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The use of arabic gum, maltodextrin and surfactants in the microencapsulation of phytosterols by spray drying

Carla Agustina Di Battista *, Diana Constenla, María Verónica Ramírez-Rigo, Juliana Piña

Planta Piloto de Ingeniería Química (PLAPIQUI), Universidad Nacional del Sur, CONICET, Camino La Carrindanga KM 7, CP: 8000 Bahía Blanca, Argentina

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ABSTRACT

The addition of phytosterols in aqueous-based food matrices is challenging because of their poor physicochemical properties (non-water soluble and hydrophobic powder). By using spray drying, phytosterols microparticles were formulated and developed in this work. Arabic gum, maltodextrin and one of two different surfactants were thoroughly studied as wall materials. Increasing concentrations of Tween 20 (T20) or sodium lauryl sulfate (SDS), from 0.1 to 2.65% w/v, were evaluated. The feed suspension characteristics (viscosity, interfacial properties and particle size distribution), process yield (PY), encapsulation efficiency (EE), phytosterols retention (R) and size of the microparticles were analyzed. The presence of surfactants in the suspension to be spray dried has significant effects on the studied responses. T20 led to process yields around 65% (2% w/v surfactant concentration). On the other hand, the microparticles obtained using 2% w/v of SDS were the best in terms of EE (about 50%), R (close to 40%) and particle size (5.89 μm), being the PY acceptable (almost 55%). According to the open literature, which indicates that average particle sizes lower than 25 μm favor the phytosterols bioavailability, the microparticles obtained in this work are promising for phytosterols delivery.

1. Introduction

Phytosterols (PS) are vegetable sterols with a similar structure to cholesterol, which cannot be absorbed into the blood stream but are widely recognized as lowering absorption of cholesterol and their serum levels [1]. It has been found that PS exert their hypocholesterolemic effect if they are dispersed [2]. Indeed, the PS must be administered finely divided in order to facilitate their exposure to the bile salts, and preferably in particles smaller than 50 μm to reduce the sandy mouth feel [3]. Furthermore, it has been demonstrated that average particle sizes about 25 μm favor the incorporation of PS into the micellar phase in the intestine improving the bioavailability [2].

Phytosterols and their derivates (stanols, esters and stanol-esters) have been included in fat- or oil-based foods products, which are clearly restricted in diets for hypercholesterolemia [3]. Therefore, the incorporation of PS in aqueous-based formulations (like beverages, soups and others) is an attractive field of application. The hydrophobic and water insoluble nature of PS, which make them poor candidates for stable dispersions, hinder their applicability on intermediate or final aqueous-based products [4].

Several authors focused on particle size reduction to improve the phytosterols dispersibility. Among others, the following techniques have been investigated: a) dry milling of cooled material (e.g., in air mill, air attrition mill, high energy hammer mill, impact mill [2,5]); b) high pressure homogenization of mixtures or dispersions including emulsifiers [6–8]; and c) high pressure homogenization or shearing of melted material [6,9,10]. However, all these techniques are complex and time- and energy-consuming because they require more than one step (homogenization or milling, including cooling or heating) to obtain the desired particle size. Furthermore, for solid phytosterols, several abrasive effects of the homogenizer valves or parts of the milling equipment have been found [6,9,11].

Microencapsulation is a common technique used to provide a physical barrier between the active ingredient and the other components of the product [12]. Among other methods, *spray drying* and *spray chilling* have been successfully applied for encapsulation of food ingredients because it allows producing particles of high quality and stability by means of relatively flexible, simple, low-cost and continuous processes [13,14]. Spray drying consists in the atomization of a solution or liquid suspension into tiny drops, followed by drying in a stream of hot air to produce solid microparticles [15]. On the other hand, spray chilling involves the atomization of a hot melt fluid (solution or suspension) into a cooled chamber to obtain the solid product [16].

To the best of our knowledge, only Alvim et al. [17] studied the microencapsulation of phytosterols by spray chilling using a lipid mixture

Abbreviations: PS, phytosterols; AG, arabic gum; MD, maltodextrin; HLB, hydrophilic–lipophilic balance; SDS, sodium lauryl sulfate; T20, polysorbate Tween 20; PSD, particle size distribution; PY, process yield [%]; EE, encapsulation efficiency [%]; TP, total content of phytosterols [%]; FP, free phytosterols [%]; R, phytosterols retention [%]; D[3,2], Sauter mean [μm]; DSC, differential scanning calorimetry; XRD, X-ray diffraction.

* Corresponding author.
E-mail addresses: adibattista@plapiqui.edu.ar (C.A. Di Battista), dconstenla@plapiqui.edu.ar (D. Constenla), vrrigo@plapiqui.edu.ar (M.V. Ramírez-Rigo), julianap@plapiqui.edu.ar (J. Piña).

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of low trans-hydrogenated vegetable fat and stearic acid as wall material. The authors analyzed the effect of the ratio of the wall components on the particle size (between 13.8 and 32.2 μm) and morphology. Non data of the process yield, encapsulation efficiency, feed properties and additional product features were reported. Moreover, the wall includes a fatty and trans-material, being not adequate to produce water-dispersible microparticles.

Regarding the microencapsulation of PS by spray drying, very few publications are available in the open literature (i.e., just two patents [3,4]). Auweter et al. [4] proposed the dissolution of PS in an organic solvent (like acetone), followed by the dispersion of the mixture into an aqueous matrix of Na-caseinate and modified starch. After solvent removal, the dispersion was spray dried to obtain a powder product. The mayor disadvantage of this proposal is the use of solvents, which negatively affect the product healthiness and production costs (i.e., a solvent removal step is required). On the other hand, Auriou [3] proposed the creation of micelles comprising PS and surfactants with HLB between 8 and 18 (as sucro-ester) in an aqueous medium, followed by coating of the micelles with starches (a mixture of octenylsuccionate and corn starch) in a spray-drying step, leading to particle sizes between 10–100 μm.

Although these contributions are interesting, none of them covers completely the rational design of a particulate system containing PS with adequate bioavailability and consumer acceptability (particle size lower than 25 μm) by organic solvent free-microencapsulation via spray drying. In fact, the relationships between operating variables, feed composition, process performance and product quality have not been studied. It is well-known that feed formulation is one of the key steps in microencapsulation by spray drying [12].

Mixtures of arabic gum and maltodextrin have been broadly used as wall materials in the microencapsulation by spray drying of many food ingredients [12]. However, no evidences of the use of this mixture for encapsulation of insoluble and waxy solids have been found. Moreover, the research field of microencapsulation of non-water soluble solids has not been widely explored.

Phytosterols processing by spray drying requires stabilized feed emulsions or suspensions. Emulsification implies the use of high temperatures (above the PS melting point, i.e., ≈136 °C) [9,18] or esterified PS (which have lower melting points) [19–21]. However, high temperatures could negatively affect the PS oxidative stability while esterified PS need to be hydrolyzed in order to inhibit cholesterol absorption [22]. On the other hand, the stabilization of aqueous suspensions can be achieved by adding surfactants [23]. Indeed, the use of surfactants in spray drying has proved positive effects; among others, improved stability of the dispersions to be spray dried and, thus, increased encapsulation efficiency [24]. Furthermore, the addition of surfactants tends to diminish the surface tension of the continuous phase, promoting particle disaggregation in the disperse phase [25].

In this context, the aim of this work is to rationally study the microencapsulation of phytosterols using a mixture of arabic gum and maltodextrin as wall materials. Particularly, the effect of the addition of two different surfactants, nonionic (Tween 20) or anionic (Sodium lauryl sulfate), on the process yield, encapsulation efficiency, phytosterols retention and product particle size is evaluated. Complementarily, important parameters of the feed formulation are analyzed: wetting of phytosterols by the wall solutions (containing the wall materials and the surfactant), viscosity of the feed suspensions and size of the particles in these feed suspensions.

2. Materials and methods

2.1. Materials

Phytosterols powder and arabic gum were supplied by Grupo Saporiti (Buenos Aires, Argentina). The PS powder consisted in a mixture of β-sitosterol (35–55% w/w), campesterol (18–27% w/w), stigmasterol (21–35% w/w) and about of 0–7% w/w of other vegetable sterols. According to X-ray diffraction (XRD) and differential scanning calorimetry (DSC) measurements previously performed, pure phytosterols present a crystalline structure and a melting point around 136 °C (see Supplementary data, Figures S1 and S2, respectively). The particle size distribution of raw material was measured by laser diffraction (see Section 2.2.2.3), being the Sauter mean 46.4 ± 1.1 μm.

Maltodextrin Globe® 019150 (dextrose equivalent, DE 15) was supplied by Todo Droga (Córdoba, Argentina). The phytosterols, arabic gum (AG) and maltodextrin (MD) were food grade. The pro-analysis grade surfactants, sodium lauryl sulfate (SDS) (HLB 40, molecular weight 289) and polysorbate Tween 20 (T20) (HLB 16, molecular weight 1228) were supplied by Cicarelli® Reagents S.A. (Santa Fe, Argentina).

2.2. Methods

2.2.1. Liquid feed preparation

The liquid feed to the spray dryer was prepared by dissolution of the wall materials and the surfactant in distilled water, followed by dispersion of the PS powder and homogenization. Table 1 shows the *feed suspensions* composition for all the studied cases: reference feed (*FR*, without surfactant), feeds comprising T20 in different concentrations (*FT1* to *FT6*) and feeds containing SDS in different concentrations (*FS1* to *FS6*).

Briefly, 15 g of arabic gum and 5 g of maltodextrin were dispersed in 100 mL of hot distilled water (50 °C) under magnetic stirring, until complete dissolution and hydration (about 30 min). Then, the surfactant (SDS or T20) was added to form the *wall solutions* (Table 1). Finally, 6.66 g of PS were dispersed under continuous agitation for 1 h. Afterward, the aqueous suspensions to be spray dried were homogenized using a Pro II Homogenizer over 9 min at 25,000–35,000 rpm and room temperature. These conditions and the AG, MD and PS contents in the suspensions were selected based on previous exploratory experiments.

2.2.2. Liquid feed characterization

2.2.2.1. Contact angle between phytosterols and wall solutions. For each wall solution (aqueous solution of AG, MD and surfactant; see Table 1) the contact angle on phytosterols was determined by the sessile drop method; i.e., by tangential observation of a tiny solution droplet (20 μL) that was placed over a glass plate, which was previously coated with a thin layer of phytosterols. The phytosterols layer was prepared by dissolution of PS powder in hexane, followed by deposition of this solution on the entire surface of the glass plate. The high volatility of hexane facilitated the formation of a thin and uniform solid PS layer. The wetting experiments were performed at room temperature in a Krüss DSA Mk2 goniometer equipped with image analysis software (Drop Shape Analysis, Krüss GmbH, Germany). The contact angle was measured at the initial state (t = 0 min) and 5 min after the drop deposition. The assays were performed in duplicate.

2.2.2.2. Viscosity of the feed suspensions. The viscosity of the suspensions was determined with a controlled-stress rheometer Physica MCR 301 Anton Paar (Ostfildem, Germany) at room temperature and shear rates from 0 to 1000 s^{-1}. A coaxial-cylinder geometry (CC27-SN16635) was used. The viscosity was calculated from the steady-shear flow curves, as the ratio between shear stress and shear rate. All measurements were performed in duplicate.

2.2.2.3. Particle size distribution of the feed suspensions. The particle size distribution (PSD) of the homogenized suspensions was measured by laser light diffraction using a Horiba LA-950 V2 device (Irvine, United States). Average particle size was expressed as D[3,2], i.e., the Sauter