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Gum arabic/starch/maltodextrin/inulin as wall materials on the microencapsulation of rosemary essential oil

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ABSTRACT

The effects of the partial or total replacement of gum arabic by modified starch, maltodextrin and inulin on the characteristics of rosemary essential oil microencapsulated by spray drying were evaluated in this study. The lowest level of water absorption under conditions of high relative humidity was observed in treatments containing inulin. The wettability property of the powders was improved by the addition of inulin. The total replacement of gum arabic by modified starch or a mixture of modified starch and maltodextrin (1:1, m/m) did not significantly affect the efficiency of encapsulation, although higher T_g values were exhibited by microcapsules prepared using pure gum arabic or gum arabic and inulin. 1,8-cineol, camphor and α-pinene were the main components identified by gas chromatography in the oils extracted from the microcapsules. The particles had smoother surfaces and more folds when gum arabic or inulin was present. Larger particles were observed in the powders prepared with pure gum arabic or modified starch.

1. Introduction

Microencapsulation can potentially offer numerous benefits to the food ingredients being encapsulated. Various properties of active materials may be changed by encapsulation. For instance, handling and flow properties can be improved by converting a liquid to a powdered encapsulated form (Jafari, Assadpoor, He, & Bhandari, 2008). The main compounds of the essential oils, responsible for the flavour and the functional properties, are volatiles and chemically unstable in the presence of oxygen, moisture and heat. Stability of essential oils can be enhanced through microencapsulation by spray drying. Spray drying is the most commonly used encapsulation technique in the food industry (Ré, 1998; Reineccius, 2004). The process of spray drying is economical and flexible, uses equipment that is readily available, and produces powder particles of good quality (Jafari et al., 2008). Some examples of encapsulated flavours are oregano essential oil using gum arabic, maltodextrin and modified starch as wall materials (Botrel et al., 2012), oregano, citronella and marjoram flavours encapsulated into milk protein-based matrices (Baranauskiené, Venskutonis, Dewettinck, & Verhé, 2006) and cardamom oleoresin microencapsulated using gum arabic and modified starch (Krishnan, Krishirsagar, & Singhal, 2005).

Flavours are essential to some foods, play important roles in consumer satisfaction and can promote the consumption of those products. However, the stability of flavours in foods, especially in food processing, has attracted intense attention due to its relationship to the quality and acceptability (Jun-Xia, Hai-Yan, & Jian, 2011). *Rosmarinus officinalis* L. is widely accepted as one of the herb spices with the strongest aroma and highest antioxidant activity. Rosemary has also been extensively studied due to its antimicrobial activity (Genena, Hense, Smânia Junior, & Souza, 2008). Gachkar et al. (2007) reported that antibacterial, antioxidant and free radical-scavenging activities were exhibited by rosemary essential oil extracted using steam distillation.

The selection of a suitable wall material is critical to a microencapsulation spray-drying process that avoids changes due to oxidation, chemical interactions or volatilisation (Botrel et al., 2012) and maximises the retention of the essential oil after the drying process is completed. Research on microencapsulation spray drying has concentrated on improving the encapsulation efficiency and extending the shelf life of the product to produce high quality encapsulated powders. The main factors that affect the encapsulation efficiency are the properties of the wall and core materials (Gharsallaoui, Roudaut, Chambin, Voilley, & Saurel, 2007; Goula & Adamopoulos, 2012; Jafari et al., 2008; Reineccius, 2004).

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In this context, it is of utmost importance to compare the effects of different carriers on the properties of the spray-dried essential oils. Gum arabic, which it is one of the most common wall materials used in microencapsulation by spray drying, is a polymer consisting of D-glucuronic acid, L-rhamnose, D-galactose and L-arabinose, including approximately 2% protein (Dickinson, 2003) and can produces stable emulsions with most oils (Gharsallaoui et al., 2007; Jafari et al., 2008; Reineccius, 2004). Capsul® is a starch that is chemically modified by incorporating a lipophilic component aimed at conferring emulsifying properties (Rocha, Fávaro-Trindade, & Grosso, 2012). When modified with octenyl succinic anhydride, the normally hydrophilic starch gains hydrophobic elements in the form of octenyl groups, resulting in molecules with an amphiphilic character (Sweedman, Tizzotti, Schäfer, & Gilbert, 2013). This modification gives the material an excellent capacity for retaining volatiles during atomisation in a spray-dryer (Rocha et al., 2012). Maltodextrin is a hydrolysed starch produced by partial hydrolysis of starch with acid or enzymes that is commonly used as the wall material in microencapsulation of food ingredients (Gharsallaoui et al., 2007; Goula & Adamopoulos, 2012). It offers advantages such as relatively low cost, neutral aroma and taste, low viscosity at high solids concentrations and good protection of flavours against oxidation. However, the greatest limitation of this wall material is its low emulsifying capacity and marginal retention of volatiles (Buffo & Reineccius, 2000; Krishnan et al., 2005), so it is generally used in mixtures with other wall materials. Inulin is composed of fructose units with β(2-1) links with glucose at the end of the chain (Bakowska-Barczak & Kolodziejczyk, 2011) and, as the maltodextrin, it is an interesting encapsulating agent. It is a dietary fibre which shows prebiotic effects and can improves calcium bioavailability (Robert, García, Reyes, Chávez, & Santos, 2012).

Although gum arabic is considered to be an excellent wall material, several problems are associated with the use of this material for microencapsulation, including its high cost and limited supply (Kanakdande, Bhosale, & Singhal, 2007; Krishnan et al., 2005). Thus, the search for complete or partial substitutes for gum arabic has been encouraged. For instance, modified starch and maltodextrin have become a viable alternative to gum arabic because they are abundant and low-cost materials (Peng, Li, Guan, & Zhao, 2013) and inulin is beneficial for human health. There are no published works reporting the effect of different types of wall materials, particularly inulin, on the encapsulation efficiency, retention of volatiles and the particle properties of microencapsulated rosemary essential oil. This study evaluated the effects of gum arabic, modified starch, maltodextrin and inulin on the microparticles' properties, such as the oil retention, product stability, retention of volatiles and the morphology and size of the powders.

2. Materials and methods

2.1. Materials

Tunisian rosemary (*R. officinalis* leaf oil) essential oil (Ferquima, Vargem Grande Paulista, Brazil) was used as the core material. Gum arabic (Colloides Naturels Brasil, São Paulo, Brazil), high-performance inulin (degree of polymerisation > 23, Orafti®HP, BENEO-Orafti, Tienen, Belgium), 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. Experimental design

The experiments were conducted in a completely randomised design with three replications, as shown in Table 1. An analysis of variance was performed using Statistica software (ver.8, Stat Soft. Inc., Tulsa, USA) to evaluate the effects of the six encapsulation formulations on the characteristics of powders of microencapsulated rosemary essential oil. The differences between the mean values obtained were analysed using the Tukey test at 5% probability ($p < 0.05$).

Table 1
Composition of the wall materials for the each treatment used as a feed solution for the spray-drying process.

#	Wall material ($g\,100\,g^{-1}$ of solution)				Core material ($g\,100\,g^{-1}$ of solution)
	Gum arabic (GA)	Modified Starch (MS)	Maltodextrin (MD)	Inulin (IN)	Rosemary essential oil
1	20.0	–	–	–	5.0
2	–	20.0	–	–	5.0
3	–	10.0	10.0	–	5.0
4	–	10.0	–	10.0	5.0
5	10.0	–	10.0	–	5.0
6	10.0	–	–	10.0	5.0

2.3. Preparation of emulsions

The wall materials were dissolved in distilled water with stirring. The solutions were prepared one day before being emulsified and were kept overnight at room temperature to ensure full saturation of the polymer molecules. Then, rosemary essential oil was gradually added to the wall material solution with stirring at 3500 rpm over 10 min, using a rotor–stator blender (Ultra-Turrax IKA T18 basic, Wilmington, USA), to form an emulsion. The emulsion was used as the feed liquid for the spray-drying process. For each treatment, approximately 1 L of sample was prepared for the production of microencapsulated powders. The wall material concentration was 20% (Fernandes, Borges, & Botrel, 2013a; Fernandes et al., 2013b) and the amount of rosemary essential oil used was 25% of the mass of the wall materials (Jafari et al., 2008).

2.4. Microencapsulation by spray drying

The emulsions were dried using a spray-dryer (model MSD 1.0; Labmaq do Brasil, Ribeirão Preto, Brazil) equipped with a two-fluid nozzle atomiser. The following operational conditions were used, as described in previous studies: inlet temperature of 170 °C and feed rate of $0.9\,L\,h^{-1}$ (Fernandes et al., 2013a, 2013b). The dried powder was collected and stored in opaque airtight containers at 4 °C until further analysis.

2.5. Characterisation of the microcapsules

2.5.1. 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).

2.5.2. Wettability and solubility

The 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 sediment, sink, be submersed and disappear from the water's surface was recorded and used for a comparison of the extent of wettability of the samples.

The solubility of the powders was evaluated according to the method proposed by Cano-Chauca, Stringheta, Ramos, and Cal-Vidal (2005), with modifications. The powders were weighed (1 g)