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Encapsulation efficiency and oxidative stability of flaxseed oil microencapsulated by spray drying using different combinations of wall materials

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ABSTRACT

This study aimed at evaluating the potential of maltodextrin combination with different wall materials in the microencapsulation of flaxseed oil by spray drying, in order to maximize encapsulation efficiency and minimize lipid oxidation. Maltodextrin (MD) was mixed with gum Arabic (GA), whey protein concentrate (WPC) or two types of modified starch (Hi-Cap 100™ and Capsul TA®) at a 25:75 ratio. The feed emulsions used for particle production were characterized for stability, viscosity and droplet size. The best encapsulation efficiency was obtained for MD:Hi-Cap followed by the MD:Capsul combination, while the lowest encapsulation efficiency was obtained for MD:WPC, which also showed poorer emulsion stability. Particles were hollow, with the active material embedded in the wall material matrix, and had no apparent cracks or fissures. During the oxidative stability study, MD:WPC combination was the wall material that best protected the active material against lipid oxidation.

1. Introduction

There is an increasing demand for nutritive and healthy foods in the market and this fact has led the food industry to focus their research in products of this nature. Flaxseed oil is a polyunsaturated oil extracted from the flax plant (*Linum usitatissimim*) rich in α-linolenic acid (ALA), the essential fatty acid omega (ω)-3, which represents about 57% of its total fatty acids. The high content of n-3 fatty acid present in this oil allows the attribution of functional food, which means that besides the nutritional functions, its consumption may have beneficial effects on health (Vaisey-Genser and Morris, 2003).

On the other hand, one of the major problems associated with oils rich in polyunsaturated fatty acids (PUFAs) is their high susceptibility to oxidative deterioration and consequent production of undesirable flavor. Thus, there is a need to protect these oils in order to make them more stable during handling, processing and storage (Augustin et al., 2006).

Spray drying is a process widely used for microencapsulation of oils and flavours (Fuchs et al., 2006; Ahn et al., 2008; Bae and Lee, 2008; Partanen et al., 2008). It results in powders with good quality, low water activity, easier handling and storage and also protects the active material against undesirable reactions. Both wall material selection and emulsion properties (stability, viscosity and droplet size) can affect the process efficiency and the microencapsulated product stability. A successful microencapsulation must result in a powder with minimum surface oil and maximum retention of the active material.

Gum Arabic is one of the most common wall materials used in microencapsulation by spray drying. Although it presents many desirable characteristics to be a good encapsulating agent (high solubility, low viscosity and good emulsifying properties), the oscillation in supply, as well as the increasing prices, is leading researches to look for alternative wall materials that could replace it or be used in combination with it (Charve and Reineccius, 2009).

Maltodextrin is a hydrolyzed starch commonly used as wall material in microencapsulation of food ingredients (Gharsallaoui et al., 2007). It offers advantages such as relatively low cost, neutral aroma and taste, low viscosity at high solids concentrations and good protection against oxidation. However, the biggest problem of this wall material is its low emulsifying capacity. Therefore, it is desirable to use maltodextrin in combination with other surface active biopolymers, such as gum Arabic (Fernandes et al., 2008; Bule et al., 2010), modified starches (Soottitantawat et al., 2003; Bule et al., 2010) and proteins (Hogan et al., 2003; Bae and Lee, 2008) in order to obtain an effective microencapsulation by spray drying.

The selection of wall material combinations affects both the emulsion properties and the particles' characteristics after drying

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and during storage. It is well described that emulsion characteristics such as stability, viscosity, droplets size, as well as powder properties such as surface oil, particle size, density, morphology and oxidative stability, are influenced by the type of encapsulating agent used (Jafari et al., 2008b).

Most of the works found in the literature on microencapsulation of PUFA-rich oils uses fish oil as active material (Hogan et al., 2003; Augustin et al., 2006; Serfert et al., 2009; Anwar and Kunz, 2011).

Fish oil and flaxseed oil are very rich in polyunsaturated fatty acids, but they do not have the same composition. Some works found in literature have reported the microencapsulation of fish oil containing approximately 27% (Serfert et al., 2009), 29% (Aghbashlo et al., 2012), or 33% (Drusch et al., 2009b) of omega-3 fatty acids, while the flaxseed oil contains bout 53% linolenic acid (Tonon et al., 2011). Therefore, even though they could show a similar tendency when microencapsulated, it is not possible to assure they will present the same behavior during spray drying and storage.

Very little information is available on microencapsulation of flaxseed oil (Partanen et al., 2008; Omar et al., 2009; Tonon et al., 2011) and none of the published works reported the influence of different types of wall materials on the encapsulation efficiency and oxidative stability of this oil.

The objective of this work was to evaluate the potential of maltodextrin combination with four types of wall materials (gum Arabic, whey protein concentrate and two types of modified starches), as alternative materials for microencapsulation of flaxseed oil by spray drying. The feed emulsions were characterized for stability, viscosity and droplet size, while the microcapsules were characterized for encapsulation efficiency, moisture content, particle size, bulk density, morphology and oxidative stability.

2. Material and methods

2.1. Material

Flaxseed oil (Lino Oil, Paranambi, Brazil) was used as active material with the following fatty acid composition: 5.77% 16:0, 4.57% 18:0, 21.11% 18:1, 14.30% 18:2 and 53.35% 18:3. The wall materials used were: maltodextrin MOR-REX® 1910 with 10 DE (Corn Products, Mogi Guaçu, Brazil) (MD), whey protein concentrate WPC 80® (Alibra Ingredients, Campinas, Brazil) (WPC), gum Arabic Instantgum BA® (Colloids Naturels CNI, São Paulo, Brazil) (GA) and two chemically *n*-octenyl succinic andydrid (OSAN)-modified starches: Capsul TA® (derived from Tapioca starch) and Hi-Cap 100™ (derived from waxy maize) (National Starch, São Paulo, Brazil).

2.2. Emulsion preparation

The wall materials were added to distilled water at 25 °C and the mixture was stirred until completely dissolved. The total solid concentration (wall material + oil) was fixed at 30%. Flaxseed oil was then added to the wall material solution at a concentration of 20% with respect to total solids (Ahn et al., 2008; Jafari et al., 2008a; Charve and Reineccius, 2009). Emulsions were formed using an Ultra-Turrax homogenizer MA-102 (Marconi, Piracicaba, Brazil) operating at 18,000 rpm for 5 min.

2.3. Emulsion characterization

2.3.1. Emulsion stability

Immediately after the emulsion preparation, 25 mL aliquots of each sample were transferred to graduated cylinders of 25 mL, sealed, stored at room temperature for one day, and the volume of the upper phase measured after 24 h. The stability was measured by % of separation and expressed as:

$$\%\,\text{Separation} = \left(\frac{H_1}{H_0}\right) \times 100 \quad (1)$$

Where: H_o represents the emulsion initial height and H_1 is the upper phase height.

2.3.2. Emulsion viscosity

Emulsion viscosity was measured thought the determination of steady-shear flow curves using a Physica MCR301 Rheometer (Anton Paar, Graz, Austria). Measurements were made in triplicate, using stainless steel plate-plate geometry with a diameter of 75 mm and a gap of 0.2 mm. Temperature was controlled at 25 °C by a Peltier system. Rheograms were analyzed according to empirical models and the emulsions viscosity was calculated as the relationship between shear stress and shear rate.

2.3.3. Emulsion droplet size

The droplet size distribution was measured using a laser light diffraction instrument, Mastersizer S (Malvern Instruments, Malvern, UK). A small sample was suspended in water using magnetic agitation, and the droplet size distribution was monitored during each measurement until successive readings became constant. The emulsion droplet size was expressed as D_{32}, the Sauter mean diameter.

2.4. Microencapsulation by spray drying

Spray drying process was performed in a laboratory scale spray dryer Lab Plant SD-05 (Huddersfield, England), with a nozzle atomization system with 0.5 mm diameter nozzle and main spray chamber of 500 × 215 mm. The emulsions were fed into the main chamber through a peristaltic pump and the feed flow rate was controlled by the pump rotation speed. Drying air flow rate was 73 m^3/h and compressor air pressure was 0.06 MPa. Inlet and outlet air temperature were 180 ± 2 and 110 ± 2 °C, respectively, and feed flow rate was 12 ± 2 g/min.

2.5. Powders analysis

2.5.1. Encapsulation efficiency

Encapsulation efficiency (*EE*) was determined according to the method described by Bae and Lee (2008). Fifteen milliliters of hexane were added to 1.5 g of powder in a glass jar with a lid, which was shaken by hand for the extraction of free oil, during 2 min, at room temperature. The solvent mixture was filtered through a Whatman filter paper n° 1 and the powder collected on the filter was rinsed three times with 20 mL of hexane. Then, the solvent was left to evaporate at room temperature and after at 60 °C, until constant weight. The non-encapsulated oil (surface oil) was determined by mass difference between the initial clean flask and that containing the extracted oil residue (Jafari et al., 2008b). Total oil was assumed to be equal to the initial oil, since preliminary tests revealed that all the initial oil was retained, which was expected, since flaxseed oil is not volatile. Encapsulation efficiency (EE) was calculated from Eq. (2).

$$EE = \left(\frac{TO - SO}{TO}\right) \times 100 \quad (2)$$

where *TO* is the total oil content and *SO* is the surface oil content.