Comparison of spray drying and spray chilling methods to obtain microparticles containing phytosterol

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INTRODUCTION AND OBJECTIVE

Food industry has been searching for technologies to develop innovative products with special properties that meet a new consumers profile more concerned about the impact of food on their health (BFT, 2010). The properties of microencapsulation as protection, controlled release and delivery of various substances make this technology a good alternative for the development of products with special properties (Alvim, 2013; Ré, 2006). The different food matrices require different microparticles and consequently different methods and wall materials. Among the methods for microencapsulation, spray drying and spray chilling are widely used because their advantages such as good retention of volatile components, suitable process conditions and possibility of large scale production (Gharsallaouil, 2007; Alvim, 2013). The aim of this study was to compare the techniques of spray chilling (hydrophobic particles) and spray drying (hydrophilic particles) in the production of microencapsulated phytosterol.

MATERIALS AND METHODS

Phytosterol (PHY) (Cognis, Brazil), Arabic gum (AG) (CNI Colloids, Brazil), Stearic acid (SA) (Synth, Brazil), Low trans hydrogenated vegetable fat (HVF) (Bunge, Brazil), other reagents in analytical grade.

Samples preparation

Spray Chilling: A mixture of HVF and SA (wall material, 1:1 w/w, Melting Point: 50 °C) was melted to 70 °C. The PHY (core) was added to the lipid mixture and homogenized by mechanical stirring and under heating plate. The mixture of wall material and the core, kept at 70 °C, was processed by spray chilling. *Spray drying:* The PHY was dispersed in an AG solution using an Ultra-Turrax homogenizer (11000 rpm / 3 min) and was spray dried. Process conditions are described in Table 1.

Table 1. Process conditions

Process conditions	Drying	Chilling
Sample Temperature(°C)	40 ± 2	70 ± 2
Inlet temperature (°C)	150 ± 2	5 ± 3
Outlet temperature(°C)	75 ± 3	15 ± 2
Sample feed (ml/min)*	8	12

*Spray Drier: peristaltic pump; Spray Chiller: gravity. Common conditions: nozzle air pressure: 50 mbar, air flow 8.3 L/min, aspirator: 100%. Equipment: Mini Spray Dryer B290 + spray chilling module (nozzle 0.7 mm, Büchi, Uster, Switzerland).

Microparticles Characterization

Mean diameter: Samples were observed by optical microscopy (BX4, Olympus, Tokyo, Japan) using the objective of 100x, and the images were acquired using a digital camera (Q-Color3, Olympus, Tokyo, Japan). The mean diameters (500 particles) were determined by the freeware software Scion Image (www.scioncorp.com) (Alvim, 2010).

Encapsulation efficiency (%EE): The phytosterol fractions (campesterol, brassicasterol, stigmasterol and b-sitosterol) on free substance and after microencapsulation were carried by gas chromatography (Almeida, 2009).

Morphology: Appearance of samples was evaluated by optical microscopy (microscope BX4, Olympus) and scanning electron microscopy (DSM 940A FOCUS, Zeiss) (Alvim, 2010).

Statistical analyzes: Different mean values were statistically evaluated by analysis of variance (anova), using the software Statistica® 5.5 (StatSoft, Inc., Tulsa, OK, USA). Mean separation was determined using the Tukey test at $P \leq 0.05$.

RESULTS AND DISCUSSION

The average particles diameters for samples were different (Table 2, $p \leq 0.05$), but were within the expected range for the process conditions and type of atomizer used.

Table 2. Microparticles mean size.

Process	Mean size (μm)
Spray Drying	11.9 ± 0.4 b*
Spray Chilling	19.9 ± 0.3 a

*Different letters: statistical differences ($p<0.05$).

These diameters differences can be explained by the principle of microparticles formation in each process. In spray drying, microdroplets formed in the atomization can shrinkage during evaporation of the solvent, generating smaller particles than the droplets formed initially (Mezhericher, 2010). In the case of spray chilling, the formation of particles is based on hardening of the lipid melted matrix submitted to low temperature, preserving the original diameter of the atomized microdroplets.

The encapsulation efficiencies (Figure 1) were very different depending on the process employed. For spray chilling, the fractions of PHY were almost completely encapsulated and preserved in lipid microparticles (99.5% on average), with values very similar, with no statistical differences ($p < 0.05$). High encapsulation efficiencies (78.3 to 97.8 %) were observed by Leonel (2010) for glucose encapsulated in lipid microparticles. On the other hand, for microparticles containing PHY obtained by spray drying, it was observed that only 20% on average of the fractions were preserved. Effects of temperature in the spray drying process and particularly the emulsion stability of the PHY on solution of AG during processing may be responsible for the low encapsulation efficiencies observed.

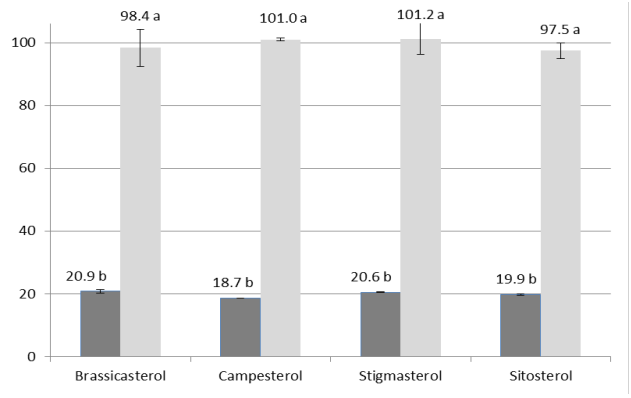

Figure 1. EE (%) of the PHY microparticles. Spray drying (■) or spray chilling (□).*Different letters: statistically significant differences ($p<0.05$).

The morphologies of the microparticles (Figure 2) showed spherical shapes and high polydispersity. Tonon (2011) and Leonel (2010) observed similar average diameters and the same morphologies for microparticles, obtained by spray chilling or spray drying encapsulating various core materials.

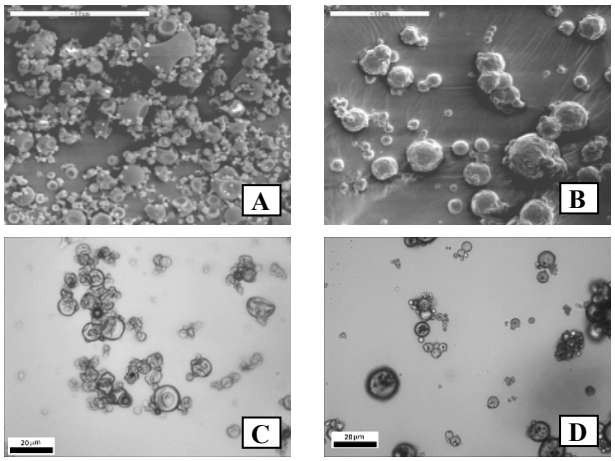

Figure 2: Morphology of microparticles. Spray Drying: A – SEM, (700X, bar = 50 µm); C – MO (200X, bar = 20 µm). Spray Chilling: B – SEM, (500X, bar = 50 µm); D – MO (200X, bar = 20 µm).

CONCLUSIONS

Production of phytosterol microparticles by spray drying or spray chilling, has been successfully tested. The EE% of the FHY (core material) was close to 100 % when using the spray chilling process, while for the spray drying the EE% was around 20 % indicating that the retention and preservation of core are more sensitive to this process and aspects like emulsion stability and process conditions should be carefully dimensioned to ensure that microencapsulation can be successful. The appearance of the microparticles for both samples was similar, due to the same method of formatting the microdroplet (atomization). In microencapsulation, the choice of the method of particle formation is essential to the success of the production of microparticles with desired characteristics and functionalities.

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