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Influence of wall matrix systems on the properties of spray-dried microparticles containing fish oil

Diego Alvarenga Botrel [a,*], Regiane Victória de Barros Fernandes [a,b], Soraia Vilela Borges [a], Maria Irene Yoshida [c]

[a] Food Science Department, Federal University of Lavras, 37200-000 Lavras, MG, Brazil
[b] Institute of Agricultural Sciences, Campus de Rio Paranaíba, Federal University of Viçosa, 38810-000 Rio Paranaíba, MG, Brazil
[c] Chemistry Department, Federal University of Minas Gerais, 31270-901 Belo Horizonte, MG, Brazil

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ABSTRACT

This study was conducted to evaluate the effects of the partial substitution of whey protein isolate (WPI) by inulin (IN) or maltodextrin (MD) as wall materials on the characteristics of microparticles containing fish produced by spray drying. Three treatments, WPI, WPI/MD (1:1) and WPI/IN (1:1), were evaluated in a completely randomised design with three replicates. The solubility and hygroscopicity of the particles were not affected by the wall material, attaining average values of 79% and 5.5 $g \cdot 100\ g^{-1}$, respectively. The partial substitution of WPI by inulin improved the wettability properties of the powders and reduced the occurrence of surface oil on the particles. The surface oil content was 5.6%, 6.5% and 7.7% for the particles produced using WPI/IN, WPI/MD and WPI, respectively. The particles presented smoother surfaces, with a smaller number of folds, when inulin was present. The GAB model was chosen as the model best adjusted to the isotherms, with values of moisture content on the monolayer (X_m) equal to 0.036 $g \cdot g^{-1}$, 0.026 $g \cdot g^{-1}$ and 0.074 $g \cdot g^{-1}$ for the WPI, WPI/MD and WPI/IN microparticles, respectively. All of the powders obtained from these treatments exhibited a no crystalline structure, and the glass transition temperatures of these anhydrous powders were 168 °C, 149 °C and 131 °C for the WPI, WPI/MD and WPI/IN microparticles, respectively. The use of inulin and maltodextrin proved to be good alternative secondary wall materials for fish oil together with WPI.

1. Introduction

The supplementation of food with omega-3 fatty acids, particularly docosahexanoic acid (DHA) and eicosapentaenoic acid (EPA), has attracted much attention in the food industry. Fish oil contains a high concentration of $n-3$ polyunsaturated fatty acids (PUFAs), which have documented beneficial effects on human health, including preventing cardiac arrhythmia and sudden death from myocardial infarction and reducing serum triacylglycerides (Drusch, Serfert, Scampicchio, Schmidt-Hansberg, & Schwarz, 2007). However, the incorporation of PUFAs in processed foods is limited by their low solubility, "fishy" flavour, poor handling properties and susceptibility to oxidation (Aghbashlo, Mobli, Madadlou, & Rafiee, 2012; Wang, Tian, & Chen, 2011). Encapsulation of fish oil can render it suitable for addition to food by giving to this ingredient powdery flow characteristics.

Encapsulation is a technique by which solids and liquids are enclosed in microcapsules that can release their contents at controlled rates under specific conditions. Encapsulation, by enclosing compounds within an edible coating material, protects bioactive compounds from environmental factors, retards the evaporation of a volatile core, controls the rate at which the encapsulant leaves the microcapsule, improves the handling properties of a sticky or oily material, allows easy storage and transportation, masks the taste or odour of the core material and protects a reactive core from chemical attack (Aghbashlo et al., 2012; Fang & Bhandari, 2010). Spray drying is the encapsulation technique most commonly employed in the food industry. The process is economical and flexible, uses equipment that is readily available, and produces powder particles of good quality (Jafari, Assadpoor, Bhandari, & He, 2008a). However, the use of relatively high working temperatures during the drying process may induce heat degradation and alter thermosensitive products.

Dry solid particles are obtained by hot-air drying liquid droplets produced at the top of the chamber. Drying occurs during their descent to the bottom of the chamber due to their contact with flowing dry air. The residence time of the particles in the hot air is very short (Turchiuli et al., 2005).

Numerous wall materials have been studied for their suitability as wall materials in spray drying. The wall materials for encapsulating oils should have emulsifying properties, film-forming and drying properties, high water solubility and low viscosity (Bae & Lee, 2008; Kagami et al., 2003; Ré, 1998). The ability of particles to mix with water is one of the most important properties of reconstitution (Bae & Lee, 2008). The

* Corresponding author. Tel.: +55 35 38291391.
E-mail address: diegobotrel@dca.ufla.br (D.A. Botrel).

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choice of wall material is crucial for the production of microparticles by spray drying because it has a strong influence on the emulsion properties before drying, retention of the core during the process and shelf-life of the powder after drying (Jafari et al., 2008a). Typical wall materials include proteins and carbohydrates. Furthermore, the incorporation of hydrolysed carbohydrates into wall systems has been shown to improve the drying properties of the wall matrix, most likely by enhancing the formation of a dry crust around the drying droplets and increasing the oxidative stability by reducing oxygen permeability (Kagami et al., 2003; Sheu & Rosenberg, 1998).

Whey proteins have been shown to be an excellent encapsulating agent for oils/fats and volatiles (Bae & Lee, 2008; Kagami et al., 2003). Proteins have been widely used for stabilising food emulsions. Proteins have been studied mostly in combination with carbohydrates for dried emulsions (Baik et al., 2004; Kagami et al., 2003; Keogh et al., 2001). Hydrolysed starch is generally added as a secondary wall material to improve the drying properties (Bae & Lee, 2008). However, these carbohydrates cannot generally be used as wall materials in the absence of a surface-active wall constituent because they lack emulsification properties (Bangs & Reineccius, 1988). Inulin is a natural storage carbohydrate found in chicory roots, Jerusalem artichoke, yacon tubers, wheat, asparagus and onions (Castro, Céspedes, Carballo, Bergenståhl, & Tornberg, 2013; Ronkart et al., 2006). It is a mixture of polysaccharides composed of fructose unit chains (linked by β-(2 → 1) D-fructosyl-fructose bonds) of various lengths, generally terminated by a single glucose unit (Ronkart et al., 2007). The inulin from chicory roots is commercialised as a purified food ingredient. Among the several commercial inulin types available, all have very high purity and they differ with regard to their powder characteristics and carbohydrate composition. Standard inulin, as it is extracted from chicory roots, always contains a small amount of sugars (up to 10%) (Coussement, 1999). In particular, this taste-free fructan increases the stability of foams and emulsions and offers a wide range of health benefits, such as effects on intestinal function and health, increased mineral absorption, reduction of the risk of colon cancer and the modulation of appetite (Franck & Bosscher, 2009). Inulin can be an alternative to carbohydrates as a secondary wall material for spray-drying food components.

Efforts have been made to develop new materials and formulations for spray drying process. The main objective of this work was to evaluate different wall matrix systems in the production of fish oil microparticles using spray drying technology. This study also investigated the partial substitution of whey protein isolate as the primary wall material by maltodextrin and inulin and its effects on the physical and handling properties of the produced powders. Maltodextrin is a common wall material used in spray drying processes in the food industry. However, inulin was not yet fully studied for this type of application and can be an interesting alternative for this purpose.

2. Materials and methods

2.1. Materials

Fish oil (Sundown Naturals, Boca Raton, USA), used as core material, presents 18% of eicosapentanoic acid (EPA) (C20:5) and 12% of docosahexanoic acid (DHA) (C22:6) in its compositions, as indicated by the supplier. Whey protein isolate (WPI) (minimum of 90% of protein) (Alibra Ingredientes Ltda, Campinas, Brazil), maltodextrin (MD) (molecular weight ~1800) (Maltogil-DE10, Cargil, São Paulo, Brazil) and inulin (IN) (molecular weight ~1980) (with the degree of polymerization higher than 10) (Orafti®GR, BENEO-Orafti, Tienen, Belgium) were used as wall materials.

2.2. Preparation of emulsions and spray drying

Wall material solutions were prepared by dissolving WPI, MD and IN in distilled water for each formulation. The solutions were prepared on the day before emulsification and maintained overnight at room temperature to ensure the full hydration of the polymer molecules. 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 wall material composition varied according to the treatments (Table 1). The mass ratio of oil to wall material was maintained at 1:3 (w/w); a typical core to wall material ratio around of 1:4 is mostly applied, according to the literature (Jafari et al., 2008a). The percentage of solids (wall material) used in the feed solution was 15% (w/w) for all of the treatments and was within the wall material concentration recommended for oil encapsulation, which is 10% to 30% (Rosenberg & Young, 1993). For each treatment, approximately 1 L of sample was prepared for the production of the microparticles in one-batch 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 atomiser. The following process variables were used: inlet air temperature of 180 °C, which leads to the rapid formation of a semi-permeable membrane, which is desirable (Jafari et al., 2008a), feed flow rate of 0.85 $L \cdot h^{-1}$ and atomising air flow of 40 $L \cdot min^{-1}$.

2.3. Rheological parameters

The rheological measurements were conducted using a concentric cylinder viscosimeter (Brookfield DVIII Ultra, Brookfield Engineering Laboratories, Stoughton, MA, USA), a cylindrical sample chamber 13R/RP (19.05-mm diameter and 64.77-mm deep), and a SC4-18 spindle (17.48-mm diameter and 35.53-mm long). For each test, the temperature of the filled sample cup (6.7 mL) and spindle were equilibrated (at 25 °C). Flow curves were obtained at shear rates of 0.1–330 s^{-1}. Three experimental runs were performed for each material, and the resulting shear stress was the average of three experimental values. The shear stress and shear rate data were obtained using Rheocalc software (version V3.1; Brookfield Engineering Laboratories, MA, USA). The power law (Eq. (1)) was used to analyse the flow properties of the emulsified samples, as follows.

$$\sigma = k \cdot \gamma^n \tag{1}$$

where σ = the shear stress (Pa), γ = the shear rate (s^{-1}), k = the consistency index ($Pa \cdot s^n$), and n = the flow behaviour index (Bourne, 2002). The consistency coefficient provides an indication of the flow properties of the mixture and the flow behaviour index indicates how close the mixture is to Newtonian fluid behaviour.

2.4. Particle size distribution and morphology

Particle size distribution was measured using a laser light diffraction instrument, Mastersizer 2000, model Hydro 2000 MU (Malvern Instruments, Worcestershire, UK). A small powder sample was suspended in isopropyl alcohol under agitation, and the particle size distribution was monitored during each measurement until successive readings became constant. The volume-weighted mean diameter (D[4,3]) was

Table 1
Wall material composition of each treatment used as the feed solution in the spray drying process.

#	Wall material ($g \cdot 100\ g^{-1}$ of solution)			Core material ($g \cdot 100\ g^{-1}$ of solution)
	WPI	Maltodextrin (MD)	Inulin (IN)	Fish oil
1	15.0	–	–	5.0
2	7.5	7.5	–	5.0
3	7.5	–	7.5	5.0