

MICROENCAPSULATION OF TURMERIC OLEORESIN IN BINARY AND TERNARY BLENDS OF GUM ARABIC, MALTODEXTRIN AND MODIFIED STARCH

Microencapsulação de oleoresina de cúrcuma em misturas binárias e ternárias de goma arábica, maltodextrina e amido modificado

Diana Maria Cano-Higuita¹, Harvey Alexander Villa Vélez²,
Vania Regina Nicoletti Telis²

ABSTRACT

Spray-drying is a suitable method to obtain microencapsulated active substances in the powdered form, resulting in powders with improved protection against environmental factors as well as with higher solubility in water, as in the case of turmeric oleoresin. The present study investigated the spray-drying process of turmeric oleoresin microencapsulated with binary and ternary mixtures of different wall materials: gum Arabic, maltodextrin, and modified corn starch. A statistical simplex centroid experimental design was used considering the encapsulation efficiency, curcumin retention, process yield, water content, solubility, and particle morphology as the analyzed responses. Wall matrices containing higher proportions of modified starch and gum Arabic resulted in higher encapsulation efficiency and curcumin retention, whereas the process yield and water content increased with higher proportions of maltodextrin and gum Arabic, respectively. Regression models of the responses were obtained using a surface response method (ANOVA way), showing statistical values of $R^2 > 0.790$. Also, mean analysis was carried out by Tukey's test, permitting to observe some statistical differences between the blends.

Index terms: Spray drying; drying aids; encapsulation efficiency; yield; curcumin.

RESUMO

A secagem por atomização é um método adequado para a obtenção de substâncias ativas microencapsuladas na forma de pó, resultando em um produto com uma melhor proteção contra diferentes fatores ambientais, bem como com uma maior solubilidade em água, para o caso da oleoresina de cúrcuma. Assim, no presente estudo, pesquisou-se o processo de secagem por atomização de oleoresina de cúrcuma microencapsulada com misturas binárias e ternárias de diferentes materiais de parede: goma arábica, maltodextrina e amido de milho modificado. Um planejamento experimental estatístico simplex centróide foi utilizado, considerando a eficiência de encapsulação, retenção de curcumina, rendimento do processo, teor de água, solubilidade e análise morfológica das partículas. Materiais de parede, contendo altas proporções de amido modificado e goma arábica, resultaram em alta eficiência de encapsulação e retenção de curcumina, enquanto que o rendimento do processo e o conteúdo de água, aumentaram com proporções mais elevadas de maltodextrina e goma arábica, respectivamente. Modelos de regressão para as respostas foram obtidos, pelo método de superfície de resposta (ANOVA), obtendo-se valores estatísticos de $R^2 > 0.790$. Além disso, uma análise de médias foi realizada pelo teste de Tukey, sendo observadas algumas diferenças estatísticas entre as misturas.

Termos para indexação: Secagem por aspersão; coadjuvantes de secagem; eficiência de encapsulação; rendimento; curcumina.

INTRODUCTION

The rhizome of turmeric (*Curcuma longa* L.), a plant from the Zingiberaceae family, is widely cultivated throughout tropical and subtropical regions of the world, mainly in India and China. Turmeric powder is extensively used as a spice, food preservative and coloring agent, and turmeric oleoresin is the organic extract obtained from the turmeric rhizomes. The phenolic compound called curcumin, a bright yellow dye, is the major turmeric pigment and, along with demethoxy curcumin and bis-demethoxy curcumin, is the main representative of the

curcuminoids. This group of phenolic compounds is responsible by the already proven therapeutic properties of *Curcuma longa*, which include antioxidant, anti-inflammatory, antimicrobial, and antitumoral activities (Martins et al., 2013; Araújo et al., 2010).

Despite being an important permitted natural colorant used in food, curcumin presents poor stability and low aqueous solubility. In fact, curcumin is practically insoluble in acidic solutions, unstable in solutions with a basic pH and breaks down easily, producing mainly ferulic acid, feruloylmethane and yellow brown condensation products, which invalidates

¹Universidade Estadual Paulista Julio de Mesquita Filho/UNESP – Departamento de Engenharia e Tecnologia de Alimentos – Rua Cristóvão Colombo – 2265 – Jardim Nazareth – 15054-000 – São José do Rio Preto – SP – Brasil – dianacano-higuita@gmail.com

²Universidade Estadual Paulista Julio de Mesquita Filho/UNESP – Departamento de Engenharia e Tecnologia de Alimentos – 15054-000 – São José do Rio Preto – SP – Brasil

Received in October 21, 2014 and approved in November 3, 2014

its use in pharmaceuticals and limits its use in food processing industries (Wang et al., 2009). Turmeric oleoresin microencapsulation may overcome these drawbacks by enhancing curcumin stability and improving turmeric solubility in aqueous media, facilitating its use as a natural pigment.

In the food-processing field, microencapsulation techniques have been widely used to protect food ingredients (i.e. flavors, essential oils, lipids, oleoresins, and colorants) against deterioration, volatile losses or interaction with other ingredients. The protective mechanism is to form a membrane, the wall system, around droplets or particles of the encapsulated material, the core. Microencapsulation not only protects against losses and chemical changes during food production and storage, but also enables production of food ingredients in the form of powdered products with new properties (Desai; Park, 2005; Ré, 2006; Ré, 1998).

Spray drying is an appropriate technique to convert fluid foods into powders, resulting in easy storage and transportation, and food and pharmaceutical industries make intense use of this process for the preparation of microencapsulated active ingredients. It is relative simple and allows the creation of microspherical structures including the core material embedded into a continuous matrix of protective biopolymers, starting from an aqueous polymeric system in which the active material had been dissolved, dispersed, or emulsified (Desai; Park, 2005; Ré, 2006; Jafari et al., 2008).

Different biopolymers have been used in developing microcapsules for the controlled release of various core materials. The selection of an adequate wall material for a particular application is important, since each substance possesses unique emulsifying and film forming properties that define its ability to function as an encapsulant. Carbohydrates, such as maltodextrins, starches, corn syrup solids, and gum Arabic, have all been widely used as encapsulating agents, but taking into account the difficulty in finding a single substance that meets all the requirements to act as a suitable wall material, in practice they are used in combination with each other (Jafari et al., 2008; Goula; Adamopoulos, 2012; Baranauskiene et al., 2006). Gum Arabic is highly soluble and surface-active, being extensively used as an encapsulation matrix for oils and flavors, however its high cost and irregular availability have motivated research for alternative encapsulation matrices. Maltodextrins have low cost, but lack of emulsifying capacity and lead to low volatile retention (Madene et al., 2006). Modified starches partially reproduce the functional properties of gum Arabic and have shown good encapsulation efficiency when blended with gum Arabic and

maltodextrin, particularly for microencapsulation of spice oleoresins. Ternary blends of gum Arabic, maltodextrin and modified starch gave higher protection to cardamom (Krishnan; Bhosale; Singhal, 2005a), cinnamon (Vaidya et al., 2006) and cumin (Kanakdande et al., 2007) oleoresins than gum Arabic alone.

Based on these considerations, the aim of this work was to investigate the effect of different combinations of gum Arabic, maltodextrin and modified corn starch as wall materials on the microencapsulation of turmeric oleoresin by spray drying. The results were evaluated in terms of process yield, curcumin retention, encapsulation efficiency, water content, solubility of the microcapsules and particle morphology.

MATERIAL AND METHODS

Raw material and sample preparation

Turmeric oleoresin OC-500 (Baculerê, Olímpia, Brazil), gum Arabic (GA) (Synth, São Paulo, Brazil), maltodextrin (MD) Mor-Rex® 1910, with dextrose equivalent of 10 (Corn Products, São Paulo, Brazil) and modified starch (MS) (Hi-Cap™ 100, National Starch, USA) were acquired directly from the industries. A batch of each of these biopolymers was stored in a hermetic flask at room temperature, (25 ± 2) °C; their respective water contents were determined in order to be considered in the preparation of the wall material suspensions.

For sample preparation, the wall materials (GA, MD and MS) were dispersed in deionized water at room temperature, (25 ± 2) °C, and mixed until complete dissolution. After dispersion, the biopolymer suspensions were allowed to rest at refrigerator temperature, (4 ± 1) °C, for 20-24 h to ensure complete hydration of the polymers. Once hydrated, the biopolymers were mixed with the turmeric oleoresin using a rotor-stator homogenizer Ultra-Turrax T25 Basic (IKA, Wilmington, USA) operating at 18.000 rpm for 20 min in an ice bath. The emulsions were prepared at 30% ($w \cdot w^{-1}$) total solids with an oil load of 15% (dry basis). All three biopolymers were combined following a simplex centroid experimental design consisting of three components, giving rise to fourteen treatments (Table 1).

Microencapsulation by spray drying

The emulsions were dried using a pilot scale spray dryer (B-290, Büchi, Flawil, Switzerland) equipped with a drying chamber with dimensions of 61 cm high and 20 cm diameter and a spray nozzle of 0.7 mm. The air compressor pressure was set at 6 $\text{kgf} \cdot \text{cm}^{-2}$ and the emulsion was fed to the nozzle by using a peristaltic pump. Preliminary tests were carried out to establish the drying conditions and the

Table 1: Centroid experimental design for the wall biopolymer blends.

Wall material formulation	Biopolymer proportions (% dry wall material)		
	Maltodextrin	Gum Arabic	Modified starch
MD:GA (75:25)	75	25	0
MD:GA (50:50)	50	50	0
MD:GA (25:75)	25	75	0
GA (100)	0	100	0
GA:MS (25:75)	0	25	75
GA:MS (50:50)	0	50	50
MS (100)	0	0	100
MD:MS (75:25)	75	0	25
MD:MS (50:50)	50	0	50
MD:MS (25:75)	25	0	75
MD(100)	100	0	0
MD:GA:MS (17:66:17)	17	66	17
MD:GA:MS (33:33:33)	33	33	33
MD:GA:MS (66:17:17)	66	17	17
MD:GA:MS (17:17:66)	17	17	66

following set of operation parameters was adopted: airflow rate of $536 \text{ L} \cdot \text{h}^{-1}$, feed flow rate of $3.9 \text{ mL} \cdot \text{min}^{-1}$, and inlet air temperature of $170 \text{ }^\circ\text{C}$. The powder samples collected from the base of the cyclone were packaged in airtight low-density polyethylene bags, covered with aluminum foil and stored in a desiccator containing silica gel for further analyzes.

Analytical methods

Encapsulation efficiency (EE)

The encapsulation efficiency (EE, %) was determined as the ratio of encapsulated curcumin to the total amount of curcumin in the microcapsules (Equation 1).

$$EE = \frac{(\text{Curcumin}_{total} - \text{Curcumin}_{surface})}{\text{Curcumin}_{total}} \times 100 \quad (1)$$

where Curcumin_{total} is the total content of curcumin in the dried microcapsules and $\text{Curcumin}_{surface}$ is the amount of non-encapsulated curcumin present in the surface of the microcapsules (Mortaza et al., 2012).

Surface curcumin

The amount of curcumin present on the surface of the microcapsules ($\text{Curcumin}_{surface}$) was measured

using a spectrophotometer SP-220 (Biospectro, Curitiba, Brazil). Surface curcumin was extracted with methanol following the procedure described by Kaushik and Roos (2007) with some modifications. Approximately 0.05 g of microcapsules were mixed with 15 mL of methanol in plastic tubes with mild manual agitation, followed by centrifugation (model Z306, Hermle Labor Technik, Wehingen, Germany) for 20 min at $10.414 \times g$ at $20 \text{ }^\circ\text{C}$. An aliquot of 0.4 mL from the collected supernatant was added to 3.6 mL of methanol and the curcumin present in the mixture was quantified by reading the absorbance at 425 nm. Triplicate determinations were performed for each sample. A standard curve was obtained by measuring absorbance of curcumin mixed in methanol at various concentrations (w.w⁻¹). A blank sample of methanol was used in the spectrophotometer along with the methanol containing curcumin to correct the effect of absorbance by methanol.

Total curcumin

A sample of about 0.05 g of microcapsules was dissolved in 5 mL of deionized water and kept in a thermostatic bath at $60 \text{ }^\circ\text{C}$ for 40 min, being stirred each 5 min in vortex mixer (V1 Plus, Boeco, Hamburg, Germany). After that, 15 mL of methanol were added to the sample, which was finally stirred in vortex and centrifuged

two times at $10.414 \times g$ for 10 min at 20 °C. An aliquot of 0.4 mL of supernatant was transferred to an amber glass vial and methanol was added until completing a total volume of 4 mL. The amount of curcumin present in the mixture was quantified by reading the absorbance at 425 nm. Triplicate determinations were performed for each sample (Kaushik; Roos, 2007).

Curcumin retention (CR)

Curcumin retention (CR, %) was determined as the ratio of total curcumin content in the microcapsules after spray drying to the total curcumin initially added to the prepared emulsion (Equation 2) (Frascareli et al., 2012).

$$CR = \frac{\text{Curcumin}_{\text{total}}}{\text{Curcumin}_{\text{initial}}} \times 100 \quad (2)$$

Process yield (Py)

The powder yield was determined as the ratio of the weight of powder (dry basis) collected at the dryer exit to the weight of dry matter in the emulsion taken for drying (Equation 3) (Martins et al., 2013):

$$Py(\%) = \frac{\text{weight of powder (dry basis)}}{\text{dry mass of raw material}} \times 100 \quad (3)$$

Water content (X)

The water content (X, dry basis %) of microcapsules was determined gravimetrically by drying in a vacuum oven at 70 °C until constant weight, according to the AOAC method 926.12 (AOAC, 1997).

Solubility

A sample of about 1 g of microcapsules was added to 100 mL of distilled water and subjected to magnetic stirring for 5 min. The dispersion was filtered in filter paper and a 20-mL aliquot of the filtrate was transferred to a tared Petri dish and dried in oven at 105 °C for 5 h. The percent solubility was calculated by gravimetric analysis (Cano-Chauca et al., 2005).

Particle morphology

Samples were immobilized in an appropriate support with the help of a conductive adhesive tape and coated with gold in a sputter coater (Polaron SC7620, Oregon, US) and examined with a scanning electron microscope (FEI Inspect S 50, SEM Electron Microscopy Ltd., US. Laboratório de Caracterização Estrutural DEMa/

UFSCAR). SEM was carried out at 5kV magnification of 2000 and 5000 times.

Statistical analyses

Statistical analyses were carried out using the computational software Minitab v.16 (Minitab Inc., Pennsylvania, USA) by an one-way ANOVA using the Tukey's test with $\alpha = 95\%$. The ANOVA settings used in the model selection were: model-type "interaction", the grouping terms mode "continuous" and statistical validation "sum of squares" to determine the hierarchy of the terms.

RESULTS AND DISCUSSION

Encapsulation efficiency and curcumin retention

The encapsulation efficiency ranged from 8 to 46% and was significantly influenced by the wall material formulation as can be observed in table 2, which includes all the other response variables (Py, CR, X, and solubility). The highest value of efficiency, 45.23 %, was found when using a binary blend GA:MS (50:50), followed by an efficiency of 40.56% obtained for MD:GA (25:75).

There was no significant differences ($p < 0.05$) in encapsulation efficiency when analyzing only the ternary blends, indicating that any of them could be a good alternative to minimize the gum Arabic content, reducing operational costs and contributing to enable the microencapsulation of turmeric oleoresin by spray drying process. These results are in accordance with other published works regarding spray drying microencapsulation of spice oleoresins. Krishnan, Bhosale and Singhal (2005a), Vaidya et al. (2006), and Kanakdande et al. (2007) obtained similar results, respectively, for cardamom, cinnamon, and cumin oleoresins when using different combinations of gum Arabic, maltodextrin and modified starch as wall material. These authors concluded that a ternary blend of these biopolymers provided higher protection and, consequently, increased stability of the active material during storage than gum Arabic alone.

Taking into account the blends composed of different proportions of MD:GA, it is possible to observe a decrease in the encapsulation efficiency with increasing MD:GA ratio (Table 2). This result is attributed to the fact that pure maltodextrin presents very low surface activity, leading to poor encapsulation and requiring maltodextrin to be blended with materials that possess good emulsifying properties, such as gum Arabic, in

order to yield products with desired characteristics in terms of stability (Desai; Park, 2005; Madene et al., 2006). According to Krishnan, Bhosale and Singhal (2005b) pure gum Arabic was found to be a better wall material for encapsulation of cardamom oleoresin than pure maltodextrin or pure modified starch; the efficient entrapment of the constituents and volatiles in gum Arabic was attributed to the biopolymer good film forming capability and plastic behavior, rather than a glassy property, which prevents cracking of the protection matrix. Gum Arabic is recognized for having emulsifying properties and has the ability to form films around oil droplets, which results from the highly branched arabinogalactan-protein structure containing both protein and polysaccharide moieties. The hydrophobic polypeptides anchor the polysaccharide onto the surface of the oil droplet and the hydrophilic carbohydrate chains prevent the aggregation by forming a thick charged layer, proportioning a stabilization mechanism, characteristic of few polysaccharide-protein systems, which gives it an advantage over other types of wall materials (Adamiec et al., 2012; Nakamura et al., 2006).

Samples containing only maltodextrin and modified starch (MS and MD:MS) showed the lower

encapsulation efficiencies, reinforcing the relevance of gum Arabic in the encapsulating matrix used for hydrophobic materials. According to table 3, the model that resulted in the best fit for encapsulation efficiency was the cubic model, with correlation coefficient, $R^2 = 0.790$. The response surface corresponding to efficiency is shown in figure 1.

Regarding the results for curcumin retention, the higher values were obtained for the following wall material formulations: GA:MS (25:75), with 68.43%; MD:GA (25:75), with 65.46%; and the ternary mixture MD:GA:MS (17:17:66), with 61.98%. All these three formulations showed encapsulation efficiencies higher than 30 %. On the other hand, it is possible to observe (Table 2) that for pure modified starch, MS (100), although the curcumin retention was considerable high (55.48 %), the encapsulation efficiency was poor. A similar combination of results was obtained for MD:GA (75:25). This means that a great amount of the initial curcumin had been recovered in the dried product, but it was not efficiently entrapped and protected by the wall matrix. The major part of it remained in the particle surface and hence it susceptible to faster degradation.

Table 2: Properties of turmeric oleoresin microcapsules produced with different wall material formulations.

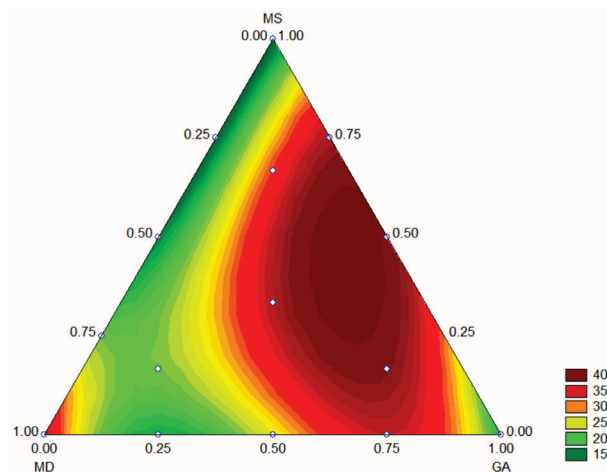
Wall material formulation	EE (%)*	Py (%)*	CR (%)*	X (% dry basis)*	Solubility (%)*
MD:GA (75:25)	11.78 ± 0.51g	55.83	40.35 ± 1.01h	4.81 ± 0.06a	91.69 ± 0.04d
MD:GA (50:50)	24.60 ± 0.49e	52.30	49.59 ± 0.00f	2.14 ± 0.04de	96.50 ± 0.05ab
MD:GA (25:75)	40.56 ± 0.05b	43.33	65.46 ± 0.13b	2.40 ± 0.08d	95.65 ± 0.25bc
GA (100)	16.73 ± 0.19f	29.73	24.87 ± 0.18k	4.12 ± 0.05b	92.57 ± 0.28cd
GA:MS (25:75)	32.74 ± 0.15d	26.49	68.43 ± 0.44a	1.58 ± 0.05f	99.25 ± 0.00a
GA:MS (50:50)	45.23 ± 0.04a	23.97	59.15 ± 0.19d	2.14 ± 0.05de	95.63 ± 0.53bc
MS (100)	11.84 ± 1.70g	51.10	55.48 ± 0.17e	1.01 ± 0.06gh	96.80 ± 0.45ab
MD:MS (75:25)	20.72 ± 0.35f	55.82	31.33 ± 0.00j	2.90 ± 0.00c	97.35 ± 0.15ab
MD:MS (50:50)	7.96 ± 0.38h	56.62	35.41 ± 0.00i	1.30 ± 0.01fg	97.88 ± 0.83ab
MD:MS (25:75)	18.94 ± 1.32f	57.68	41.61 ± 0.36h	0.84 ± 0.16h	95.90 ± 0.02abc
MD(100)	36.37 ± 1.36c	16.89	36.02 ± 0.74i	2.98 ± 0.03c	97.75 ± 0.45ab
MD:GA:MS (17:66:17)	31.51 ± 0.03d	33.01	36.35 ± 0.32i	2.78 ± 0.68c	96.90 ± 0.02ab
MD:GA:MS (33:33:33)	33.84 ± 0.39d	38.64	45.21 ± 0.39g	2.35 ± 0.24de	97.33 ± 0.07ab
MD:GA:MS (66:17:17)	33.18 ± 0.29d	56.88	54.82 ± 0.08e	2.78 ± 0.44c	85.35 ± 0.50e
MD:GA:MS (17:17:66)	31.23 ± 0.60d	57.40	61.98 ± 0.35cd	2.10 ± 0.09de	86.83 ± 0.48e

*Mean values ± standard error (n = 2). Different letters in the same column indicate significant difference at $p < 0.05$ comparison between different wall material using the same drying process. *EE* (Encapsulation Efficiency); *Py* (Process Yield); *CR* (Curcumin Retention); *X* (Water Content).

Table 3: Regression models from surface response.

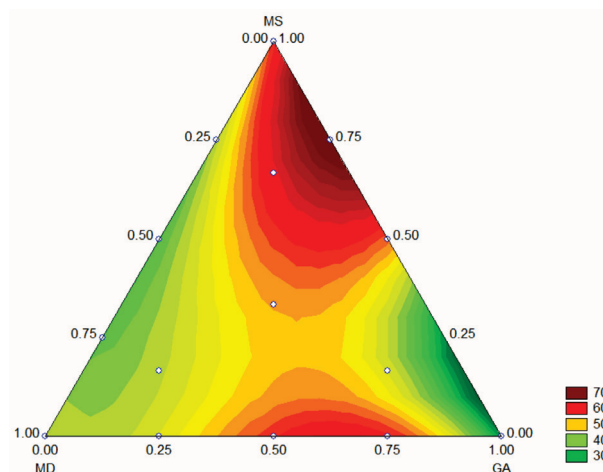
Response	Model	p	R ²
Encapsulation efficiency (EE)	*EE (%) = 36.176×MD + 17.423×GA + 12.394×MS - 4.038×MD×GA - 37.24×MD×MS + 98.210×GA×MS + 206.238×MD×GA×MS - 146.339×MD×GA×(MD-GA) - 17.812×MD×MS×(MD-MS) - 49.323×GA×MS×(GA-MS)	0.003	0.790
Curcumin retention (CR)	*CR (%) = 42.182×MD + 26.536×GA + 54.755×MS + 83.430×MD×GA - 50.282×MD×MS + 47.976×GA×MS - 4.132×MD×GA×MS - 111.531×MD×GA×(MD-GA) + 13.630×MD×MS×(MD-MS) - 183.481×GA×MS×(GA-MS)	0.003	0.793
Water content (X)	*X (% d.b.) = 3.227×MD + 4.196×GA + 1.013×MS - 3.396×MD×GA - 2.669×MD×MS - 1.166×GA×MS + 15.981×MD×GA×MS + 12.602 MD×GA×(MD-GA) + 2.648×MD×MS×(MD-MS) - 0.940×GA×MS×(GA-MS)	0.001	0.850
Process Yield (Py)	**Py (%) = 25.585×MD + 26.121×GA + 50.374×MS + 112.485×MD×GA + 92.531×MD×MS - 73.496×GA×MS	0.003	0.807

* Cubic regression model by Anova test. ** Quadratic regression model by Anova test. p: probability of factor F ($\alpha = 95\%$). R²: coefficient of correlation.

**Figure 1:** Response surface for encapsulation efficiency (EE).

The model that resulted in the best fit for curcumin retention was the cubic model (Table 3) with coefficient of correlation ($R^2 = 0.793$) and the corresponding response surface presented in figure 2. Lower values of curcumin retention occurred at higher maltodextrin contents, which is the same trend observed in figure 1 for encapsulation efficiency. These results can be attributed to the presence of large amounts of oleoresin that were not encapsulated, being subjected to subsequent losses during the spray drying process, either by volatilization or by adherence to the dryer

walls. This tendency has been related by authors as McNamee et al. (1998), Ahn et al. (2008) and Tan et al. (2005).

**Figure 2:** Response surface for curcumin retention (CR).

Process yield (Py)

The process yield obtained for the experimental runs ranged from 23.97 to 57.68% as shown in table 2. These results are coherent for a laboratory-scale spray dryer apparatus because low yields (around 50%) are usually reported in spray dryers with capacity of 1.0 kg H₂O·h⁻¹ (Ameri; Maa, 2007; Filková et al., 2006).

The regression analysis showed that a quadratic model (Table 3) could be fitted to the process yield response with $R^2 = 0.807$. Figure 3 indicates that higher process yields were observed at higher proportions of maltodextrin and modified corn starch, whereas increasing proportion of gum Arabic led to decreasing yield.

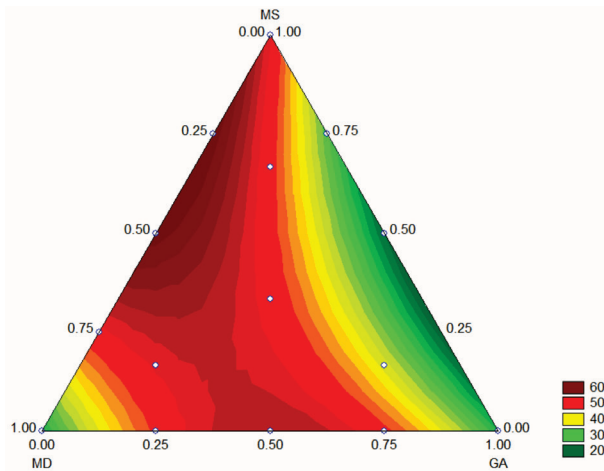


Figure 3: Response surface for process yield (Py).

This result is probably related to a lower deposition of solids on the dryer walls during spray drying of samples containing higher proportions of maltodextrin and modified starch. Ferrari et al. (2013) reported that blackberry powder produced with gum Arabic as the carrier agent showed lower glass transition temperature (T_g) than those produced with maltodextrin. The higher glass transition temperature of maltodextrin minimizes the solid deposition on the dryer walls (Abbas et al., 2010).

Water content and solubility

The moisture content of the turmeric oleoresin microcapsules resulted to be in the range of 0.84 and 4.81 % dry basis (Table 2). The best fitted model was the cubic one (Table 3), which showed the highest correlation coefficient, $R^2 = 0.850$. The response surface corresponding to water content can be seen figure 4.

The samples with higher proportions of modified starch showed the lowest water contents, whereas higher proportions of gum Arabic resulted in higher moisture. Martinelli et al. (2007) observed that lemon juice powder produced with maltodextrin as a drying aid was less hygroscopic than that produced using gum Arabic as the carrier. Tonon et al. (2012) also observed that flaxseed

oil microcapsules produced with modified starch as wall material showed lower values of moisture than those produced with gum Arabic, which was attributed to the high hygroscopicity of the gum, as it has many hydrophilic ramifications, which promote higher water adsorption from the ambient air.

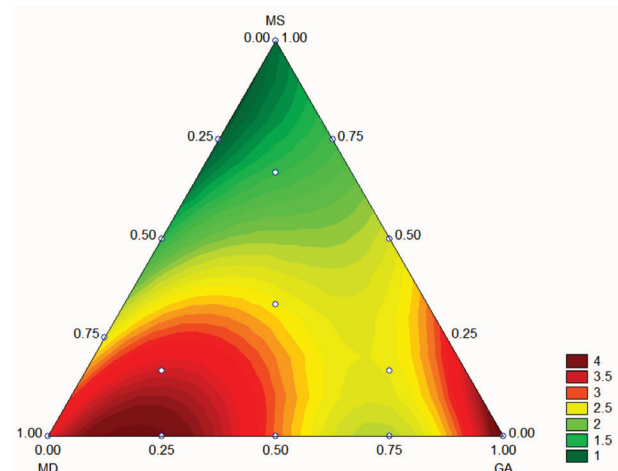


Figure 4: Response surface for water content (X).

The solubility results included in table 2 indicate that microencapsulation of turmeric oleoresin using carbohydrate materials as wall matrices was able to confer high solubility to this active agent since, independently of the wall material formulation, the microcapsule solubility was higher than 85 %. According to table 2, there were only few conditions that resulted in significant differences between solubility values ($p > 0.05$), in such a way that it was not possible to obtain a predictive regression model. The obtained model resulted in a very low value of R^2 .

Particle morphology

Some of the studied formulations were selected for evaluation of particle morphology. The capsules prepared with pure gum Arabic were taken as the reference, since this material is widely used for this kind of applications. Regarding the possibility of total or partially substitution of gum Arabic, the other samples evaluated were the binary blend MD:MS (0.75:0.25), which resulted in the highest encapsulation efficiency between the formulations containing only MS and MD, and the sample MD:GA:MS (33:33:33), which resulted in the best combination of *EE*, *Py*, and solubility between the ternary blends.

The microcapsules obtained using pure gum Arabic and a ternary mixture of MD:GA:MS had smooth surfaces, but some also showed the formation of teeth on the surface, indicating the occurrence of shrinkage. The external surfaces indicate the existence of solid walls, with no cracks or breaks, an attribute that is essential to ensure low permeability to gases, better protection and retention of oleoresin (Aghbashlo et al., 2013; Kha et al., 2014), as can be seen in figure 5.

The microcapsules obtained from the sample MD:MS (0.75:0.25) were slightly circular presenting wrinkles on the wall, and no cracks or fissures. The particle size corresponding to this formulation appeared to be larger than the other samples when observed at the same magnification (5000x).

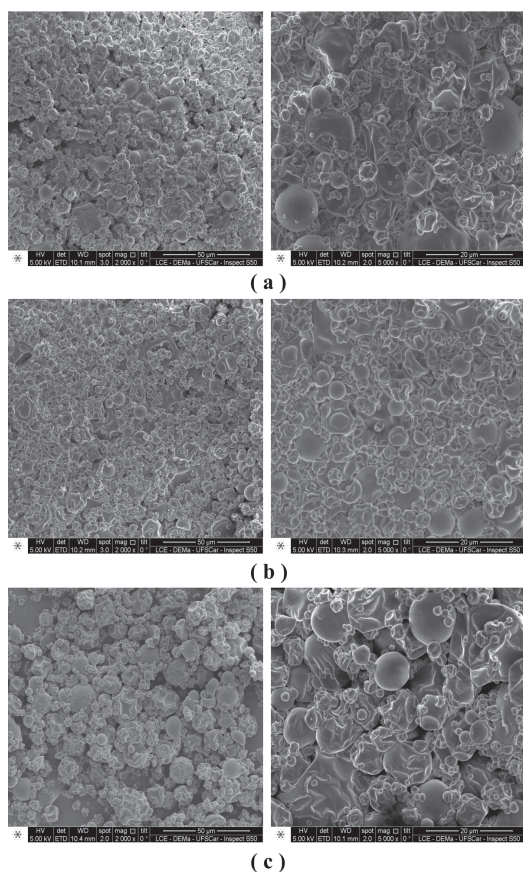


Figure 5: SEM images of microcapsules produced by spray drying with different wall materials: (a) GA:MD:MS (33 % gum Arabic:33 % maltodextrin:33 % modified starch) (b) GA (gum Arabic); (c) MD:MS (75 % maltodextrin:25 % modified starch); magnification (left 2000x) and (right 5000x).

Spray-dried materials are typically hollow spheres. Vacuole formation results from the shrinking process that occurs after the hardening of the outer surface followed by the expansion of the air bubbles trapped inside the droplet (Fernandes et al., 2014). Contraction and deformation of the particles dried by spray drying are related to the temperature and diffusion of water, since long drying time makes the structure deform, shrink (which results in roughness), and collapse (which result in breakage) (Chen; Ozkan, 2007).

CONCLUSIONS

In this study it was possible to encapsulate turmeric oleoresin by the spray drying method using binary and ternary mixtures of gum Arabic, maltodextrin and modified corn starch as wall materials. The simplex centroid experimental design was a valuable tool for evaluating the main effects of the proportions of gum Arabic, maltodextrin and modified corn starch on the responses: encapsulation efficiency, curcumin retention, process yield and water content. The response surfaces showed that mixtures containing higher proportions of modified starch and gum Arabic resulted in higher encapsulation efficiency and curcumin retention, whereas the process yield and water content increased with higher proportions of maltodextrin and gum Arabic, respectively. All the studied wall matrices resulted in microcapsule solubility higher than 85 %. The capsules obtained showed typical morphology of spray dried particles, with some differences in size being observed depending on the wall material formulation.

ACKNOWLEDGEMENTS

The authors are grateful to the Sao Paulo State Research Support Agency, FAPESP, for the financial support (Processes 2010/09614-3 and 2009/13033-9).

REFERENCES

- ABBAS, K.A. et al. The significance of glass transition temperature in processing of selected fried food products: A review. **Modern Applied Science**. 4:3-21, 2010.
- ADAMIEC, J. et al. Microencapsulation of kaffir lime oil and its functional properties. **Drying Technology: An International Journal**. 30:914-920, 2012.
- AGHBASHLO, M. et al. Influence of wall material and inlet drying air temperature on the microencapsulation of fish oil by spray drying. **Food and Bioprocess Technology**. 6:1561-1569, 2013.

- AHN, J.H. et al. Optimization of microencapsulation of seed oil response surface methodology. **Food Chemistry**. 107:98-105, 2008.
- AMERI, M.; MAA, Y.F. Spray drying of biopharmaceuticals. Stability and process considerations. **Drying Technology**. 24:763-768, 2007.
- AOAC. Association of Official Analytical Chemists, Gaithersburg, AOAC International, p. 1-5. 1997
- ARAÚJO, R.R. et al. The preparation of ternary solid dispersions of an herbal drug via drying of liquid feed. **Drying Technology: An International Journal**. 28:412-421, 2010.
- BARANAUSKIENE, R. et al. Properties of oregano (*Origanum vulgare* L.), citronella (*Cymbopogon nardus* G.) and marjoram (*Majorana hortensis* L.) flavors encapsulated into milk protein based matrices. **Food Research International**. 39:413-425, 2006.
- CANO-CHAUCA, M. et al. Effect of the carriers on the microstructure of mango powder obtained by spray drying and its functional characterization. **Innovative Food Science and Emerging Technologies**. 6:420-428, 2005.
- CHEN, X.D.; OZKAN, N. Stickiness, functionality, and microstructure of food powders. **Drying Technology: An International Journal**. 25:959-969, 2007.
- DESAI, K.G.H.; PARK, H.J. Recent developments in microencapsulation of food ingredients. **Drying Technology: An International Journal**. 23:1361-1394, 2005.
- FERNANDES, R.V.B. et al. Gum arabic/starch/ maltodextrin/inulin as wall materials on the microencapsulation of rosemary oil. **Carbohydrate Polymers**. 101:532, 2014.
- FERRARI, C.C. et al. Storage stability of spray dried blackberry powder produced with maltodextrin or gum arabic. **Drying Technology: An International Journal**. 31:470-478, 2013.
- FILKOVÁ, I. et al. Industrial spray drying systems. In: MUJUMDAR, A.S. **Handbook of industrial drying**. Boca Raton: CRC Press, 2006. 215p.
- FRASCARELI, E.C. et al. Effect of process conditions on the microencapsulation of coffee oil by spray drying. **Food and Bioproducts Processing**. 90:413-424, 2012.
- GOULA, A.M.; ADAMOPOULOS, K.G. A new technique for spray dried encapsulation of lycopene. **Drying Technology: An International Journal**. 30:641-652, 2012.
- JAFARI, S.M. et al. Encapsulation efficiency of food flavours and oils during spray drying. **Drying Technology**. 26:816-835, 2008.
- KANAKDANDE, D. et al. Stability of cumin oleoresin microencapsulated in different combination of gum arabic, maltodextrin, and modified starch. **Carbohydrate Polymers**. 67:536-541, 2007.
- KAUSHIK, V.; ROOS, Y.H. Limonene encapsulation in freeze-drying of gum arabic-sucrose-gelatin systems. **Society of Food Science and Technology**. 40:1381-1391, 2007.
- KHA, T.C. et al. Microencapsulation of gac oil by spray drying: Optimization of wall material concentration and oil load using response surface methodology. **Drying Technology: An International Journal**. 32:385-397, 2014.
- KRISHNAN, S.; BHOSALE, R.; SINGHAL, R.S. Microencapsulation of cardamom oleoresin: Evaluation of blends of gum arabic, maltodextrin and a modified starch as wall materials. **Carbohydrate Polymers**. 61:95-102, 2005a.
- KRISHNAN, S.; KSHIRSAGAR, A.C.; SINGHAL, R.S. The use of gum arabic and modified starch in the microencapsulation of a food flavoring agent. **Carbohydrate Polymers**. 62:309-315, 2005b.
- MADENE, A. et al. Flavour encapsulation and controlled release- a review. **International Journal of Food Science and Technology**. 41:1-21, 2006.
- MARTINELLI, L. et al. Thermodynamic and quality properties of lemon juice powder as affected by maltodextrin and Arabic gum. **Drying Technology**. 25:2035-2045, 2007.

- MARTINS, M.R. et al. Curcuminoid content and antioxidant activity in spray dried microparticles containing turmeric extract. **Food Research International**. 50:657-663, 2013.
- MCNAME, B.F. et al. Emulsification and microencapsulation properties of gum arabic. **Journal of Agricultural and Food Chemistry**. 46:4551-4555, 1998.
- MORTAZA, A. et al. Optimization of emulsification procedure for mutual maximizing the encapsulation and exergy efficiencies of fish oil microencapsulation. **Powder Technology**. 225:107-117, 2012.
- NAKAMURA, A. et al. Soy soluble polysaccharide stabilization at oil-water interfaces. **Food Hydrocolloids**. 20:277-283, 2006.
- RÉ, M.I. Microencapsulation by spray drying. **Drying Technology: An International Journal**. 16: 1195-1236, 1998.
- _____. Formulating drug delivery systems by spray drying. **Drying Technology: An International Journal**. 24:433-446, 2006.
- TAN, L.H. et al. Effect of oil loading on microspheres produced by spray drying. **Journal Microencapsulation**. 22:253-259, 2005.
- TONON, R.V. et al. Microencapsulation of flaxseed oil by spray drying. Effect of oil load and type of wall material. **Drying Technology**. 30:1491-1501, 2012.
- VAIDYA, S. et al. Microencapsulation of cinnamon oleoresin by spray drying using different wall materials. **Drying Technology: An International Journal**. 24:983-992, 2006.
- WANG, Y. et al. Study on the antibiotic activity of microcapsule curcumin against foodborne pathogens. **International Journal of Food Microbiology**. 136:71-74, 2009.

ERRATUM

In the manuscript entitled Microencapsulation of turmeric oleoresin in binary and ternary blends of gum arabic, maltodextrin and modified starch, published in the volume 39, number 2, page 176, the equation 3:

$$Py(\%) = \frac{\text{iiiiiiiiiiiiii}}{\text{dry mass of raw material}} \times 100 \quad (3)$$

Should be replaced by:

$$Py(\%) = \frac{\text{dry mass of microparticles}}{\text{dry mass of raw material}} * 100 \quad (3)$$