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Effect of solids content and oil load on the microencapsulation process of rosemary essential oil

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ABSTRACT

Microencapsulation by spray-drying is widely used in the preparation of flavors in the food industry. This study sought to evaluate the influence of oil load and wall material ratios on the properties of rosemary essential oil microencapsulated by spray-drying, using maltodextrin and modified starch as carriers. Increasing the oil load of the emulsion, the obtained particles presented higher moisture content, lower hygroscopicity and higher total oil content. The increased wall material content lengthened the wettability time. This research further suggested that the optimal wall concentration and oil load conditions are 20.9% and 29.4%, respectively. The encapsulated oil composition proved to be quite similar to pure oil. The mean particle size was 12.2 μm and the analysis of the particles revealed surfaces with some depressions, however without fissures.

1. Introduction

Essential oils and their plant components have been regarded with great interest as the sources of many natural products. *Rosmarinus officinalis* L. (Lamiaceae), commonly known as rosemary, is a household plant used throughout the world as a food-flavoring agent (Laborda et al., 2013; Couto et al., 2012) and is widely accepted as having a high level of antioxidant activity (Hosni et al., 2013; Wojdyło et al., 2007; Peng et al., 2005). Its antioxidant activity justifies the use of rosemary in a broad range of applications, including in food preservatives (Hamre et al., 2010), cosmetics (Lee et al., 2011), nutraceuticals and phytomedicines (Ibarra et al., 2010). In the food industry, rosemary is a very frequently used herb and its extracts are added to some products to improve their oxidative stability and to improve the organoleptic properties (Sui et al., 2012). Several of studies reported beneficial effects of rosemary essential oil as natural antioxidants in preventing color deterioration and/or lipid oxidation in meat products (Doolaege et al., 2012); for example, in beef, pork and chicken (Kim et al., 2013). The rosemary essential oil presented effects on the reduction in the odor characteristics related to lipid oxidation in meat products treated with rosemary extracts (Kim et al., 2013; Nissen et al., 2004; Nassu et al., 2003). However, like other oils, rosemary essential oil is sensitive to external factors such as heat, light and oxygen. The functionality and application of oils as well as the shelf life of products containing it, could be limited (Huynh et al., 2008).

Flavor plays an important role in consumer satisfaction and influences food consumption. Microencapsulating volatile ingredients prior to use in foods helps to preserve flavor, limiting aroma degradation or loss during processing and storage and therefore makes food more attractive to consumers (Xiao et al., 2011). Besides, it converts the oil into a free-flowing powder (Krishnan et al., 2005) which can be applied in the development of new food formulations and products. The stability of flavors in different foods presents great importance because of its relationship with the quality and acceptability of food products (Yang et al., 2009).

Microencapsulation is the process by which tiny particles or droplets are surrounded by a coating wall or embedded in a homogeneous or heterogeneous matrix, creating small capsules (Calvo et al., 2011; Gharsallaoui et al., 2007). The food industry employs microencapsulation by spray-drying in the preparation of dry stable additives, such as essential oils and flavors (Fuchs et al., 2006; Kha et al., 2010). During the drying process, the feed solution is sprayed in droplets in a stream of hot air (Saravacos and Kostaropoulos, 2002). The liquid droplets dry in seconds as a result

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of highly efficient heat and mass transfer (Toledo, 2007). The wall matrix can be composed of one or several different types of components. Carbohydrates, such as modified starches, corn syrup solids and maltodextrins, have generally been considered good encapsulating agents because they exhibit low viscosities at levels of high solids concentration, good solubility, and their use leads to high amounts of encapsulated oils (Drusch et al., 2006; Gharsallaoui et al., 2007).

The main factors that affect the encapsulation efficiency of microencapsulated oils and flavors include the following: the type of wall material, the properties of the core materials (including their concentration and volatility), the characteristics of the infeed emulsion (including total solids, viscosity, and the size of the droplets) and the conditions of the spray-drying process (including atomization type, inlet air temperature, air flow, and humidity levels) (Jafari et al., 2008). During spray-drying microencapsulation, it is important to prevent volatile losses and to maximize the amount of core material inside the powder particles. For volatiles in particular, improving the retention of core material in the final encapsulated powder is very important because much of these ingredients can be lost during the process (Jafari et al., 2007). Thus, it is important to optimize the spray-drying process to obtain minimal surface oil in the powder particles. Reports on microencapsulation of rosemary essential oils in literature are still limited. Microencapsulation using spray drying process has been described for encapsulate *Cinnamon* oleoresin (Vaidya et al., 2006), oregano, citronella and marjoram essential oil (Baranauskiené et al., 2006), lemon myrtle oil (Huynh et al., 2008), sweet orange oil (Yang et al., 2009) and others.

This work sought to evaluate the influence of the total solid concentration and essential oil concentration on the microencapsulation of rosemary essential oil through spray drying, using maltodextrin and modified starch (1:1) as wall materials.

2. Material and methods

2.1. Materials

Tunisian rosemary (*R. officinalis* leaf oil) essential oil (Ferquima Ind. e Com. Ltda, Vargem Grande Paulista, Brazil) was used as the core material. Maltodextrin (Maltogil DE 10, Gargil, São Paulo, Brazil) and modified starch (Capsul®, National Starch Food Innovation, São Paulo, Brazil) were used as wall materials.

2.2. Microencapsulation by spray drying

A solution of maltodextrin and modified starch was prepared by dissolving these powdered materials in distilled water one day before emulsification and kept overnight in room temperature to warrant a full saturation of the polymer molecules. Rosemary essential oil was progressively added to the wall material solution while stirring at 3500 rpm for 10 min using a rotor-stator blender (Ultra-Turrax IKA T18 basic, Wilmington, USA). The emulsion was used as the feed liquid of the spray drying. The composition of the emulsion is represented in Table 1, according to experimental design. The ratio of the components used to prepare the wall materials composition was 1:1 (starch:maltodextrin) (w/w). The wall materials concentration was based on the total mass of the emulsion and the oil load was calculated based on the mass of wall materials (solids w/w). The emulsions were dried using a spray-dryer (model MSD 1.0; Labmaq do Brasil, Ribeirão Preto, Brazil) equipped with a two-fluid nozzle atomizer. The operational conditions of spray-drying consisted of air inlet temperature of 190 °C, feed rate of 0.7 L h^{-1} and atomizing air flow of 40 L min^{-1}.

Table 1
Experimental design for the spray drying tests.

Assay	Coded variables		Process variables	
	X1	X2	Wall materials (%) (w/w)	Oil load (%) (w/w)
1	−1	−1	10	10
2	−1	+1	10	30
3	+1	−1	30	10
4	+1	+1	30	30
5	−1.41	0	5.86	20
6	+1.41	0	34.14	20
7	0	−1.41	20	5.86
8	0	+1.41	20	34.14
9	0	0	20	20
10	0	0	20	20
11	0	0	20	20
12	0	0	20	20

The wall materials concentration was based on the total mass of the emulsion and the oil load was calculated based on the mass of wall materials.

2.3. Characterization of the microcapsules

2.3.1. Moisture content

The moisture content of the powder was determined gravimetrically by oven-drying at 105 °C to constant weight (Association of Official Analytical Chemists (AOAC, 2007)).

2.3.2. Wettability

Wettability of the powders was determined using the method described by Fuchs et al. (2006). One gram of powder was sprinkled over the surface of 100 mL of distilled water at 20 °C without agitation. The time taken for the powder particles to become sediment, to sink, or to become submersed and disappear from the water's surface was measured and used in a comparison of the extent of wettability between samples.

2.3.3. Solubility

The solubility of the powder was determined under procedures described by Cano-Chauca et al. (2005). The powder was 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. A 20-mL aliquot of the supernatant was transferred to pre-weighed Petri dishes and oven-dried at 105 °C overnight. The solubility (%) was calculated by the weight difference.

2.3.4. Hygroscopicity

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

2.3.5. Bulk tapped density (ρ_b) and particle density (ρ_p)

Approximately 5 g of powder were freely poured into a 25-mL glass graduated cylinder, and the samples were repeatedly tapped manually by lifting and dropping the cylinder under its own weight at a vertical distance until a negligible difference in volume between succeeding measurements was observed. Given the mass (m) and the apparent (tapped) volume (V) of the powder, the powder bulk density was computed as mV^{-1} ($g\,mL^{-1}$) (Barbosa-Canovas et al., 2005; Goula and Adamopoulus, 2008). The particle densities of powders were calculated by adopting the pycnometer method, where 2.5 g (±0.04 g) of each spray-dried powder were placed in an empty liquid pycnometer (25 mL), and filled with a measured volume of toluene. Toluene was used because it can