05$) on EnE, ReE, moisture and bulk density of the encapsulated OEsO. Oil concentration of 10%, emulsification temperature 35°C and ultrasound exposure time of 0 min produced the best encapsulation quality of OEsO, considering EnE (98.54%) and ReE (98.54%) simultaneously. Under such conditions, the moisture content (5.0%) and apparent density (0.430 g/mL) of the encapsulated OEsO ensure its proper preservation, as well as its handling and storage. The results of this study demonstrate the relevance of emulsion preparation conditions on the encapsulation quality of spray-dried OEsO, which could impact the industrial processing of this food ingredient.

1. Introduction

Essential oils (EsOs) are aromatic and volatile liquids recovered from plant material, including flowers, roots, bark, leaves, seeds, peels, fruits, wood, and whole plants, taking specific names from the original sources [1]. EsOs are used as ingredients in industries related to health, beauty, and food, reaching global trade of $ US 6,050 million by 2021, of which 8.54% corresponded to (OEsO) essential oil orange [2].

In the food industry, OEsO is commonly used as a flavoring agent in the preparation of beverages, desserts, salad dressings, as well as a flavor enhancer in sauces and marinades [3]. In addition, OEsO has applications as a natural food preservative due to its antimicrobial properties [4]. The nutraceutical products are another very important area of opportunity for the OEsO, due to their immune, antioxidant, anti-inflammatory, anxiolytic and digestive properties [5].

Like all EsOs, OEsO contains chemicals that undergo degradation due to light, air, heat, strong acids, and alkalis, in addition to being almost insoluble in water [6]. To avoid or delay the degradation of the EsOs,

certain treatments or conditions must be applied against the action of these factors [7], among which microencapsulation stands out [8]. Microencapsulation is the process of coating small particles and droplets or the integration of compounds in a homogeneous or heterogeneous matrix to obtain small capsules [9].

For the encapsulation of the EsOs various technologies such as complex coacervation [10] extrusion [11], lyophilization [4], ionic gelation [12], emulsification [13], interfacial polymerization [14], complexation by molecular inclusion [15], liposomal entrapment [16], fluidized bed drying [17], spray freeze drying [18], and spray drying [19] have been applied. However, from these processes, spray drying is the oldest and most used in the food industry because it presents many advantages, such as low manufacturing costs, large-scale continuous production, great availability of matrices for encapsulation, as well as adequate efficiency in retention and stability of volatile substances [20].

Spray drying involves the atomization of emulsions in a drying chamber at a relatively high temperature, resulting in rapid evaporation of water and thus the formation of an almost instantaneous shell in which oils are trapped [21]. Encapsulation by spray drying comprises 4 stages: i) preparation of the dispersion, ii) homogenization of the dispersion-formation of the emulsion, iii) atomization of the emulsion, and iv) dehydration of the atomized particles [22]. The two most critical steps that determine the encapsulation effectiveness and the physicochemical characteristics and stability of the EsOs by spray drying are the selection of the coating material and the emulsification conditions [9].

The main carriers or wall materials used in encapsulation processes include carbohydrates, gums, fibers, proteins, and waxes. Of them, polysaccharide-type carbohydrates such as maltodextrins, modified starches, gum Arabic, carrageenan, and carboxymethyl cellulose are frequently employed as materials for encapsulation by spray drying, due to their cost-benefit and favorable properties [23]. However, gum Arabic, which is a biopolymer consisting of D-glucuronic acid, L-rhamnose, D-galactose and L-arabinose, with approximately 2% of associated protein content, is appreciated due to its excellent results for encapsulation [24]. Furthermore, this hydrocolloid has high water solubility, film-forming capacity and good emulsifying characteristics, and desirable properties for the preparation of emulsions for encapsulation of bioactive substances [25]. The proteins in the gum Arabic molecule possibly function as a stabilizer, providing the ability to form microcapsules and emulsify with all the EsOs [19].

Once the wall material has been selected, the next stage for EsOs encapsulation by spray drying is the preparation of the emulsion. The emulsion is elaborated by mixing the wall material suspension and EsO at high speed [26]. Emulsion stability is a critical factor for encapsulation of EsOs because it defines the encapsulation efficiency, oil retention, and storage stability of the microparticles [24]. Conditions such as total content of solids, temperature, time and type of mixing or homogenization for emulsifying are considered critical to prepare an effective infeed emulsion to be spray dried [27]. Currently, among the devices for generating emulsions, high-pressure systems, ultrasonic homogenizers, rotor-stator, and membrane systems stand out, whose use depends on the viscosity of the medium and the desired droplet size in the emulsion [9].

Regarding the research that has been carried out recently on the encapsulation of orange essential oil by spray drying, efforts have focused on evaluating the quality of encapsulation with carbohydrates, proteins, or their combinations, both from traditional sources and new sources. Barboza de Souza et al. [28] tested gum Arabic, maltodextrin, cellulose nanofibrils and their combinations. Márquez-Gómez et al. [29] studied native rice starch, modified rice starch, maltodextrin and hydrolyzed gelatin and their mixtures. Lopes Francisco et al. [30] used pea and soy proteins. However, gum Arabic stands out as a wall material because it exhibits low viscosity and high solubility in aqueous media, as well as being an excellent emulsifier, which favors encapsulation by spray drying [31]. To the best of our knowledge, no detailed studies have been carried out on the impact of emulsion preparation conditions on the encapsulation quality of OEsO by spray drying.

Therefore, the aim of this study was to evaluate the effect of the oil concentration, temperature and,

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ultrasonication time as emulsion preparation conditions on the encapsulation quality of OEsO by spray drying.

2. Materials and methods

2.1 Materials

The OEsO (density at 25°C = 843 kg/m^3, refractive index = 1.4232, optical density = + 96.2°, evaporation residue = 2.6% and optical rotation = 1.7%) and the gum Arabic used in this study were of food grade and were purchased from the company Aceites Esenciales de Occidente, S.A. of C.V. (Guadalajara, Jal., Mexico). All other substances used in this work were analytical grade.

2.2 Formulation and preparation of the emulsions

For the emulsion formulations, a 3^3 factorial design was considered [32]. A base mixture of gum Arabic (19%) and deionized water (81%) was employed as encapsulation media. To the encapsuling, base mixture was added the concentration of OEsO (10%, 20% or 30%) at a defined temperature (25°C, 35°C or 55°C), to finally expose it to ultrasound (0 min, 2 min or 4 min).

First, the gum Arabic was added to deionized water at 50°C and mixed for 16 h at room temperature (25°C) with a magnetic stirrer. Next, the water-gum Arabic mixture was adjusted to the emulsification temperature, the OEsO was added and it was homogenized at medium speed in an Oster model BLST4655 domestic blender (Newell Brand de México, S.A. de C.V., México) for 7 min. Finally, the obtained emulsion was placed in a 500 mL beaker to be treated in a Branson model 2410 MT ultrasonic bath (Branson Ultrasonic Corporations, USA).

2.3 Characterization of the emulsions

Since the oil concentration is crucial to determine the feasibility of emulsion formation, its characterization was carried out based on the OEsO concentration. Viscosity and pH at 25°C were measured with Brookfield viscometer LVT (AMETEK Brookfield Inc., USA) and a Hanna pH meter model HI991001 (Hanna Instruments Inc., USA), respectively. The morphology and size of the emulsified OEsO droplets were determined with a Leica Microsystems model DM500 optical microscope (Leica Microsystems GmbH, Germany), using a 40x objective lens.

The separation of serum from the emulsions was taken as the stability index [33]. The emulsions were placed in 100 mL graduated cylinders and observed for 0, 24, 48, 72, 96, h, at 25 °C. The height of the separated serum from the emulsions was monitored and the % separation was calculated using the following equation [34]:

$$\text{Separation (\%)} = \frac{H_1}{H_0} \times 100$$

where H_0 = emulsion initial height and H_1 = upper phase height.

2.4 Emulsion encapsulation by spray drying

The emulsions (2 L) were processed in a GEA NIRO A/S Production Minor spray dryer (Niro Atomizer, Japan) at 25 °C as feed temperature, 185 °C and 114 °C as air inlet and outlet temperatures to the dryer. The spray dried samples were stored in glass amber bottles at 5° C until their use.

2.5 Encapsulation (EnE) and retention (ReE) efficiencies

EnE and ReE of OEsO were calculated by the following equations [35]:

$$EnE\ (\%) = \frac{A_T - A_S}{A_T} \times 100$$

$$ReE\ (\%) = \frac{A_T}{A_I} \times 100$$

where A_T = content of oil in power (g), A_S = content of surface oil in powder (g), and A_I = initial oil mass added to feed emulsion.

A_T was determined according to a slightly adjusted procedure reported by Jafari et al. [36]. Twenty grams of the powder was mixed with 150 mL of deionized water and distilled in a Clevenger apparatus for 3 h. A_T in the microcapsules was obtained by multiplying the volume of the oil contained in the graduated burette by the OEsO density (0.831 g/cm^3 at 20 °C).

A_S was measured according to Dima et al. [37] with some light modifications. Four grams of the powder was stirred with 10 mL n-hexane for a 10 min, followed by suspension filtration and washing with 6 mL petroleum ether three times. The powder was dried in an oven at 90 °C to constant weight. A_S was expressed as a difference between the initial weight of microcapsules and the mass of the dried powder.

2.6 Moisture and bulk density

The moisture content (MC) of the powders of encapsulated OEsO was determined by the co-distillation technique with toluene [38], while the apparent density (ρ_A) was considered as the ratio of

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their mass to occupied volume using a 100 mL graduated cylinder [39].

2.7 Statistical analysis

To assess the effect of the factors used in the OEsO emulsification preparation on the response variables of the encapsulated OEsO by spray drying, an analysis of variance (ANOVA) was carried out through a 3^3-factorial design with two repetitions, using Statgraphics Centurion Version XV software (Statpoint Technologies, Inc. Virginia, USA). Also, the results of the analysis for characterization of the OEsO emulsion were subjected to ANOVA. Duncan's test was employed to define the difference between treatments ($p < 0.05$).

3. Results and discussion

3.1 Characteristics of OEsO emulsions

The % serum separation of the OEsO emulsions at oil concentrations of 10%, 20% and 30% was zero, hence they were highly stable during the evaluation time of 96 h (Fig. 1) and suitable for encapsulation by spray drying. The encapsulation of emulsions by spray drying requires stable emulsions to ensure high oil retention of microcapsules [30].

The effects of the OEsO concentration on the viscosity, pH, and droplet size (diameter) of the emulsions are shown in Table 1.

Table 1. Effect of orange essential oil concentration on the viscosity, pH and droplet size of orange essential oil emulsions.

Orange oil (%)	Viscosity (cP at 25°C)	pH	Droplet size (μm)
10	22.4 ± 0.1[a]	4.05 ± 0.02[a]	8.1 ± 2.1[a]
20	20.4 ± 0.1[b]	4.20 ± 0.02[b]	8.9 ± 2.4[a]
30	20.0 ± 0.1[c]	4.25 ± 0.01[c]	9.3 ± 2.5[a]

Within each column, means with different lowercase letter are significantly different ($p < 0.05$).

With increasing oil concentration, a similar morphology (Fig. 2) and no appreciable difference in droplet size of the OEsO emulsions were observed, as well as a slight decrease in viscosity from 24.4 cP to 20.0 cP and a small increase in pH from 4.05 to 4.25. Contrary to the reduction in viscosity at higher concentrations of OEsO in the emulsions of this study, an increase in this characteristic was observed for the *Zanthoxylum schinifolium* essential oil emulsions [40].

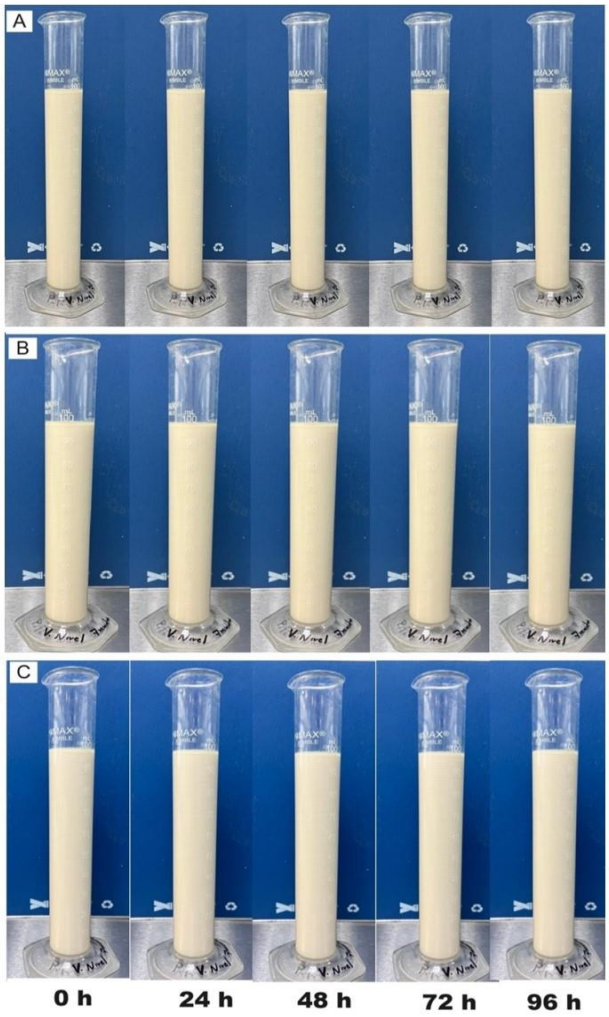

Figure 1. Effect of orange oil essential (OEsO) concentration on the emulsification stability as a function of time. A= 10% OEsO, B= 20% OEsO, C= 30% OEsO.

A low viscosity of the emulsions, as found in this study, is decisive for their encapsulation by spray drying, which also influences the generation of spherical, dense and regular microparticles [41].

3.2 EnE of OEsO

EnE is an indicator used to measure the proportion of the initial essential oil in the emulsion that is trapped within the outer layer of a wall material or capsule after encapsulation by spray drying. A high value of this parameter is desirable because is associated with the degree of protection of the functional properties of EsOs [21]. Table 2 shows the ANOVA of the results of the factors involved in the preparation conditions of the OEsO emulsions on EnE. As can be seen, both the main factors (OEsO concentration, temperature of emulsifying, and ultrasound exposure time), as well

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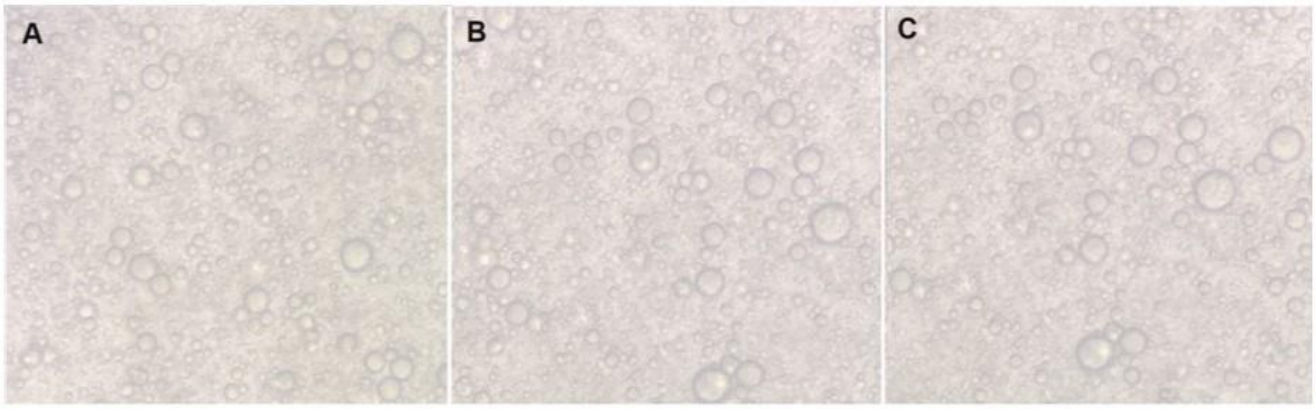

Figure 2. Morphology of the emulsion droplets prepared at different orange oil essential (OEsO) concentrations. A= 10% OEsO, B= 20% OEsO, C= 30% OEsO.

Table 2. Analysis of variance through a 3^3-factorial design for encapsulation and retention efficiencies of orange essential oil processed by spray drying.

Source of variation	Degree of freedom	Encapsulation efficiency				Retention efficiency			
		Sum of squares	Mean sum of squares	F-value	P-value	Sum of squares	Mean sum of squares	F-value	P-value
Main effects									
Orange essential oil concentration (A)	2	394.36	197.18	54124.91	0.0000	755.59	377.80	86582.14	0.0000
Temperature of emulsifying (B)	2	570.32	285.16	78274.64	0.0000	172.84	86.42	19805.02	0.0000
Ultrasound exposure time (C)	2	1476.88	738.44	202697.08	0.0000	334.86	167.43	38371.39	0.0000
Interactions									
A × B	4	569.01	142.25	39047.22	0.0000	127.45	31.86	7302.07	0.0000
A × C	4	148.46	37.12	10187.81	0.0000	76.90	19.23	4406.18	0.0000
B × C	4	1122.76	280.70	77047.71	0.0000	192.04	48.01	11002.47	0.0000
A × B × C	4	324.50	40.56	11134.09	0.0000	209.17	26.15	5992.05	0.0000
Residual	27	0.0984	0.0036			0.1178	0.0044		
Total (corrected)	53	4606.40				1868.97			

as their double and triple interactions were highly significant ($p < 0.05$) in the EnE.

Fig. 3 shows the effect of the levels of main factors used in the emulsion preparation on the EnE of OEsO encapsulated by spray drying. At 10% of OEsO, the EnE was 88%, while for 20% and 30% OEsO concentrations, the EnEs were 85.0% and 81.4%, respectively (Fig. 3A). Some studies have reported that when the oil concentration increases, a decrease in EnE occurs because the wall material is not enough to completely cover the oil droplets [42, 43].

When the emulsion preparation temperature increased from 25°C to 35°C the EnE decreased from 88.8% to 84.8.3%, and then to 80.5% at 55°C (Fig. 3B).

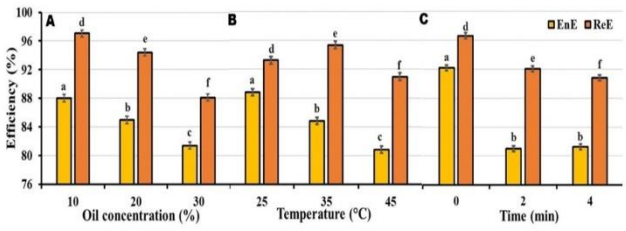

Figure 3. Effect of oil concentration (A), emulsification temperature (B), and ultrasound exposure time (C), as emulsion preparation factors, on the encapsulation efficiency (EnE) and retention efficiency (ReE) of orange essential oil processed by spray drying.

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The reduction in the EnE of emulsions prepared at high temperatures could be because under such conditions the viscosity decreases and collisions between the droplets increase, which weakens the mechanical strength of the interfacial film that surrounds the droplets and improves demulsification efficiency [44].

In the case of ultrasound exposure time, the EnE was reduced to 81% and 81.2% after the emulsions were treated for 2 min and 4 min, respectively, compared to value of 92.2% for those not ultrasonicated (Fig. 3C). Although it has been widely proven that the physical effects induced by ultrasonic cavitation increase the rupture of oil droplets, making them smaller and facilitating the formation of stable emulsions [45, 46], when ultrasound is applied to already formed emulsions, the effect that is achieved is demulsification [44], which could explain the reduction of the EnE.

The effect of the double interactions of the factors concerned in the preparation of the emulsions on the EnE of OEsO encapsulated by spray drying is shown in Table 3. Mostly, for each oil concentration, as the emulsification temperature increased, the EnE decreased. The largest reductions in EnE of 10.9% and 17.1% were observed when the emulsions were prepared at oil concentrations of 10% and 20%, respectively, and the emulsification temperatures were increased from 25°C to 55°C. C. However, the highest EnE values were achieved at 10% oil concentration and 25-35°C (98.38-91.15) and at 20% oil concentration at 25°C (94.59%).

The EnE of OEsOs encapsulated by spray drying by the combined effect of oil concentration, emulsifying temperature, and ultrasound exposure time is shown in Table 4. In most cases, the combined effect of the three factors involved in the emulsion preparation conditions presented a linear trend in the EnE. For example, the combined effect at 10% oil concentration and 25°C showed a linear trend for EnE at ultrasound exposure times of 0 min (94.77%), 2 min (93.65%) and 4 min (82.73%).

The range of values obtained for EnE of the triple interactions (Table 4) was 68.40% (55% oil concentration, 55°C emulsifying temperature, 4 min ultrasound exposure time) to 98.54% (10% oil concentration, 35°C emulsifying temperature, 0 min ultrasound exposure time). The maximum EnE (98.54%) obtained in this study was higher than those reached for OEsO encapsulation using spray drying by Aguiar et al. [47], Velazquez-Contreras et al. [48], and Rojas Moreno et al. [49] of 97.0%, 78%, and 48.89%, respectively, but lower than the 99.89% and 98.82% achieved by de Melo Ramos et al. [50] and Márquez-Gómez et al. [29]. On the other hand, an EnE value of 92.6% was reported for the microencapsulation of Persian lime peel essential oil by spray drying [51], which was lower than the value obtained for the OEsO in this study.

3.3 ReE of OEsO

ReE is the parameter that describes the essential oil maintenance in the capsule after spray drying. A high ReE value is desirable because it implies a minimization of the losses of EsOs after spray drying [35].

Table 2 shows the ANOVA of the results of the factors used in the preparation of the OEsO emulsions on ReE. Both the main factors (OEsO concentration, temperature of emulsifying, and ultrasound exposure time), as well as their double and triple interactions, were highly significant ($p < 0.05$) in the ReE.

Fig. 3 shows the impact of the levels of main factors employed in the emulsion preparation on ReE of OEsO encapsulated by spray drying. ReE had the same trend that presented EnE for the factors of OEsO concentration and time of exposure to ultrasound. At OEsO concentrations of 10%, 20% and 30%, ReEs of 97.0%, 94.4% and 88.1% (Fig. 1A) were obtained, respectively, while for ultrasound exposure times of 0 min, 2 min and 4 min, were 96.6%, 92.1%. % and 90.8% (Fig. 1C). The reduction in ReE at higher OEsO concentrations may be due to too few gum Arabic to form a sufficiently strong structural matrix surrounding the emulsion droplets [43], while ultrasonic treatment produced some rupture in the emulsions with the consequent decrease in the ReEs after spray drying [44].

The effect of the double interactions of the factors utilized in the preparation of the emulsions on ReE of the OEsO encapsulated by spray drying is shown in Table 3. ReE had the same behavior as EnE for the double interactions, showing the highest values when 10% oil concentration was at 35°C (99.20%), 10% oil concentration and 0 min time of ultrasound exposure

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Table 3. Effect of the double interactions of the emulsion preparation conditions on the encapsulation and retention efficiencies, moisture, and bulk density of orange essential oil processed by spray drying.

Interactions		Efficiency (%)		Moisture (%)	Bulk density (g/mL)
Oil (%)	Temperature (°C)	Encapsulation	Retention		
10	25	90.38 ± 0.023[b]	96.41 ± 0.026[b]	5.87 ± 0.01[a]	0.40 ± 0.001[b]
	35	91.15 ± 0.027[a]	99.20 ± 0.023[a]	5.16 ± 0.01[b]	0.42 ± 0.001[a]
	55	82.56 ± 0.031[c]	95.51 ± 0.024[c]	4.96 ± 0.02[c]	0.39 ± 0.002[c]
20	25	94.59 ± 0.020[a]	97.37 ± 0.025[a]	5.90 ± 0.01[a]	0.40 ± 0.001[b]
	35	82.00 ± 0.032[b]	95.38 ± 0.027[b]	5.33 ± 0.02[b]	0.41 ± 0.001[a]
	55	78.39 ± 0.033[c]	90.45 ± 0.028[c]	4.66 ± 0.02[c]	0.38 ± 0.002[c]
30	25	81.42 ± 0.031[a]	85.95 ± 0.029[c]	5.54 ± 0.01[b]	0.39 ± 0.001[b]
	35	81.27 ± 0.029[b]	91.46 ± 0.027[a]	5.63 ± 0.01[a]	0.43 ± 0.001[a]
	55	81.57 ± 0.030[c]	86.94 ± 0.031[b]	3.93 ± 0.02[c]	0.37 ± 0.002[c]
Oil (%)	**Ultrasound time (min)**				
10	0	96.77 ± 0.025[a]	98.76 ± 0.015[a]	5.43 ± 0.01[b]	0.41 ± 0.001[a]
	2	84.68 ± 0.028[b]	96.31 ± 0.020[b]	4.93 ± 0.01[c]	0.40 ± 0.001[b]
	4	82.64 ± 0.029[c]	96.04 ± 0.023[c]	5.65 ± 0.01[a]	0.40 ± 0.001[b]
20	0	89.75 ± 0.026[a]	97.42 ± 0.021[a]	5.44 ± 0.01[b]	0.39 ± 0.001[b]
	2	80.79 ± 0.033[c]	94.11 ± 0.028[b]	4.92 ± 0.01[c]	0.41 ± 0.001[a]
	4	84.45 ± 0.028[b]	91.66 ± 0.031[c]	5.53 ± 0.01[a]	0.38 ± 0.002[c]
30	0	90.11 ± 0.025[a]	93.71 ± 0.033[a]	5.00 ± 0.01[b]	0.40 ± 0.001[a]
	2	77.50 ± 0.038[b]	85.83 ± 0.042[b]	4.96 ± 0.01[c]	0.39 ± 0.001[b]
	4	76.65 ± 0.039[c]	84.81 ± 0.041[c]	5.14 ± 0.01[a]	0.40 ± 0.001[a]
Temperature (°C)	**Ultrasound time (min)**				
25	0	90.38 ± 0.023[b]	96.41 ± 0.021[a]	5.48 ± 0.01[c]	0.40 ± 0.001[a]
	2	91.46 ± 0.021[a]	94.79 ± 0.023[b]	5.75 ± 0.01[b]	0.39 ± 0.002[b]
	4	84.55 ± 0.035[c]	88.53 ± 0.025[c]	6.07 ± 0.01[a]	0.39 ± 0.002[b]
35	0	94.38 ± 0.019[a]	96.87 ± 0.022[a]	5.27 ± 0.01[b]	0.42 ± 0.001[b]
	2	73.89 ± 0.042[c]	93.76 ± 0.029[c]	4.96 ± 0.01[c]	0.43 ± 0.001[a]
	4	86.16 ± 0.033[b]	95.42 ± 0.024[b]	5.88 ± 0.01[a]	0.41 ± 0.001[c]
55	0	91.86 ± 0.022[a]	96.62 ± 0.022[a]	5.10 ± 0.01[a]	0.34 ± 0.002[c]
	2	77.62 ± 0.038[b]	87.71 ± 0.028[c]	4.10 ± 0.02[c]	0.38 ± 0.001[b]
	4	73.03 ± 0.041[c]	88.57 ± 0.027[b]	4.36 ± 0.02[b]	0.39 ± 0.001[a]

Values are the mean ± standard deviation of two repetitions. Means of the interactions: Oil concentration × Temperature, Oil concentration × Ultrasound time, and Temperature × Ultrasound time, with distinct lowercase letters for Temperature (25°C, 35°C, and 55°C), Ultrasound time (0 min, 2 min, and 4 min), and Ultrasound time (0 min, 2 min, and 4 min), respectively, are significantly different ($p < 0.05$).

(98.76%), and 35°C and 0 min of ultrasound exposure time (96.87%) were used as emulsion preparation conditions.

The ReE of the OEsOs encapsulated by spray drying due to the triple interactions is shown in Table 4. In most cases, the combined effect of the three factors involved in the emulsion preparation conditions presented a quadratic trend for ReE. For instance, at 20% oil concentration and 35°C a quadratic trend for ReE was observed for ultrasound exposure times of 0 min (99.32%), 2 min (92.67%) and 4 min (94.15%).

ReE of OEsOs of this study ranged from 79.80% (30% oil concentration, 55°C emulsifying temperature, 4 min ultrasound exposure time) to 99.80% (10% oil

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Table 4. Effect of emulsion preparation conditions on encapsulation and retention efficiencies, humidity and bulk density of orange essential oil processed by spray drying.

Emulsion preparation conditions			Encapsulation efficiency (%)	Retention efficiency (%)	Moisture (%)	Bulk density (g/mL)
Oil (%)	Temperature (°C)	Ultrasound exposure time (min)				
10	25	0	94.77 ± 0.067^{r}	99.80 ± 0.067^{s}	6.16 ± 0.004^{q}	0.418 ± 0.0003^{p}
		2	93.65 ± 0.066^{p}	99.00 ± 0.066^{o}	5.16 ± 0.004^{i}	0.385 ± 0.0003^{f}
		4	82.73 ± 0.059^{i}	90.42 ± 0.059^{h}	6.30 ± 0.004^{r}	0.394 ± 0.0003^{j}
	35	0	98.54 ± 0.070^{u}	98.54 ± 0.070^{ñ}	5.00 ± 0.004^{g}	0.430 ± 0.0003^{s}
		2	83.62 ± 0.059^{k}	99.91 ± 0.059^{s}	4.83 ± 0.003^{f}	0.431 ± 0.0003^{t}
		4	91.29 ± 0.065^{m}	99.14 ± 0.065^{p}	5.66 ± 0.004^{l}	0.401 ± 0.0003^{l}
	55	0	96.99 ± 0.069^{s}	97.95 ± 0.069^{n}	5.10 ± 0.004^{h}	0.381 ± 0.0003^{e}
		2	76.78 ± 0.054^{e}	90.03 ± 0.054^{g}	4.80 ± 0.004^{e}	0.388 ± 0.0003^{g}
		4	73.90 ± 0.052^{d}	98.54 ± 0.052^{ñ}	5.00 ± 0.004^{g}	0.404 ± 0.0003^{m}
20	25	0	93.65 ± 0.066^{p}	99.00 ± 0.066^{o}	5.50 ± 0.004^{k}	0.385 ± 0.0003^{f}
		2	96.94 ± 0.069^{s}	99.64 ± 0.069^{r}	6.10 ± 0.004^{p}	0.421 ± 0.0003^{q}
		4	93.17 ± 0.066^{o}	93.46 ± 0.066^{k}	6.10 ± 0.004^{p}	0.390 ± 0.0003^{h}
	35	0	93.98 ± 0.066^{q}	99.32 ± 0.066^{q}	5.83 ± 0.004^{m}	0.410 ± 0.0003^{m}
		2	68.64 ± 0.049^{e}	92.67 ± 0.049^{i}	4.16 ± 0.003^{c}	0.431 ± 0.0003^{t}
		4	83.39 ± 0.059^{j}	94.15 ± 0.059^{m}	6.00 ± 0.004^{ñ}	0.392 ± 0.0003^{i}
	55	0	81.61 ± 0.058^{h}	93.95 ± 0.058^{l}	5.00 ± 0.004^{g}	0.377 ± 0.0003^{b}
		2	76.78 ± 0.054^{e}	90.03 ± 0.054^{g}	4.50 ± 0.003^{c}	0.388 ± 0.0003^{g}
		4	76.78 ± 0.054^{e}	87.38 ± 0.054^{e}	4.50 ± 0.003^{c}	0.378 ± 0.0003^{c}
30	25	0	82.73 ± 0.059^{i}	90.42 ± 0.059^{h}	4.80 ± 0.003^{d}	0.394 ± 0.0003^{j}
		2	83.79 ± 0.059^{l}	85.74 ± 0.059^{d}	6.00 ± 0.004^{o}	0.379 ± 0.0003^{d}
		4	77.75 ± 0.055^{f}	81.69 ± 0.055^{b}	5.83 ± 0.004^{m}	0.401 ± 0.0003^{k}
	35	0	90.61 ± 0.064^{m}	92.75 ± 0.064^{i}	5.00 ± 0.004^{g}	0.412 ± 0.0003^{m}
		2	69.41 ± 0.049^{c}	88.69 ± 0.049^{f}	5.90 ± 0.004^{n}	0.441 ± 0.0003^{u}
		4	83.79 ± 0.059^{l}	92.95 ± 0.059^{j}	6.00 ± 0.004^{o}	0.423 ± 0.0003^{r}
	55	0	96.99 ± 0.069^{t}	97.95 ± 0.069^{n}	5.20 ± 0.004^{j}	0.381 ± 0.0003^{e}
		2	79.31 ± 0.056^{g}	83.08 ± 0.056^{c}	3.00 ± 0.002^{a}	0.358 ± 0.0003^{a}
		4	68.40 ± 0.048^{a}	79.80 ± 0.048^{a}	3.60 ± 0.003^{b}	0.378 ± 0.0003^{c}

Values are the mean ± standard deviation of two repetitions. Means with different lowercase letters in the same column are significantly different ($p \leq 0.05$)

concentration, 25°C emulsifying temperature, 4 min ultrasound exposure time). The best value of ReE (Table 3) was higher than those reported by de Souza et al. [28], Flores Martínez et al. [52], Velazquez Contreras et al. [48], Rojas Moreno et al. [49], and de Melo Ramos et al. [50] of 81.30% 97.54%, 72%, 48.49%, and 97.60%, respectively. However, Lopes Francisco et al. [30] reported ReE of up to 100% for encapsulation of OEsO. In contrast, Bajac et al. [53] only obtained a ReE value of 84.67% for microencapsulation of juniper berry essential oil by spray drying using gum arabic as a carrier.

According to Nguyen et al. [27], the encapsulation efficiency of EOs by spray drying is dependent on several factors, such as the type and concentration of the encapsulating agents, the EsO concentration and its properties, the characteristics of the feed emulsion, and the drying conditions. However, when two or more parameters are considered to determine encapsulation efficiency, the best conditions for one of them may be different from another [54]. In this study, the conditions that simultaneously favored high values of EnE and ReE were oil concentration 10%, emulsification temperature 35°C and ultrasound exposure time 0 min, giving a value of 98.54% for both indicators.

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Table 5. Analysis of variance through a 3^3-factorial design for moisture and density of orange essential oil processed by spray drying.

Source of variation	Degree of freedom	Moisture				Bulk density			
		Sum of squares	Mean sum of squares	F-value	P-value	Sum of squares	Mean sum of squares	F-value	P-value
Main effects									
Orange essential oil concentration (A)	2	0.9512	0.4756	34063.41	0.0000	5.8×10^{-4}	2.9×10^{-4}	3609.65	0.0000
Temperature of emulsifying (B)	2	14.673	7.3367	525456.37	0.0000	0.0129	0.0065	80874.03	0.0000
Ultrasound exposure time (C)	2	2.3999	1.1999	85940.78	0.0000	4.2×10^{-4}	2.1×10^{-4}	2611.69	0.0000
Interactions									
A × B	4	3.5903	0.8975	64285.46	0.0000	3.4×10^{-4}	3.4×10^{-4}	4201.69	0.0000
A × C	4	0.6545	0.1636	11720.43	0.0000	2.6×10^{-4}	6.5×10^{-4}	8106.11	0.0000
B × C	4	4.5041	1.1260	80646.20	0.0000	0.0025	6.1×10^{-4}	7702.19	0.0000
A × B × C	4	7.8564	0.9820	70335.28	0.0000	0.0025	3.2×10^{-4}	3952.08	0.0000
Residual	27	0.0003	1.4×10^{-5}			2.2×10^{-4}	7.9×10^{-8}		
Total (corrected)	53	34.6305				0.0228			

3.4 Moisture content of encapsulated OEsO

The moisture content of encapsulated essential oils is a very important criterion that clouts their handling properties, as well as their oxidative and microbial stability [55], which is achieved at values in the range of 4-6% and thus long-term storage [35, 40, 53].

Table 5 shows the ANOVA of the results of the factors involved in the preparation of the OEsO emulsions on moisture content. The main factors (OEsO concentration, temperature of emulsifying, and ultrasound exposure time), as well as their double and triple interactions, were highly significant ($p < 0.05$) for moisture content.

The levels of the main factors used in the preparation of the emulsion significantly influenced ($p < 0.05$) the moisture content of the OEsO encapsulated by spray drying (Fig. 4). As the oil concentration and emulsification temperature increased, the moisture content decreased significantly ($p < 0.05$). The decrease in moisture content was to values of 5.29% and 5.03% for oil concentrations of 20% and 30%, respectively, from a value of 5.33% for oil concentration of 10% (Fig. 4A). When the emulsification temperature increased from 25°C to 35°C, the moisture content varied from 5.77% to 5.37%, respectively, and then to 4.52% for 55°C (Fig. 4B). In the case of ultrasonication time, the moisture content

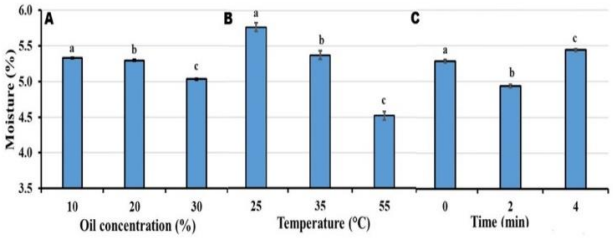

Figure 4. Effect of oil concentration (A), emulsification temperature (B), and ultrasound exposure time (C), as emulsion preparation factors, on the moisture content of orange essential oil processed by spray drying.

first decreased, but then increased with respect to 0 min (Fig. 4C). Specifically, for 0 min of ultrasonication, the moisture content was 5.29%, while for 2 min and 4 min of ultrasonication, the moisture contents were 4.93% and 5.44%, respectively.

In studies with flaxseed [42] and coffee [56] oils encapsulated by spray drying using gum Arabic as a carrier, the higher the concentration of the oil in the emulsion, the lower the moisture content in the product, possibly due to the small amount of water that had to be eliminated, which agrees with the results of this work.

The effect of double interactions of emulsion preparation factors on the moisture content of OEsO encapsulated by spray drying is shown in Table 3. In general, for each oil concentration, as the

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emulsification temperature rose, the moisture content decreased. The reductions in moisture content at oil concentrations of 10%, 20% and 30% when the temperature increased from 25°C to 55°C were 15.5%, 21.0% and 29.0%, respectively. In the case of the oil concentration-ultrasound exposure time interaction, for each oil concentration, the moisture content decreased at the ultrasonication time of 2 min, but then increased when it was 4 min, compared to 0 min. The increases in moisture content were 4.0%, 1.7%, 2.8% at oil concentrations of 10%, 20% and 30%, respectively, when the ultrasonication time varied from 0 min to 4 min. In the emulsion temperature-ultrasonic exposure time interaction, at 25°C moisture content rose as the ultrasonication time boosted, but a 55°C the opposite was observed, compared to 0 min of ultrasonication. However, at 35°C moisture content first decreased and then augmented as the ultrasonication time rose.

The maximum moisture content of 6.07% by double interactions was obtained for 25°C as the emulsifying temperature and 4 min of ultrasonication time, while the minimum of 3.93% was for 30% as oil concentration and 55°C corresponding to the emulsifying temperature (Table 3). In the case of the triple interactions, the range of moisture content was 3.00-6.30%, with the lowest value corresponding to 30 %, 55°C and 2 min as oil concentration, emulsifying temperature, and ultrasonication time, respectively, while the highest to 10%, 25°C, and 4 min (Table 4).

Atli et al. [57] reported values of moisture of 3.17-5.24% for capsules of cumin (*Cuminum cyminum* L.) seed essential oil by spray drying, using concentrations of 10-20% of cumin oil in the emulsions, and concentrations of 1-3% and 25-35% of chickpea protein isolate and maltodextrin as carriers, respectively. The values of moisture content for capsules of *Zanthoxylum schinifolium* essential oil through emulsification followed by spray drying, using concentrations of maltodextrins of 7-15% were 4.25-5.99% [39]. In the case of orange essential oil microencapsulated by spray drying using maltodextrin as wall material, Nguyen et al. [54] found moisture values of 2.43-5.03%.

3.5 Bulk density of encapsulated OEsO

The apparent density of food powders is a characteristic that has economic and functional implications in their packaging, distribution, and storage [58]. A high apparent density is desirable because it favors a lower package volume, limits the amount of air between the particles, thus reducing the possibility of product oxidation and increasing storage stability [39].

Table 5 shows the ANOVA of the results of the factors involved in the preparation of the OEsO emulsions on bulk density. The main factors (OEsO concentration, temperature of emulsifying, and ultrasound exposure time), as well as their double and triple interactions were highly significant ($p < 0.05$) for bulk density.

Fig. 5 shows the effect of the levels of the main factors used in the preparation of the emulsion on the apparent density of the OEsO encapsulated by spray drying.

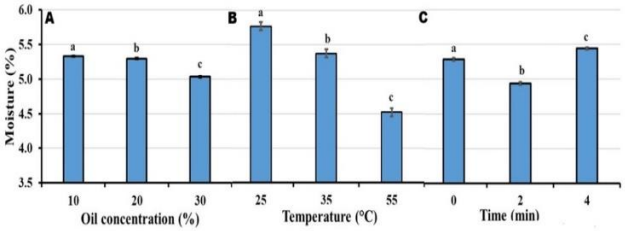

Figure 5. Effect of oil concentration (A), emulsification temperature (B), and ultrasound exposure time (C), as emulsion preparation factors, on the bulk density of orange essential oil processed by spray drying.

The apparent density increased significantly ($p < 0.05$) as the oil concentration augmented (Fig. 5A), while for the emulsification temperature and ultrasonication time, the apparent density rose significantly ($p < 0.05$) at medium level and then diminished significantly ($p < 0.05$) at the high level (Figs. 5A, 5B). The augment in the bulk density with the increase of oil concentration also was observed by Tonon et al. [42] for flaxseed oil encapsulated by spray drying using whey protein concentrate, gum Arabic, and modified starch as carriers.

In general, the behavior of the apparent density by double interactions was quadratic since at the medium level of each factor, the apparent density increased or decreased with respect to the low level, and at the high level, it increased or decreased with respect to the medium level. For example, at 10% oil concentration, the bulk density increased when the emulsification temperature went from 25°C to 35°C,

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but then decreased at 55°C (Table 3). The range of the bulk density by double interactions was 0.34 g/mL to 0.43 g/mL, corresponding to 55°C as emulsifying temperature and 0 min of ultrasonication, and 30% oil concentration and 35°C as emulsifying temperature, respectively. However, the influence of the 3 factors used in the preparation of the emulsion modified the apparent density range values of the OEsO encapsulated by spray drying to 0.358 g/mL to 0.441 g/mL, belonging to the conditions of oil concentration, emulsifying temperature and ultrasonication time of 30%, 55°C, 2 min and 30%, 35°C, and 2 min, respectively (Table 4).

The values of bulk density of the encapsulated OEsOs of this study fall in the range of those obtained for rosemary [59], hop [60], and fennel [61] essential oils, which were 0.25-0.37 g/mL, 0.29 g/mL, and 0.26-0.59 g/mL, respectively.

4. Conclusions

The oil concentration, the emulsification temperature and the ultrasound exposure time, as factors for preparing the emulsion to be encapsulated by spray drying, significantly influenced ($p < 0.05$) the EnE, the ReE, the content of humidity, and the apparent density of OEsO microcapsules. The double and triple interactions of the used factors in the preparation of the emulsions also significantly influenced ($p < 0.05$) the encapsulation quality by spray drying of the OEsO. The maximum EEn (98.54%) and ReF (99.80%) values of the encapsulated OESO were not obtained under the same emulsion preparation conditions. However, by analyzing the results of the treatments obtained through experimental design 3^3, the best conditions (orange oil concentration 10%, emulsifying temperature 35°C, and ultrasound exposure time 0 min) were determined simultaneously for EEn (98.54%) or ReE (98.54%). Furthermore, the emulsification conditions that simultaneously reached the best EEn and ReE values of the encapsulated OEsO, also had the moisture content (5.0%) and apparent density (0.430 g/mL) that ensured both its adequate stability during storage and handling. According to the results of this study, the conditions of emulsion impacted significantly ($p < 0.05$) the encapsulation quality of the OEsO by spray drying, which could help to improve the industrial process of this product. Further research is needed to determine what other factors in emulsion preparation might influence both encapsulation and product quality, including the use of various emerging technologies.

Authors' contributions

Conceptualization, B.E.U.R; Methodology, B.E.U.R.; Investigation, B.E.U.R., Writing original draft preparation, B.E.U.R.; Resources, B.E.U.R, J.A.U., Formal analysis, J.A.U., P.R.U.; Writing-review and editing, J.A.U., P.R.U.; Supervision, J.A.U.

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Availability of data and materials

The datasets used and/or analyzed during the current study will be available from the corresponding author on reasonable request, according to the journal policy.

Conflicts of interest

The authors declare that they have no known competing economic interests or personal relationships that could have appeared to influence the work reported in this manuscript.

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