

# Influence of spray drying operating conditions on microencapsulated rosemary essential oil properties

*Influência das condições operacionais da secagem por atomização nas propriedades de óleo de alecrim microencapsulado*

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## Abstract

Spray drying is an important method used by the food industry in the production of microencapsulated flavors to improve handling and dispersion properties. The objective of this study was to evaluate the influence of the process conditions on the properties of rosemary essential oil microencapsulated by spray drying using gum Arabic as encapsulant. The effects of the wall material concentration (10-30%), inlet air temperature (135-195 °C), and feed flow rate (0.5-1.0 L.h<sup>-1</sup>) on the moisture content, hygroscopicity, wettability, solubility, bulk and tapped densities, particle density, flowability, and cohesiveness were evaluated using a 2<sup>3</sup> central composite rotational experimental design. Moisture content, hygroscopicity and wettability were significantly affected by the three factors analyzed. Bulk density was positively influenced by the wall material concentration and negatively by the inlet air temperature. Particle density was influenced by the wall material concentration and the inlet air temperature variables, both in a negative manner. As for the solubility, tapped density, flowability, and cohesiveness, the models did not fit the data well. The results indicated that moderate wall material concentration (24%), low inlet air temperature (135 °C), and moderate feed flow rate (0.7 L.h<sup>-1</sup>) are the best spray drying conditions.

**Keywords:** spray dryer; gum Arabic; *Rosmarinus officinalis*.

## Resumo

A secagem por atomização é um importante método utilizado pela indústria de alimentos na produção de *flavors* microencapsulados, melhorando suas propriedades de manuseio e dispersão. O objetivo deste estudo foi avaliar a influência das condições de processo nas propriedades de óleo essencial de alecrim microencapsulado por secagem por atomização, utilizando-se goma Arábica como agente encapsulante. Os efeitos da concentração de material de parede (10% a 30%), temperatura de entrada do ar (135-195 °C) e vazão de alimentação (0.5-1.0 L.h<sup>-1</sup>) na umidade, higroscopicidade, molhabilidade, solubilidade, densidades de leito e compactada, densidade de partícula, e fluidez e coesividade foram avaliados através de um delineamento experimental composto central rotacional 2<sup>3</sup>. A umidade, higroscopicidade e molhabilidade foram afetadas significativamente pelos três fatores analisados. A densidade de leito foi influenciada positivamente pela concentração de material de parede e negativamente pela temperatura do ar de entrada. Já a densidade de partícula foi influenciada pelas variáveis concentração de material de parede e temperatura do ar de entrada, ambas de forma negativa. Para a solubilidade, densidade compactada, fluidez e coesividade, os modelos não apresentaram bons ajustes para a variação dos dados. Os resultados indicaram que uma moderada concentração de material de parede (24%), baixa temperatura de entrada do ar (135 °C) e moderada vazão de alimentação (0,7 L.h<sup>-1</sup>) foram as melhores condições para o processo de secagem por atomização.

**Palavras-chave:** secador por atomização; goma Arábica; *Rosmarinus officinalis*.

## 1 Introduction

Spray drying, a leading technology in the food industry, is the most commonly used microencapsulation method for food ingredients (REINECCIUS, 2006). This technique is a well-known process suitable for drying materials due to the very short heat contact time and the high rate of evaporation resulting in high quality, stable, functional, and low moisture content products (YOUSEFI; EMAM-DJOMEH; MOUSAVI, 2011; SARALA et al., 2012).

A vast majority of the flavor compounds used in the food industry are mainly in the liquid form at room temperature. Microencapsulation can potentially offer numerous benefits to the food ingredients being encapsulated. Handling and flow

properties can be improved by converting a liquid to solid encapsulated form. The microencapsulation procedure protects hygroscopic materials from moisture and maintain the stability of ingredients that are volatile or sensitive to heat, light, or oxidation (JAFARI et al., 2008).

Essential oils are slightly soluble in water and impart their odor and taste to the water. They contain terpenes, alcohols, esters, aldehydes, ketones, phenols, ethers, and other minor compounds (PARRIS; COOKE; HICKS, 2005). *Rosmarinus officinalis* L. (Lamiaceae), commonly known as rosemary, of the family Labiatae, is an aromatic shrub cultivated

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mainly in Mediterranean countries, such as Spain, Morocco, Tunisia, France, and Italy (FLAMINI et al., 2002).

The physical properties of the microencapsulated essential oil related to its ease of dispersion in an aqueous solution include moisture content, bulk density, particle density and respective particle porosity, and the instantanization properties (wetting, dispersibility, and solubility). These properties are influenced by the nature of the feed (solids content, viscosity, and temperature), type of spray dryer, operating speed and pressure, and inlet and outlet air temperatures (FINNEY; BUFFO; REINECCIUS, 2002; ABADIO et al., 2004; YOUSEFI; EMAM-DJOMEH; MOUSAVI, 2011).

Numerous materials, such as wall materials (encapsulating agents), are available for spray drying microencapsulation of food flavors (JAFARI et al., 2008; YANG; XIAO; DING, 2009). Gum Arabic is used by the flavor industry in spray drying applications protecting the core material from oxidation and volatilization (SHAIKH; BHOSALE; SINGHAL, 2006). Moreover, it exhibits high solubility and low viscosity in aqueous solution when compared to other hydrocolloid gums (WILLIAMS; PHILLIPS, 2000), which facilitates the spray drying process. The objective of this study was to evaluate the influence of spray drying operational conditions on the properties of microencapsulated rosemary essential oil.

## 2 Materials and methods

### 2.1 Materials

Rosemary (*Rosmarinus officinalis* Leaf Oil) essential oil (Ferquima Ind. e Com. Ltda, Vargem Grande Paulista, Brazil), originating from Tunisia, was used as the core material. Gum Arabic (Colloides Naturels Brasil, São Paulo, Brazil) was used as wall material.

### 2.2 Preparation of emulsions

Gum Arabic solution was prepared by dissolving gum Arabic in distilled water. It was prepared on the day before emulsification and kept overnight at room temperature to ensure a full saturation of the polymer molecules. Rosemary essential oil was progressively added to the wall material solution while stirring at 3.500 rpm for 10 minutes using a rotor-stator blender (Ultra-Turrax IKA T18 basic, Wilmington, USA). Wall material concentration varied according to the experimental design (10-30%). The emulsion was used as the feeding liquid in the spray drying process. For each treatment, about 1000 mL of sample was prepared for the production of the encapsulated powders. The mass ratio of rosemary essential oil to wall material was 1:4 (w/w).

### 2.3 Microencapsulation by spray drying

The emulsions were dried using a spray drier (model MSD 1.0; Labmaq do Brasil, Ribeirão Preto, Brazil) equipped with a two-fluid nozzle atomizer. Inlet air temperatures (135-195 °C) and feed flow rates (0.5-1.0 L.h<sup>-1</sup>) were varied, and drying air flow was kept at 40 L.min<sup>-1</sup>. The dried powders were collected and stored in opaque, air tight containers at 4 °C while waiting for further analysis. All the measurements were conducted in triplicate.

### 2.4 Experimental design

A rotatable central composite design was used to perform the tests for the microencapsulation of rosemary essential oil, considering three factors (independent variables): wall material concentration (10-30%), inlet air temperature (135-195 °C) and feed flow rate (0.5-1.0 L.h<sup>-1</sup>). Three levels of each variable and four repetitions at the center point, giving a total of 18 combinations (Table 1), were considered in this study.

**Table 1.** Experimental design for the spray drying assays.

Assay no.	Coded variables			Process variables		
	X1	X2	X3	Wall material (%)	Inlet air temperature (°C)	Feed flow rate (L.h <sup>-1</sup> )
1	-1.00	-1.00	-1.00	14.05	147	0.60
2	-1.00	-1.00	1.00	14.05	147	0.90
3	-1.00	1.00	-1.00	14.05	183	0.60
4	-1.00	1.00	1.00	14.05	183	0.90
5	1.00	-1.00	-1.00	25.95	147	0.60
6	1.00	-1.00	1.00	25.95	147	0.90
7	1.00	1.00	-1.00	25.95	183	0.60
8	1.00	1.00	1.00	25.95	183	0.90
9	-1.68	0.00	0.00	10.00	165	0.75
10	1.68	0.00	0.00	30.00	165	0.75
11	0.00	-1.68	0.00	20.00	135	0.75
12	0.00	1.68	0.00	20.00	195	0.75
13	0.00	0.00	-1.68	20.00	165	0.50
14	0.00	0.00	1.68	20.00	165	1.00
15	0.00	0.00	0.00	20.00	165	0.75
16	0.00	0.00	0.00	20.00	165	0.75
17	0.00	0.00	0.00	20.00	165	0.75
18	0.00	0.00	0.00	20.00	165	0.75

Regression models were evaluated for the following variables: moisture content, hygroscopicity, wettability, solubility, bulk and tapped densities, particle density, bulk porosity, flowability and cohesiveness (BARROS-NETO; SCARMINO; BRUNS, 2010). Some nonsignificant terms were eliminated, and the resulting equations were tested for adequacy and fit by the analysis of variance (ANOVA). The optimum conditions for the microencapsulation of rosemary essential oil were determined by analyzing the results of variables that were significantly affected by spray drying conditions, using Response Desirability Profiling from STATISTICA version 8.0 software (Stat Soft. Inc., Tulsa, USA), according to methodology described by Derringer and Suich (1980).

## 2.5 Characterization of the microcapsules

### Moisture content

The moisture content of the powders was determined by the AOAC (ASSOCIATION..., 2007) method. The powder weight loss percentage (%) after oven-drying at 105 °C until a constant weight was obtained, and moisture content (%) was calculated.

### Hygroscopicity

Hygroscopicity was determined according to the method proposed by Cai and Corke (2000) with some modifications. The powder samples of each treatment were (approximately 1 g) placed in a container with saturated NaCl solution (75.29% RH) at 25 °C. After one week, the samples were weighed, and hygroscopicity was expressed as g of adsorbed moisture per 100 g dry solids (g/100 g).

### Wettability

Wettability of the powders was determined using the method of Fuchs et al. (2006) with some modifications. The powder samples (0.1 g) were sprinkled over the surface of 100 mL of distilled water at 20 °C without agitation. The time it took until the last powder particles submerge was recorded and used for a relative comparison of the extent of wettability between the samples.

### Solubility

The solubility of the powders was evaluated according to the method proposed by Cano-Chauca et al. (2005) with modifications. The powders were weighed (1 g) and stirred in 25 mL of distilled water for 5 min using a blender. The solution was then centrifuged at 3000×g for 10 min. An aliquot of 20 mL of the supernatant was transferred to pre-weighed Petri dishes and oven-dried at 105 °C overnight. Solubility (%) was calculated as the percentage of dried supernatant in relation to the amount of powder originally added (1.0 g).

### Bulk and tapped densities

The powders were gently loaded into a 100 mL tared graduated cylinder to the 100 mL mark and weighed. The volume read directly from the cylinder was then used to

calculate the bulk density ( $\rho_{\text{bulk}}$ ) according to the relationship: mass/volume (JINAPONG; SUPHANTHARIKA; JAMNONG, 2008). For tapped density ( $\rho_{\text{tapped}}$ ), approximately 5 g of powder was freely poured into a 25 mL graduated glass cylinder, and the samples were repeatedly tapped manually by lifting and dropping the cylinder under its own weight at a vertical distance of 10 cm until negligible difference in volume between succeeding measurements was observed. Given the mass  $m$  and the apparent (tapped) volume  $V$  of the powder, the powder tapped density was computed as  $m/V$  ( $\text{g}\cdot\text{cm}^{-3}$ ) (GOULA; ADAMOPOULUS, 2008).

### Particle density

The particle densities of the powders were calculated by adopting the pycnometer method. An amount of  $2.5 \pm 0.04$  g of each treatment was placed in an empty liquid pycnometer (25 mL) and filled with a measured volume of toluene. Particle density is the total particle weight divided by its total volume. Toluene was used because of its ability to penetrate the finest external pores connected to the surface of the material without dissolving the material (KROKIDA; MAROULIS, 2001).

### Bulk porosity

Bulk porosity ( $\epsilon$ ) of the powder samples was calculated using the relationship between the tapped ( $\rho_{\text{tapped}}$ ) and particle densities ( $\rho_{\text{particle}}$ ) of the powders (Equation 1) (JINAPONG; SUPHANTHARIKA; JAMNONG, 2008) and expressed as percentage, as follows:

$$\epsilon = \frac{(\rho_{\text{particle}} - \rho_{\text{tapped}})}{\rho_{\text{particle}}} \times 100 \quad (1)$$

### Flowability and cohesiveness

Flowability and cohesiveness of the powders were evaluated in terms of Carr index (CI) and Hausner ratio (HR), respectively (JINAPONG; SUPHANTHARIKA; JAMNONG, 2008). Both CI and HR were calculated from the bulk ( $\rho_{\text{bulk}}$ ) and tapped ( $\rho_{\text{tapped}}$ ) densities of the powders using Equations 2 and 3 as follows:

$$CI = \frac{(\rho_{\text{tapped}} - \rho_{\text{bulk}})}{\rho_{\text{tapped}}} \times 100 \quad (2)$$

$$HR = \frac{\rho_{\text{tapped}}}{\rho_{\text{bulk}}} \quad (3)$$

## 3 Results and discussion

### 3.1 Response surface analysis

Table 2 shows the regression coefficients for the coded second-order polynomial equation, the F values, and the determination coefficients ( $R^2$ ). Some non-significant terms were eliminated, and the resulting equations were tested for adequacy and fitness by the analysis of variance (ANOVA). The

fitted models were suitable, showing significant regression, low residual values, no lack of fit, and satisfactory determination coefficients.

### 3.2 Moisture content

The moisture of the microcapsules (Figure 1) varied from 0.26 to 3.16%. The moisture values were close to those obtained in studies on spray drying of essential oils (1.70-4.16%) (ADAMIEC; KALEMBA, 2006) and d-limonene (1.20-2.70%) (JAFARI; HE; BHANDARI, 2007). The variable that presented higher influence on the particle moisture was the inlet air temperature. That effect of the temperature was also verified by Finney, Buffo and Reineccius (2002) and Ersus and Yurdagel (2007) in spray microencapsulation studies on orange oil and anthocyanin pigments, respectively. It was also verified that with the air drying temperature increase and flow rate decrease, the moisture content of the powder decreased. However, under high feed rates, the amount of microcapsule water increased, considering the same temperature and same amount of wall material. This occurs because, due to the high amount of product to be dried, the contact time of the emulsion with the drying air is not enough to cause the evaporation of all of the water. The

moisture of the powders increased with the elevation of the gum Arabic concentration since the molecules of this carrier agent are large, they hindered water molecule diffusion during the spray drying process. Furthermore, an increase in the encapsulating agent concentrations results in a decrease in the time to form the particle shell (TONON; GROSSO; HUBINGER, 2011) hindering water diffusion during the drying process.

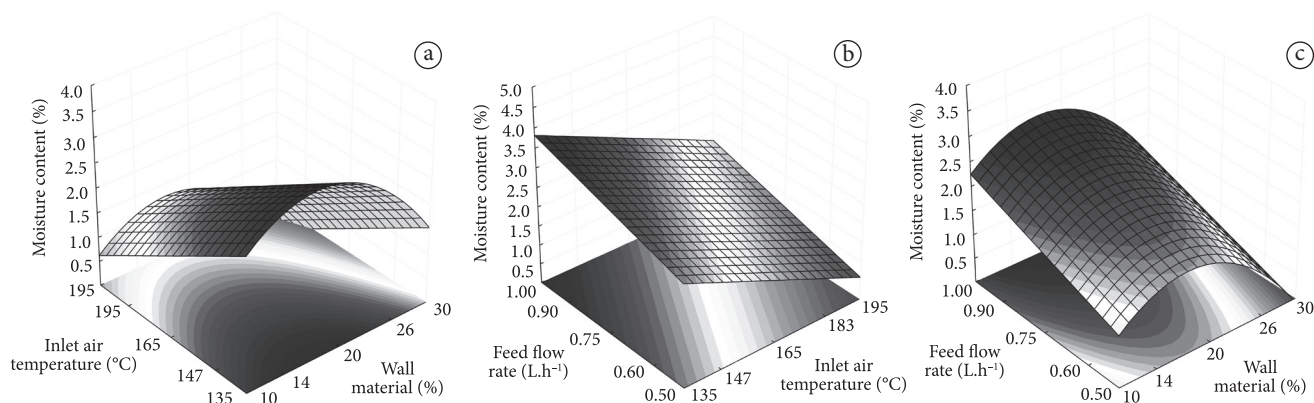
### 3.3 Hygroscopicity

The absorption of water is an important factor for powder reconstitution since it can lead to caking reducing dispersibility. The values obtained for hygroscopicity (Figure 2) varied from 15.87 to 18.90%, and they were influenced significantly by all of the independent variables. The quadratic term of the wall material concentration had a positive effect, and a minimum value of hygroscopicity of gum Arabic at concentration around 20% was estimated. The increase in the concentration of gum Arabic starting from 10% promoted a decrease in the hygroscopicity; however, at very high values the water absorption increased again. This fact can be attributed to the hygroscopic nature of the gum Arabic. The inlet air temperature was the variable that most influenced the hygroscopicity of the powders,

**Table 2.** Coded second-order regression coefficients for significant responses.

Coefficient	Moisture content (%)	Hygroscopicity (%)	Wettability (seconds)	Bulk density (g.mL <sup>-1</sup> )	Particle density (g.mL <sup>-1</sup> )
$\beta_0$	2.20**	16.67**	217.36**	0.31**	1.13**
$\beta_1$	-0.34**	ns	49.41**	0.018**	-0.054**
$\beta_{11}$	-0.40**	0.61**	44.56**	ns	ns
$\beta_2$	-0.61**	0.81**	ns	-0.02**	-0.068**
$\beta_{22}$	ns	ns	ns	ns	ns
$\beta_3$	0.35**	-0.42**	ns	ns	ns
$\beta_{33}$	ns	0.27*	ns	ns	ns
$\beta_{12}$	ns	ns	49.63**	ns	ns
$\beta_{13}$	ns	ns	33.88*	ns	ns
$\beta_{23}$	ns	ns	31.88*	0.013**	ns
$F_{\text{calculated}}$	11.53	17.54	8.04	29.21	19.72
$F_{\text{tabulated}}(0,05)$	3.18	3.18	3.11	3.34	3.68
$R^2$	0.78	0.84	0.77	0.86	0.72

ns: nonsignificant ( $p > 0.05$ ). \*\*Significant at 5% probability. \*Significant at 10% probability.



**Figure 1.** Response surfaces for moisture content (%): a) feed flow rate: 0.75 L.h<sup>-1</sup>; b) wall material: 20%; c) inlet air temperature: 165 °C.

and lower hygroscopicity values were obtained when the lowest temperatures were used, which can be explained by the fact that the powders produced under these conditions have higher moisture content and consequently lower water concentration gradient between the product and the atmosphere. Similar behavior have been found for the microencapsulation of coffee oil by spray drying using gum Arabic as the encapsulant (FRASCARELI et al., 2012). The flow rate affected moisture content and consequently the hygroscopicity of the powders in an opposite manner to that of the temperature, and an increase in the flow rate caused hygroscopicity reduction.

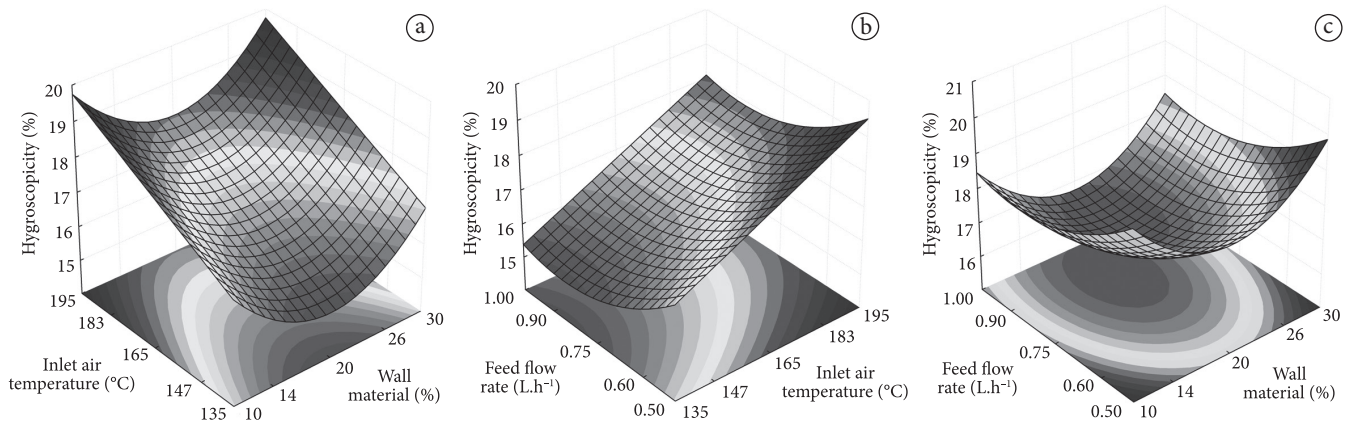
### 3.4 Wettability

The wettability is characterized as the rehydration ability of a powder in water. The capacity of the microcapsules to mix with water is one of the most important reconstitution properties (BAE; LEE, 2008). In the present study (Figure 3), the time it took the powders to become totally wet varied from 155 to 481 seconds. Values close to these were found by Jinapong, Suphantharika and Jammong (2008) and Favaro-Trindade et al. (2010) in studies on the production of soy milk instant powder and hydrolyzed casein, respectively. The concentration of wall material and the interaction of wall material and temperature

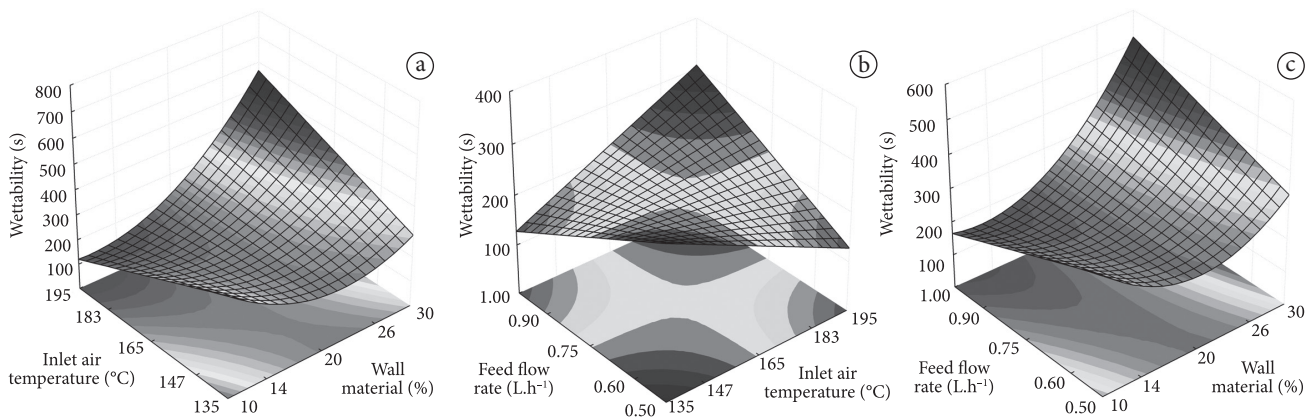
were the variables that most influenced this property values. The longest particle instantization times occurred at higher inlet air temperature and carrier agent concentration. This fact can be explained due to the lower moisture content of the powders obtained under these conditions. Caking, which usually occurs in powders with higher moisture, can contribute to wettability since the liquid penetrates into the pores more easily (BUFFO et al., 2002). A caked product allows the larger particles to settle to the bottom and disperse more easily in water (GHOSAL; INDIRA; BHATTACHARYA, 2010).

### 3.5 Solubility

The variables under study did not present significant effects ( $p > 0.05$ ) on the solubility of the powders. Other results reported in the literature do not show statistical differences under different drying conditions for this reconstitution property either (SOUSA et al., 2008; KHA; NGUYEN; ROACH, 2010). Probably the microcapsules did not show difference in this parameter because the solubility is strongly influenced by the carrier type (YOUSEFI; EMAM-DJOMEH; MOUSAVI, 2011), which was not investigated in the present study. It can be observed that all particles were relatively soluble in spite of the hydrophobic nature of the encapsulate, with results



**Figure 2.** Response surfaces for hygroscopicity (%): a) feed flow rate: 0.75 L.h<sup>-1</sup>; b) wall material: 20%; c) inlet air temperature: 165 °C.



**Figure 3.** Response surfaces for wettability (%): a) feed flow rate: 0.75 L.h<sup>-1</sup>; b) wall material: 20%; c) inlet air temperature: 165 °C.

varying from 55.75 to 67.75%. Ahmed et al. (2010), in a study on encapsulation of purple sweet potato by spray drying, found solubility values ranging from 40.24 to 56.95%. Several reports attribute superior properties to other carrier agents related to water solubility, such as maltodextrin (GRABOWSKI; TRUONG; DAUBERT, 2006; GOULA; AMOPOULOUS, 2010).

### 3.6 Bulk and tapped densities

The bulk density was influenced positively by the wall material concentration and negatively by the inlet air temperature, flow rate, and air temperature interaction. The values of this response varied from 0.25 to 0.36 g.mL<sup>-1</sup> (Figure 4). Values close to these were obtained in the encapsulation of vegetable oil by spray drying (0.32-0.34 g.mL<sup>-1</sup>) (TURCHIULI et al., 2005) in the production of soy milk powders (0.21-0.22 g.mL<sup>-1</sup>), (JINAPONG; SUPHANTHARIKA; JAMNONG, 2008) and in the microencapsulation of oregano essential oil (0.34-0.45 g.mL<sup>-1</sup>) (BOTREL et al., 2012). It was verified that density decreased with the increase in the inlet air temperature. This result was also found by Souza et al. (2009) and Goula and Adamopoulos (2008). At high temperatures, the evaporation rates are faster and consequently the dry product has a more porous structure. The increase in the drying air temperature usually causes a decrease in the apparent density since there is a greater tendency to hollow particle formation (WALTON, 2000). However, at high encapsulant concentrations, there was an increase in the bulk density of the rosemary essential oil microcapsules. The heavier material accommodates itself more easily in the spaces among the particles, resulting in higher density (TONON; BRABET; HUBINGER, 2010).

Tapped density is an important factor related to packaging, transport, and commercialization of powders; thus, this value can be useful in terms of weight and amount of material that will fit into a container (FINNEY; BUFFO; REINECCIUS, 2002). A high density dry product can be stored in smaller containers in comparison with a low density product (QUISPE-CONDORI; SALDAÑA; TEMELLI, 2011). This property was significantly influenced by the temperature variable only ( $p < 0.05$ ), presenting a negative linear effect. However, the model did not fit the data well. The results obtained varied between 0.41 and 0.52 g.mL<sup>-1</sup>.

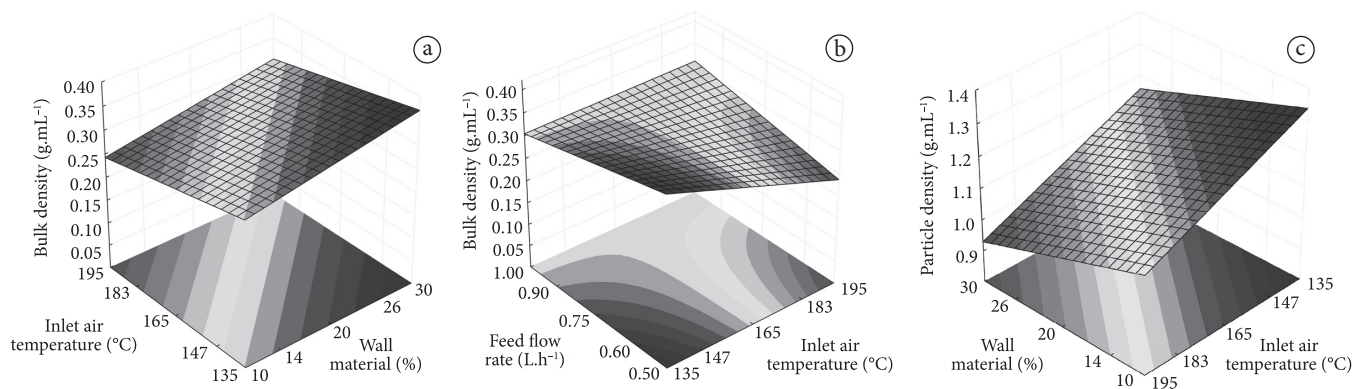
Bae and Lee (2008) and Finney, Buffo and Reineccius (2002) found similar values, 0.25-0.28 g.mL<sup>-1</sup> and 0.48-0.65 g.mL<sup>-1</sup>, for the tapped densities of avocado oil and orange essential oil, respectively. The lowest density was observed with the increase in the drying temperature. Such a fact is in agreement with the results of other studies (CAI; CORKE, 2000; CHEGINI; GHOBADIAN, 2007). At very high temperatures, the drying process is conducted very fast resulting in larger expansion of the droplets and therefore lower powder density (WALTON, 2000).

### 3.7 Particle density

Particle density was influenced by the concentration of wall material and inlet air temperature variables, both in a negative manner (Figure 4). Particle density varied from 0.97 to 1.27 g.mL<sup>-1</sup>. In studies on flavor atomization, the values found were in the range from 0.93 to 1.19 g.mL<sup>-1</sup> (FINNEY; BUFFO; REINECCIUS, 2002) and 0.74 to 0.92 g.mL<sup>-1</sup> (BOTREL et al., 2012). It can be influenced by the drying air temperature, size of the produced particles, spraying conditions, and feed emulsion (nature of the encapsulate and solid contents). Particle density can also decrease due to steam formation in the drying droplet causing the expansion of the particle whose dimensions become fixed even with the continuity of the drying process (FINNEY; BUFFO; REINECCIUS, 2002). Abadio et al. (2004) also found that with the increase in the encapsulant concentration there was a decrease in the true density of the microcapsules of pineapple juice, probably due to the lower moisture content.

### 3.8 Porosity

Another property of fundamental importance in food processing operations is porosity. It plays an important role in the reconstitution of dry products controlling the rehydration speed (KROKIDA; ZOGZAS; MAROULIS, 1997). The porosity of the powders was influenced significantly only by the wall material concentration variable ( $p < 0.05$ ), presenting a negative linear effect. However, the model did not fit the data well. The values found varied from 52.83 to 65.70%. In the study conducted by Souza et al. (2009), the values found for the porosity of spray-dried tomato pulp were 50-59%. Jinapong,



**Figure 4.** Response surfaces for bulk density (g.mL<sup>-1</sup>): a) feed flow rate: 0.75 L.h<sup>-1</sup>; b) wall material: 20%; c) and for the particle density (g.mL<sup>-1</sup>): feed flow rate: 0.75 L.h<sup>-1</sup>.

Supphantharika and Jamnong (2008) found porosity values of 70.02-74.47% for soy milk powders obtained by atomization.

### 3.9 Flowability and cohesiveness

Quality control parameters for microcapsules such as Carr Index and Hausner ratio, which evaluate the flow of the powders, should be considered (FITZPATRICK, 2005). The indexes of the powders produced in this study were influenced significantly only by the temperature variable ( $p < 0.05$ ), with a negative linear effect, and by the interaction of the inlet air temperature and flow rate ( $p < 0.05$ ). The model did not predict data variations. The Carr index values varied from 23.09 to 40.22%. The rosemary essential oil microcapsules presented bad and fair flowability. In the study of Fuchs et al. (2006) on vegetable oil, a value of 44% was found for the powders produced by spray and Jinapong, Supphantharika and Jamnong (2008) obtained values varying from 32 to 40% for atomized soy milk. This property, poor for these powders, can be related to the size of the microcapsules since smaller particles can lead to a high surface area per mass unit. The presence of high contact surface area among particles enables higher cohesion and attrition force formation that lead to flow resistance (FITZPATRICK et al., 2004; FITZPATRICK, 2005). Furthermore, the powders obtained in this study, using an encapsulate of hydrophobic nature, can lead to low flow. The composition of the surface of the powder particles plays an important role in this property because flowability involves overcoming the surface interactions among the particles (FITZPATRICK et al., 2004). A high Hausner ratio means that the powder is more cohesive and less capable of flowing freely. The data obtained for this ratio varied from 1.30 to 1.67; therefore, the microcapsules produced can be classified from intermediate to high. In a study on linseed oil microencapsulated by spray drying, Quispe-Condori, Saldaña and Temelli (2011) found values for this ratio that varied from 1.51-1.77.

### 3.10 Response optimization

For the microencapsulation of rosemary essential oil, optimum condition was maintained based on hygroscopicity, wettability, particle density, and bulk density results. These variables were significantly affected by the spray drying conditions. In spite of being significant, moisture content was not used based on the low values found (below 3.16%). Lower hygroscopicity, lower time for wettability, and higher particle and bulk density values were considered when defining the optimum conditions in this study. According to the results of response surface methodology, the combination of 24% wall material concentration, 135 °C inlet air temperature, and 0.7 L.h<sup>-1</sup> feed flow rate was found to provide the best results. Under these conditions, predicted values for hygroscopicity, wettability, particle density, and bulk density of 15.9%, 214 seconds, 1.23 g.mL<sup>-1</sup> and 0.36 g.mL<sup>-1</sup> were found, respectively.

## 4 Conclusions

The use of higher temperatures and low feed flow rate contributed to the decrease of particle moisture content and, in general, it was related to the variation of the hygroscopicity and wettability of the powders. These spray drying conditions led to a reduction in wetting time and hygroscopicity. Higher bulk densities were obtained with the use of low temperature and high

wall material concentration. Particle density was lower at high inlet air temperatures and with low gum Arabic concentrations. The combination of 24% wall material concentration, 135 °C inlet air temperature, and 0.7 L.h<sup>-1</sup> feed flow rate was found to provide the best results.

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