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RESEARCH ARTICLE

Influence of emulsification methods and spray drying parameters on the microencapsulation of turmeric oleoresin

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ABSTRACT

Turmeric (*Curcuma longa* L.) oleoresin possess valuable phenolic compounds that are susceptible to degradation, and microencapsulation is a powerful technique to increase its stability. Emulsification is a preponderant step in microencapsulation of hydrophobic compounds and physical-chemical properties of the parent emulsion affects effectiveness of spray-drying process and functional properties of the produced microcapsules. The present work aimed to evaluate the influence of emulsion formulation, emulsification methods, and spray-drying operational conditions on the encapsulation efficiency of turmeric oleoresin using maltodextrin/gelatin blends as wall material. The effects of different concentrations of maltodextrin (12 - 31.7 wt %) and gelatin (0.6 - 6 wt %), combined with three methods of emulsification - high shear homogenization with and without emulsifier addition, and sonication – were evaluated regarding emulsion droplet mean diameter and stability. Based on the results, an emulsion formulated with 26 g of maltodextrin and 0.6 g of gelatin per 100 g of emulsion was selected to study the influence of spray drying conditions - drying-air temperature (124 – 190°C), atomization airflow (275 – 536 L h^{-1}), and emulsion feeding flow (1.4 – 8.6 mL min^{-1}) - on encapsulation efficiency, water content, and solubility of turmeric oleoresin microcapsules. Sonication resulted in higher emulsion stability and, although drying-air temperature did not affect significantly the microcapsule properties, the best set of spray drying conditions was drying-air at 160 °C, atomization airflow of 420 L h^{-1}, and emulsion feeding flow of 6 mL min^{-1}. Combinations of higher atomization airflow and lower emulsion feeding flow resulted in lower values of curcumin encapsulation efficiency.

Keywords: Encapsulation; Emulsion; Maltodextrin; Gelatin; Ultrasound; Rheology; Curcumin

INTRODUCTION

Microencapsulation is a process in which a membrane encloses tiny particles of solid, liquid, or gas, forming microcapsules (Shahidi And Han, 1993; Jyothi et al., 2012). This technique is applied mainly to protect high-value core materials from harsh conditions such as light, moisture, oxygen, and interactions with other compounds (Gouin, 2004; Tonon et al., 2011). In addition, microencapsulation may improve characteristics of the final product, such as stability, solubility, and increased product lifespan, as well as promoting controlled release of the encapsulated material under specific conditions (Malacrida et al., 2013; Pereira et al., 2018). Turmeric (*Curcuma longa* L.) is a plant of the family *Zingiberaceae*, original from South India (Almeida, 2006, Nayaka et al., 2013), with proved bioactivity, including antioxidant, antimicrobial, anti-inflammatory, and anticarcinogenic properties (Govindarajan and Stahl, 1980; Jayaprakasha et al., 2005; Wang et al., 2015). Microencapsulation has been applied as a feasible alternative to facilitate dispersion and solubilization, as well as increasing stability of turmeric oleoresin and curcumin (Cano-Higuita et al., 2015a; Ferreira et al., 2016; Zuanon et al., 2017).

Most of the microencapsulation processes involves dispersion or emulsification of the active material in the wall material, followed by subsequent drying of the microcapsules (Shahidi and Han, 1993; Soottitantawat et al., 2005). In the case of hydrophobic compounds, such as turmeric oleoresin, effective microencapsulation demands that the emulsion be stable throughout the process. Emulsion stability, which is related

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to size distribution of dispersed droplets and rheological properties of the continuous phase, can be expressed using the creaming index (CI), with higher CI values indicating lower emulsion stability (Keowmaneechai and McClements, 2002; Albano et al., 2018). Achieving the smallest possible emulsion droplets, as well as attaining sufficiently high viscosity in the continuous phase contribute directly to increasing emulsion stability and encapsulation efficiency of the core material at the subsequent drying process (Sosa et al., 2014; Ferreira et al., 2016). The main methods of emulsification include high-shear mixing, high-pressure homogenization or microfluidization, membrane homogenization, and sonication (Abbas et al., 2013; Morelli et al., 2017). Sonication is expected to produce smaller droplets than high-shear homogenization due to local perturbations at the interface and because bubble implosion is much faster than its dilation (Canselier et al., 2002; McClements, 2007; Gaikwad and Pandit, 2008; Sosa et al., 2014; Zuanon et al., 2017). Ultrasound emulsification occurs mainly due to cavitation that causes mixing and/or disruption of the system, through a phenomenon by which bubbles collapse at or near the oil–water interface. Ultrasound emulsification is achieved at low frequencies (16-100 kHz), which cause stronger shear forces inducing the bubbles to collapse, and leads to production of stable emulsions in the absence or with only small addition of surfactants, with small and uniform droplets, and with lower energy input (Mongenot et al., 2000; Lertsutthiwong et al., 2009; Chandrapala et al., 2012; Shanmugam et al., 2014; Ferreira et al., 2016).

The drying process is a critical step on the microencapsulation process (Malacrida et al., 2013). Spray drying allows encapsulation because the increased superficial area resulting from atomization and the high temperature of the drying air allow the process to occur in a short period. In such a way, the space occupied by water before evaporation becomes empty and creates a low-pressure micro zone, causing the wall material to collapse, entrapping the core material in a spherical particle and reducing its contact area with the external medium (Zbicinski et al., 2002; Schmitz-Schug et al., 2016). Materials suitable to be used as encapsulating matrices in spray drying are generally required to present low viscosity at high concentrations, low hygroscopicity, high solubility, and low cost, which are the major attributes of maltodextrins (Akhavan Mahdavi et al., 2016; Pereira et al., 2018). In addition, when the core material is a hydrophobic compound, surface activity and emulsification ability become important as well, and proteins such as gelatin can fulfill these requisites (Dib Taxi et al., 2003). In a previous study, Cano-Higuita et al. (2015b) encapsulated turmeric oleoresin by spray drying using binary and ternary blends of gum Arabic, maltodextrin and modified starch and obtained encapsulation efficiencies that varied from 8 to 46%. Using modified starch and gelatin as wall material, Malacrida et al., (2015) encapsulated turmeric oleoresin by freeze-drying and obtained encapsulation efficiencies ranging from 64 to 92 %.

Based on the relevance of emulsion properties to effective microencapsulation of hydrophobic compounds, the objective of this work was to evaluate the influence of emulsion formulation and emulsification methods on droplet mean diameter and stability of turmeric oleoresin emulsions stabilized by maltodextrin/gelatin blends. In general, papers on microencapsulation of turmeric oleoresin by spray drying study the influence of the emulsion formulation of the microencapsulation parameters. Furthermore, the influence of spray drying conditions of the parent emulsion, including drying-air temperature, atomization airflow, and emulsion feeding flow, was investigated on the encapsulation efficiency, water content, and solubility of turmeric oleoresin microencapsulated in maltodextrin/gelatin wall material.

MATERIAL AND METHODS

Material

The encapsulated material was turmeric oleoresin OS-50 (Agro-Industrial Olímpia Ltda., Brazil). The wall materials were maltodextrin DE 10 (Corn Products, Brazil) and bovine gelatin bloom value 240 (Gelita, Brazil). Polyoxyethylene sorbitan monooleate - Tween 80 (Labsynth, Brazil) was used as surfactant.

Formulation and homogenization of emulsions

The adopted blends of wall materials were: 26/0.6, 24/6, 31.7/0.6, 12/6, 31.7/6, and 22/3 g of maltodextrin/gelatin, respectively, per 100 g of emulsion. Turmeric oleoresin was added before homogenization in a ratio of 15 g of oleoresin per 100 g of wall material dry mass (15 wt%). Emulsions were prepared accordingly three different procedures: using high-shear homogenization (ultra turrax, T - 25, IKA, Germany), with and without addition of emulsifier Tween 80 (1 g 100 g^{-1} of total dry mass), and using an ultrasonic probe (Sonic Ruptor 4000, Omni International, USA), following the procedure described by Ferreira et al. (2016).

Emulsion stability and droplet size

After homogenization, emulsions samples (50 mL) were transferred to transparent, graduated, plastic tubes and centrifuged at 50.2×g (CT-D 5500, Brazil) for 5 minutes. Creaming index (CI) was determined by the ratio between the height of the bottom phase (H) and the initial height of emulsion in the tube (H_0), according to Equation 1 (Keowmaneechai and Mcclements, 2002; Perrechil and Cunha, 2010).

$$CI = \frac{H}{H_o} \quad (1)$$

To evaluate the average droplet size, 0.1 mL of the emulsion was placed on a glass slide, covered with a coverslip, and observed using an optical microscope (L2000, Bioval, Brazil) coupled with a video camera (Borgognoni et al., 2006; Perrechil and Cunha, 2010). The scanned images were analysed using Image Pro Plus 6.0 software (Media Cybernetics Inc., USA). Average diameter was determined based on the measurement of 30 droplets.

Rheological behaviour of the encapsulating materials

Rheological measurements were carried out in the dispersions of encapsulating materials, without addition of turmeric oleoresin, using an oscillatory rheometer AR2000ex (TA Instruments, Delaware, USA) with parallel plate geometry and gap of 800 μm. Samples (1.8 mL) were introduced in the rheometer and left to rest for 5 minutes before measurements, in order to equilibrate the test temperature (40 °C). Temperature sweeps were performed between 40 and 20 °C at a cooling rate of 2 °C min^{-1}. The storage and dissipation moduli, G' and G", were recorded as a function of time at frequency of 0.1 Hz and strain rate of 0.05 (Firoozmand et al., 2007). At the end of the temperature sweep, the sample was left to stand in the rheometer at 20 °C for 1 hr to allow for structural arrangement. Then, a frequency sweep was performed from 0.01 to 100 Hz, at 20 °C and strain rate of 0.05.

Spray drying

The emulsion formulated with 26/0.6 g of maltodextrin/gelatin per 100 g (26M/0.6G) and produced by ultrasound homogenization was spray dried in a mini spray dryer (B-290, Büchi, Switzerland) equipped with a 0.7 mm diameter two-fluid nozzle. The studied operational conditions (and respective range of variation) were: drying-air temperature (124 to 190°C), atomization airflow (275 to 536 L h^{-1}) and emulsion feeding flow (1.4 to 8.6 mL min^{-1}), following a central composite rotational design. The aspirator rate was fixed at 90% and the emulsions were kept under magnetic stirring at a constant temperature (40°C) while being fed to the atomization nozzle.

Encapsulation efficiency, water content and solubility

Encapsulation efficiency EE (%) was expressed in terms of curcumin retention. The total curcumin content was determined following the method described by Chauhan et al. (1999) and Marfil et al. (2012). Briefly, a solution of turmeric oleoresin (0.01 mg mL^{-1}) in methanol was analysed for curcumin content by measuring the absorbance at 425 nm (SP-22, Biospectro, Brazil). Ten milligrams of microcapsules were taken in a 25 mL standard volumetric flask and the volume was completed using methanol. The solution was homogenized in a vortex for 5 min, followed by centrifugation at 704×g for 10 min. The curcumin content in the supernatant was quantified by measuring the absorbance at 425 nm. Curcumin retention in the microcapsules was calculated as:

$$EE\% = \frac{C_F}{C_o} * 100 \quad (2)$$

in which C_F is curcumin concentration (mg g^{-1}) in the spray dried powder and C_0 is curcumin concentration (mg g^{-1}) in the emulsion before spray drying.

Powder solubility was determined according to the method used by Cano-Chauca et al. (2005) and water content of the encapsulated material after spray-drying was determined gravimetrically in an oven at 105 °C for 6 hours (AOAC, 1997).

Capsule morphology

Capsule morphology was evaluated by scanning electron microscopy (SEM). The powders were attached to an adhesive tape mounted on SEM stubs, coated with 3–5 mA gold/palladium under vacuum and examined with a Scanning Electron Microscope - SEM (Zeiss, model 960, Germany), operated at 20 kV and with magnification of 1000×.

Statistical analysis

The results were expressed as arithmetic means with respective standard deviation, subjected to analysis of variance (ANOVA) and comparison of the means using Tukey's test at 5 % probability. Correlation between powder properties and spray drying operational parameters was analysed based on the response surface methodology using Minitab 17 Statistical Software (MINITAB, State College - PA, USA).

RESULTS AND DISCUSSION

Rheological analysis of maltodextrin/gelatin blends

Total solid content, the ratio between biopolymers, and their chemical properties generally affect rheology of hydrocolloid dispersions and, consequently, the stability of emulsions formulated with these dispersions (Djabourov, 1988; Tesch and Schubert, 2002). Thus, it is crucial to find the proper balance between biopolymer contents in order to achieve a stable microstructure that will contribute to the microencapsulation process (Carneiro et al., 2013).

The mechanical spectra (Fig. 1) and temperature sweeps (Fig. 2) obtained for the maltodextrin/gelatin blends evaluated as wall materials for turmeric oleoresin

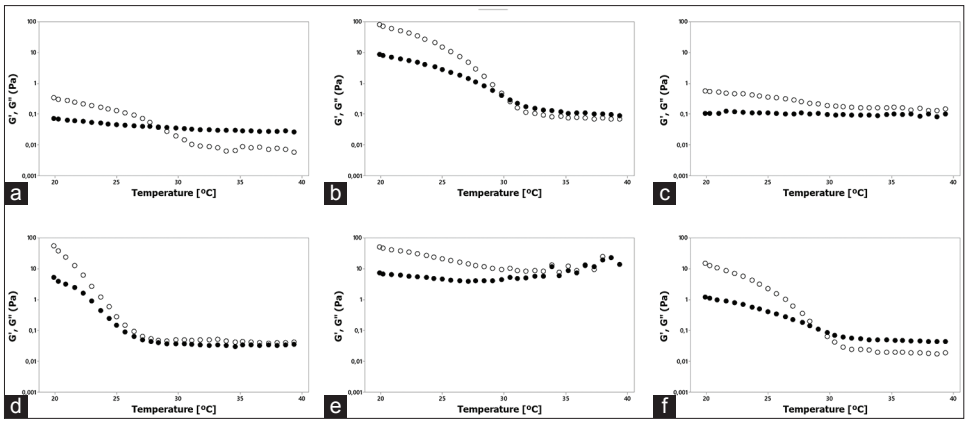

Fig 1. Temperature dependence of G' (non-filled symbols) and G" (filled symbols) of the encapsulant dispersions at different formulations: (a) 26/0.6; (b) 24/6; (c) 31.7/0.6; (d) 12/6; (e) 31.7/6; and (f) 22/3 (g 100 g-1) of maltodextrin/gelatin, respectively.

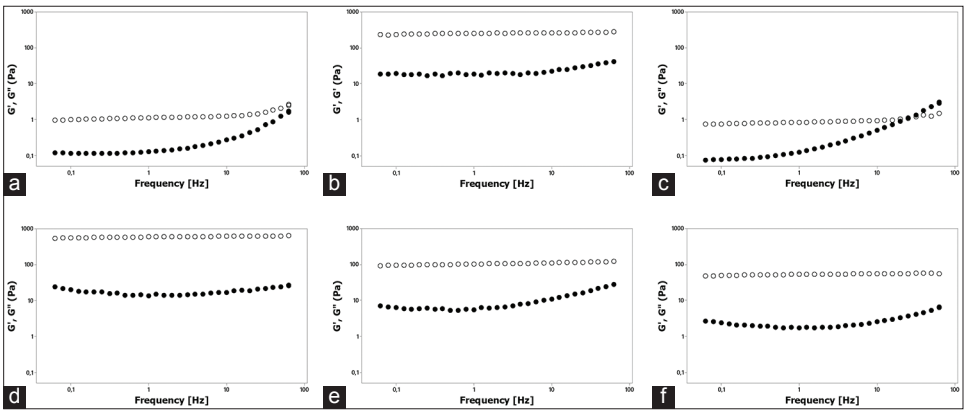

Fig 2. Frequency dependence of G' (non-filled symbols) and G" (filled symbols) of the encapsulant dispersions at different formulations: (a) 26/0.6; (b) 24/6; (c) 31.7/0.6; (d) 12/6; (e) 31.7/6; and (f) 22/3 (g 100 g-1) of maltodextrin/gelatin, respectively.

microencapsulation showed that gelatin concentration played an important role on the formation of a gel with viscoelastic properties that can improve emulsion stability (Dickinson, 2006; Dickinson, 2009). The systems 26M/0.6G and 31.7M/0.6G, which presented the smallest content of gelatin in the blends (sample codes and respective compositions are indicated in Table 1), also presented the lowest values of storage (G') and loss (G") modulus in the mechanical spectra among all the formulations studied (Fig. 1a and 1c). This indicates that the system will flow easily at the nozzle during spray drying, facilitating the atomization process (Cano-Chauca et al., 2005). The sample 26M/0.6G presented T^{gel} = 28°C - i.e. the temperature at which G' = G" in the temperature scan (Fig. 1) - and G^{gel} ~ 0.01 Pa, which is the value of storage and loss modulus at T^{gel}. This behaviour emphasizes the role of gelatin in the formation of triple helix structures (Dib Taxi et al., 2003). Concerning the blend 31.7M/0.6G (Fig. 1c), it was not possible to clearly determine the G^{gel} modulus. Also, magnitudes of G' and G" were slightly higher for 26M/0.6G in comparison to sample 31.7M/0.6G (Fig. 1a, c, and 2a, c). This may be a consequence of the lower proportion of gelatin present in the system 31.7M/0.6G. Possibly, maltodextrin molecules may act as steric spacers and inhibit protein aggregation (Ibanoglu, 2005). This behaviour is in accordance with the results of Nicoleti and Telis (2009) that observed a decrease in gel strength with the increase in total biopolymer concentration (maltodextrin and collagen blends).

As can be seen in Fig. 1d, gelatin prevails to structure formation in sample 12M/6G, in which maltodextrin/gelatin proportion is 2/1. This formulation presented the higher T^{gel} value (> 40°C), indicating that the system is able to store more energy in higher temperatures without flowing. In addition, it had a constant value of G^{gel} (~0.01 Pa), which may indicate that its structure could support stress conditions without destabilizing, improving emulsion stability. Fig. 1 shows a trend that in some cases the total solid content balances the polymer ratio, taking into consideration the values of G^{gel} and T^{gel}. This is true for samples 26M/0.6G (Fig. 1a) and 22M/3G that presented the same T^{gel} (~28°C) with different polymer ratios, whereas sample 31.7M/6G presented the highest G^{gel} (10 Pa) (Fig. 1e), due to the combination of high solid content and high gelatin concentration.

Table 1: Creaming index (CI) of turmeric oleoresin emulsions stabilized by maltodextrin/gelatin blends produced with different homogenization methods*

Sample	Maltodextrin (g 100 g^{-1})	Gelatin (g 100 g^{-1})	Creaming index (CI)[1]		
			Rotor-stator homogenization	Rotor-stator homogenization+Tween 80	Ultrasound homogenization
12M/6G	12.0	6.0	0.09±0.01^{fg}	0.84±0.01^{a}	0.05±0.01^{g}
22M/3G	22.0	3.0	0.59±0.02^{c}	0.57±0.05^{c}	0.81±0.12^{ab}
24M/6G	24.0	6.0	0.26±0.02d^{ef}	0.16±0.01^{efg}	0.30±0.06^{de}
26M/0.6G	26.0	0.6	0.35±0.03^{d}	0.66±0.01^{bc}	0.30±0.01^{de}
31.7M/0.6G	31.7	0.6	0.14±0.03^{efg}	0.60±0.10^{c}	0.19±0.00d^{efg}
31.7M/6G	31.7	6.0	0.71±0.01^{abc}	0.11±0.02^{fg}	0.29±0.02^{de}

[1]Mean values±standard deviation (n=3)
*Means that do not share a letter in columns and lines are significantly different (Tukey test, p<0.05)

Samples 22M/3G (Fig. 2f), 24M/6G (Fig. 2b), and 31.7M/6G (Fig. 2e) presented similar mechanical spectra, having G' close to 100 Pa, which means that they can support stress without destabilizing at the temperature of the experiment (20°C). Nevertheless, the 12M/6G (Fig. 2d) blend had even higher G' (~600 Pa), confirming that a smaller maltodextrin/gelatin ratio allows the system to approach the behaviour of pure gelatin, due to the lower steric impediment created by the maltodextrin chain (Ibanoglu, 2005). Thus, it becomes able to support stress at a lower concentration of total solids, which decreases the competition for water and promotes better stability conditions (Fig. 1b, d, e and Fig. 2b, d, e). It has been reported that blending polysaccharides and proteins – maltodextrin/collagen (Nicoleti; Telis, 2009), modified starch/gelatin (Marfil et al., 2012; Ferreira et al., 2016) - above certain concentrations may lead to thermodynamic incompatibilities between the biopolymers, resulting in structures less elastic than those obtained with lower concentration of pure protein.

Samples 26M/0.6G (Fig. 2a) and 31.7M/0.6G (Fig. 2c) had G^{gel} around 1 Pa, showing that the system acts rheologically as maltodextrin. Formulations 24M/6G (Figure 2b), 12M/6G (Fig. 2d), 31.7M/6G (Fig. 2e), and 22M/3G (Fig. 2f) presented mechanical spectra indicating that the samples behaved as if they were between the end of the plateau zone and the beginning of the transition zone. The width of the plateau zone relates to the nature of molecular interactions involved in the polymer network formation (Tsiami et al., 1997): a wider plateau appears when there are many non-covalent interactions, whereas a narrower one is detected when only few non-covalent interactions occur; in the absence of non-covalent interactions, the plateau zone may not appear.

Emulsion stability and droplet size

According to values of creaming index (CI) (Table 1) the emulsification method influenced emulsion stability. Also, competition for water between the encapsulating materials seemed to have an important role on two-phase stability. Considering the standard condition (high-shear homogenization without surfactant), samples 31.7M/0.6G and 12M/6G presented higher stabilities, since their CI values were the smallest found when using the same homogenization method (Table 1). The higher stability of emulsions containing higher concentration of solids could be explained based on polymer-induced depletion forces where the emulsion droplets are entrapped inside the polysaccharide gel-like matrix (Atkins, 1994; Parker et al., 1995; Moschakis et al., 2006). In addition, previous works showed that the higher the viscosity of an emulsion, the lower the probability of creaming. This happens because emulsions with higher viscosity present an increase in the drainage time of the continuous phase, when compared to low-viscosity emulsions (Tesch and Schubert, 2002). However, samples 31.7M/6G and 22M/3G had high values of CI. This may indicate that competition for water in the system caused by the high biopolymer concentration ends up weakening the emulsion's mechanical structure (Soriano et al., 199; Ferreira et al., 2016) even though, as it can be seen in Fig. 1e and 1f, these formulations presented higher values of G^{gel}. The collision rate between emulsion droplets - which promotes creaming - is affected by emulsion rheological properties (Tesch and Schubert, 2002). In such a way, it is possible to correlate the sample CI with its viscoelastic properties, taking into account that incompatibility of the biopolymers and competition for water makes the gel weaker and breaks down the emulsion structure (Tolstoguzov et al., 1988; Courthaudon et al., 1991; Khomutov et al., 1995). Regarding high-shear homogenization combined with surfactant (Tween 80) addition, samples 31.7M/6G and 24M/6G, formulated with the lower maltodextrin/gelatin ratios, showed the lowest values of CI and considerable decrease of CI compared with the standard condition. The decrease in CI is due to non-polar groups of Tween 80 that interact with the oil, while its ionic groups interact with the dispersed biopolymers, therefore increasing emulsion stability (Tolstoguzov, 1988; Atkins, 1994). The other samples showed CI > 0.5, with considerable increase compared with the standard condition.

When applying ultrasound homogenization, sample 31.7M/6G had the most significant decrease of CI comparing with high-shear emulsification, showing that this method can successfully enhance emulsification and increase stability of formulations that are not so stable when homogenized through other methods (Patist and Bates, 2008; Shi et al., 2011). On the other hand, sample 22M/3G presented a significant increase of IC, indicating that the choice of homogenization method must consider the ratio between the polymers. Compared to high-shear homogenization added with Tween 80, sonication showed good results and could be considered as a good alternative to the use of emulsifiers.

Both alternative homogenization methods proved to be able to decrease droplet sizes when compared to the standard high-shear homogenization. Samples with Tween 80 presented a maximum decrease in droplet average diameter of 52.2 % and those treated by ultrasound reached a decrease of 78 % (Table 2). In both situations, the average droplet sizes were lower than 3 μm, showing that both treatments can be used to prepare emulsions for microencapsulation. In microencapsulation of turmeric oleoresin using modified starch and gelatin a similar result was obtained (Ferreira et al., 2016), indicating that either the use of emulsifier or sonication may be good alternatives to enhance emulsification prior to microencapsulation.

Taking into account that the blend 26M/0.6G presented a decrease of 78 % in droplet size when subjected to ultrasound emulsification, in addition to presenting rheological behaviour similar to solutions composed of only maltodextrin, and thus having suitable flowing properties for the atomization step, this was selected to constitute the wall material for turmeric oleoresin in the spray drying assays.

Encapsulation efficiency (EE), solubility, and water content

Encapsulation efficiency for turmeric oleoresin microcapsules produced with different operational conditions in the spray dryer ranged from 3.5 to 77.2 % (Table 3). The wide range of values obtained show that drying parameters may substantially affect the effectiveness of microencapsulation. The operational conditions that significantly affected EE were atomization airflow and emulsion feeding flow. The combination of lower atomization airflow with lower emulsion feeding flow resulted in higher EE (Fig. 3). The drying-air temperature did not affect significantly EE, in a similar result to what was obtained by Cavalcante et al. (2017) on microencapsulation of soursop pulp by spray dryer using maltodextrin. The highest encapsulation efficiency (EE = 77.18 %) was obtained using: drying-air temperature of 160°C, atomization airflow of 418 L h^{-1}, and emulsion feeding flow of 5 mL min^{-1}, although a similar value (EE = 74.07 %) resulted when spray drying was carried out with drying-air at 130°C, atomization airflow of 418 L h^{-1}, and emulsion feeding flow of 2 mL min^{-1}. Both samples dried with atomization airflow of 536 L h^{-1} and emulsion feeding flow of 2 mL min^{-1} had the lowest values of EE (<5 %). This may be caused by the week gel structure (Fig. 1a and 2a), due to low gelatin concentration in the wall material. The scenario of high atomization airflow and low

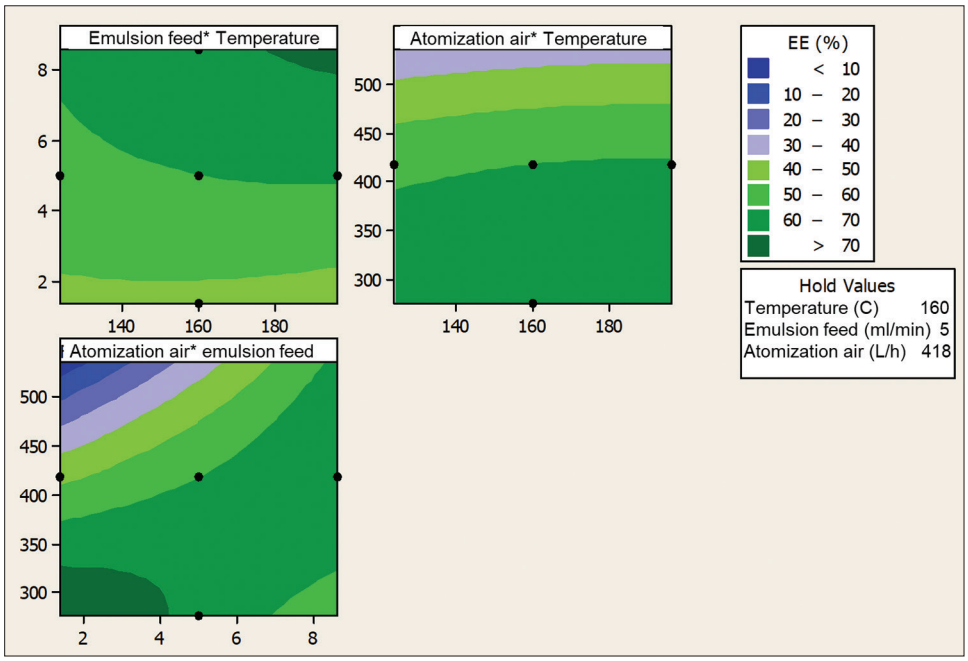

Fig 3. Graphic of contour of the influence of spray drying conditions on the encapsulation efficiency of turmeric oleoresin microencapsulated in 26M/0.6G matrix.

Table 2: Droplet diameter of turmeric oleoresin emulsions stabilized by maltodextrin/gelatin blends produced with different homogenization methods*

Sample	Maltodextrin (g 100 g^{-1})	Gelatin (g 100 g^{-1})	Droplet size (μm)[1]		
			Rotor-stator homogenization	Rotor-stator homogenization+Tween 80	Ultrasound homogenization
12M/6G	12.0	6.0	1.93±0.37^{de}	1.73±0.38^{defg}	1.14±0.34^{gh}
22M/3G	22.0	3.0	1.35±0.25^{efgh}	1.60±0.33^{defg}	1.14±0.23^{gh}
24M/6G	24.0	6.0	1.81±0.38^{def}	1.95±0.56^{de}	0.98±0.42^{h}
26M/0.6G	26.0	0.6	5.17±1.78^{a}	2.61±0.94^{c}	1.13±0.21^{gh}
31.7M/0.6G	31.7	0.6	4.29±1.01^{b}	2.06±0.51^{cd}	1.24±0.23^{fgh}
31.7M/6G	31.7	6.0	2.07±0.50^{cd}	1.72±0.50^{defg}	2.15±0.95^{cd}

[1]Mean values±standard deviation (n=30)
*Means that do not share a letter in columns and lines are significantly different (Tukey test, p<0.05)

Table 3: Encapsulation efficiency (EE), water content, and solubility of turmeric oleoresin encapsulated in the maltodextrin/gelatin blend 26M/0.6G[1] spray dried at different operational conditions*

Drying-air Temperature (°C)	Atomization airflow (L h^{-1})	Emulsion feeding flow (mL min^{-1})	Water content (%)	Solubility (%)	EE (%)[2]
160	418	5	2.72	89.22	77.18±0.16^{a}
190	300	2	3.58	88.32	65.46±3.28^{abc}
124	418	5	3.85	90.58	48.81±0.36^{d}
130	536	8	5.66	84.55	60.79±1.49^{bcd}
160	418	1.4	2.95	92.22	59.52±0.04^{bcd}
190	536	8	3.54	95.67	61.06±8.61^{abcd}
190	300	8	5.35	90.46	66.30±5.44^{abc}
130	536	2	7.61	98.77	4.79±0.27^{e}
160	275	5	5.43	86.82	64.97±1.65^{abcd}
130	300	2	4.98	87.48	74.07±3.85^{ab}
196	418	5	4.77	87.55	66.31±9.78^{abc}
160	418	8.6	6.96	86.87	53.32±0.51^{cd}
190	536	2	4.63	98.85	3.54±0.11^{e}
130	300	8	6.00	86.43	58.83±1.56^{bcd}

[1](26.0 g maltodextrin+0.6 g gelatin) 100 g^{-1}
[2]Mean values±standard deviation (n=2)
*Means that do not share a letter are significantly different (Tukey test, p<0.05)

emulsion feed flow creates an unsuitable encapsulant film on the particle`s surface during spray drying. In a previous paper, Ferreira et al. (2016) microencapsulated turmeric oleoresin in matrices of modified starch and gelatin and observed EE varying from 34.87 to 70.64 %, using 420 L h^{-1} of atomization air, drying-air at 160°C, and emulsion feeding flow of 3 mL min^{-1}. This is an indication that drying parameters must be taken into consideration together with the ratio between the polymers used to microencapsulate the core material (Akhavan Mahdavi et al., 2016; Schmitz-Schug et al., 2016; Cavalcante et al., 2017; Moser et al., 2017b). In a review of microencapsulation techniques, Pereira et al. (2018) reported values for EE using maltodextrin as wall material in spray drier varying from 43 to 90 %.

Fig. 4 shows a SEM microphotograph of the capsules produced using drying-air temperature of 160°C, atomization airflow of 418 L h^{-1}, and emulsion feeding flow of 5 mL min^{-1}. The microcapsules presented rounded shapes with a toothed surface, irregular depressions and concavities. The concavities and irregular surface can be attributed to the drying air velocity, the viscoelastic properties of the material and the shrinkage of the particles during the drying and cooling stages in the atomizer (Jafari et al., 2008; Sheu and Rosenberg, 1998). There was observed a tendency of capsules to form agglomerates. Although the microcapsules had irregular depressions and concavities, no cracks or pores were found in the surfaces of the microcapsules.

Solubility of the dried microcapsules ranged from 84.55 to 98.77% (Table 3), being significantly influenced by emulsion feeding flow, atomization airflow and the combination of these parameters. In previous works, Zuanon et al. (2017) and Moser et al. (2017a) presented similar values of solubility for spray dried microcapsules of turmeric oleoresin and grape juice, respectively. According to Kenyon (1995), the use of maltodextrin as carrier/encapsulant matrix may improve solubility attributes due to the hydrolysis degree and the re-polymerization of the polysaccharide.

Water content of spray dried powders is an indicative of the effectiveness of the dehydration process (Zuanon et al., 2017). In the present work, water content

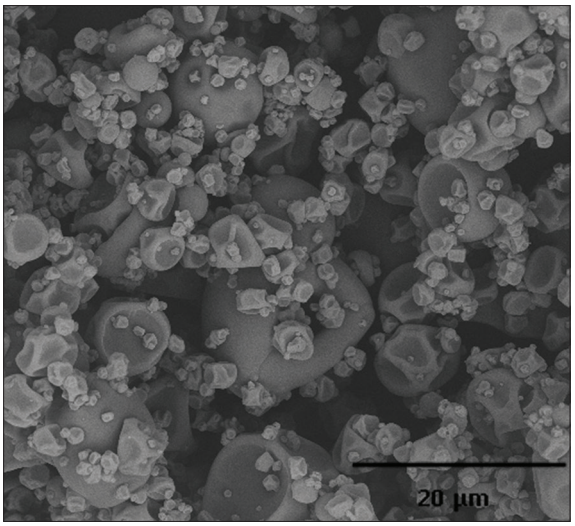

Fig 4. Microphotograph of particles produced with drying-air temperature of 160°C, atomization airflow of 418 L h^{-1}, emulsion feeding flow of 5 mL min^{-1}, and 26M/0.6G matrix.

varied from 2.68 to 7.61 % (Table 3), which shows how much the spray drying parameters may interfere on physical properties of the final powder. Cavalcante et al. (2017) presented values of water content under 1.45 %, whereas Ferreira et al. (2016) obtained microencapsulated turmeric oleoresin in modified starch/gelatin matrix with water content lower than 2.5 %.

Based on the analysis of encapsulation efficiency, water content, and solubility of the microcapsules, the recommended conditions to produce turmeric oleoresin microcapsules by spray drying a matrix composed of 26 g of maltodextrin and 0.6 g of gelatin per 100 g of emulsion are 160 °C for the drying-air temperature, atomization airflow of 420 L h^{-1} and emulsion feeding flow of 6 mL min^{-1}.

CONCLUSIONS

Based on the results presented in this paper it is possible to conclude that the homogenization method directly affected the emulsion stability and the average droplet size, with ultrasound homogenization appearing to be the most suitable for this process due to its ability to provide stable emulsions and small droplet sizes. Concerning the wall material formulation, not only gelatin concentration was important but also the polymer ratio and the total solids content, as the competition for water affected formation of the matrix microstructure and the rheological behaviour of the system. The recommended conditions for spray drying were 160 °C for the drying-air temperature, atomization airflow of 420 L h^{-1}, and emulsion feeding flow of 6 mL min^{-1}. High atomization airflow combined to low emulsion feeding flow led to lower values of encapsulation efficiency due to the high stress in the emulsion during atomization. This work highlighted the importance of controlling the physicochemical properties of the parent emulsion, the rheological properties of the encapsulant material, and the spray drying conditions in order to optimize the microencapsulation process, enhancing encapsulating efficiency and solubility of the microcapsules.

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Author's contributions

S.F. performed part of the experiments (emulsion characterization and rheological analysis), analysed the data and wrote the paper. G.M.O.P. performed spray drying assays and analysed the results. C.R.M. assisted in the data collection, data analysis and discussion. V.R.N. supervised the study and contributed to data analysis and discussion. All the authors have read and approved the final manuscript.

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