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Wet agglomeration by high shear of binary mixtures of curcumin-loaded lyophilized liposomes and cornstarch: Powder characterization and incorporation in cakes

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ABSTRACT

Wet agglomeration was used to enrich cornstarch with curcumin-loaded lyophilized liposomes, and the enriched cornstarch was incorporated in cake batters. Lyophilized liposomes were obtained using proliposome hydration and encapsulated curcumin remained stable after 70 days of storage. The enriched cornstarch was produced using a high shear method using maltodextrin as a binder, and the powders were characterized in terms of water activity, moisture content, water sorption, bulk properties, flowability, wetting time, instrumental colorimetry, scanning electron microscopy, X-ray diffraction, and infrared spectroscopy. The data obtained showed that the presence of liposomes and maltodextrin did not significantly change the cornstarch bulk structure. The enriched powders were homogeneous in both color and size distribution. Better bulk properties were observed in agglomerated powders than in non-agglomerated cornstarch, as higher flowability and lower cohesiveness. With respect to the application of the agglomerated starches in cakes, they were produced and analyzed in terms of instrumental color, texture and proximate composition. The results showed a decrease in both hardness and chewiness as well as an intense and homogeneous yellow color in cakes produced with the agglomerated cornstarch. This study indicated that it is feasible to produce a cake batter with curcumin encapsulated in liposomes as a functional ingredient.

1. Introduction

The development of techniques to incorporate bioactives in food can help to produce healthier food products. Liposomes are interesting structures for the microencapsulation of hydrophobic bioactives, overcoming drawbacks such as the difficulty of dispersion in water-based formulations and low bioaccessibility and bioavailability in the gastrointestinal tract (Parada & Aguilera, 2007). Liposomes are phospholipid-based vesicles formed when phospholipid bilayers close upon themselves due to unfavorable interactions of their edges with water (Lasic, 1993). Several studies have looked at the encapsulation of bioactives in liposomes, but their application in food matrices is still limited (Bochicchio, Barba, Grassi, & Lamberti, 2016; Marsanasco, Piotrkowski, Calabró, Alonso, & Chiaramoni, 2015; Sahari, Moghimi, Hadian, Barzegar, & Mohammadi, 2017).

Curcumin is the main pigment present in the rhizome of *Curcuma longa* and it is used as a food colorant, flavoring and preservative (Arrue, Zarate, Schott-Verdugo, & Schott, 2015). Its antioxidant activity has been studied as it can play a role in controlling or avoiding diseases induced by oxidative processes (Giri, Selvaraj, & Kalra, 2003; Xie et al., 2009). However, curcumin is poorly soluble in water and quite sensitive to light, so liposomes may be useful to increase its stability, as well as to enable its dispersion in aqueous media (Ghalandarlaki, Alizadeh, & Ashkani-Esfahani, 2014).

Wet agglomeration is a process used to increase the average particle size of a powder, using, for example, a binder solution and high shear, to promote collisions and adhesion among the particles (Iveson, Litster, Hapgood, & Ennis, 2001). This process is extensively used in industrial processes, including applications in pharmaceutics and food, as it is simple, low-cost and easily scalable (Mandato, Taliani, Aït-Kaddour, Ruiz, & Cuq, 2013). The main advantages of the wet agglomeration process include the elimination of unfavorable characteristics of fine powders, increasing flowability, compaction and homogeneity in the composition of granulated products (Cheng, Hsiau, & Liao, 2012). The process is generally done in four steps: (1) homogenization of dry food powders; (2) addition of binder solution; (3) constant stirring of

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resulting wet mass and (4) drying of the granules. High shear contributes to the homogeneous breaking of particles into small and cohesive aggregates (Harnby, 1997), which contributes to an effective dispersion of the liquid around the entire surface of the particles, leading to a better consolidation of the product (Cavinato et al., 2010).

The main aim of this study was to enrich cornstarch with curcumin-loaded lyophilized liposomes using wet agglomeration with high shear. The powders were stored for 60 days and their physicochemical stability was evaluated. The agglomerated cornstarch was then incorporated in cake batters to evaluate the possibility of using it as ingredient in a bakery product. The results of this study can be useful to improve the nutritional content of a very common ingredient in the food industry, i.e., cornstarch, and to evaluate the possibility of developing functional products with the enriched cornstarch.

2. Material and methods

2.1. Chemicals and reagents

Curcumin (Sigma-Aldrich, St. Louis, MO, USA), Phospholipon 90H (Lipoid GmbH., Ludwigshafen, Germany), and xanthan gum (Grindsted Xanthan 80, Du Pont, Cotia, SP, Brazil) and guar gum (Êxodo Científica, Hortolândia, SP, Brazil) were used. Cornstarch (Argo CS3400, Mogi Guaçu, SP, Brazil) and maltodextrin (MOR REX 1910, Mogi Guaçu, SP, Brazil) were donated by Ingredion (Mogi Guaçu, SP, Brazil). The ingredients used to produce the cake batters were: white wheat flour, unsalted margarine, sugar, whole milk, eggs (medium size), cornstarch and baking powder (Royal®, Mondeléz International, Curitiba, PR, Brazil). Ultra-pure water (Direct Q3, Millipore, Billerica, MA, USA) was used throughout the experiments.

2.2. Production and lyophilization of curcumin-loaded liposomes

Liposomes were produced according to Silva, Jange, Rocha, Chaves, and Pinho (2017). Anhydrous ethanol 99.5% (100 ml) containing 3.2 g of phospholipid and 25 mg of curcumin were dropped on 2 g of micronized sucrose previously obtained using a ball mill (CE500, Cienlab, Campinas, SP, Brazil), after 10 h of constant stirring at 100 rpm. The injection process of the ethanol solution occurred in a rotary evaporator using a peristaltic pump (Masterflex 7528-30, Cole-Parmer, Vernon Hills, IL, USA) at a flow rate of 4 ml/min. The rotating flask was maintained at 45 ± 2 °C to evaporate the ethanol. The powder (proliposomes) were stored in a vacuum desiccator in the dark and kept at room temperature (25 °C) for a maximum of 1 wk prior to their hydration.

For the liposome production, 2 g of proliposomes were hydrated with 100 ml of deionized water using ultra-agitation (IKA T25, Staufen, Germany) at 13,000 rpm for 5 min at 65 °C. The dispersion was cooled to 25 °C and a mixture of xanthan and guar gums in a 10:90 ratio was added using magnetic stirring (3600 rpm). The liposomes were frozen in liquid nitrogen (-210 °C, 2 min) and then lyophilized (L202-Liotop, São Carlos, SP, Brazil). The drying process occurred for 48 h, under a vacuum of 444 μHg and at −53 °C. The lyophilized liposomes were stored in a desiccator at 25 °C for a maximum of 4 wk.

2.3. Quantification of curcumin content in lyophilized liposomes

The curcumin was extracted from lyophilized liposomes using anhydrous ethanol. The lyophilized liposomes (20 mg) were diluted in 20 ml of ethanol and shaken. The bioactive concentration was calculated using a standard curve of pure curcumin (≥ 94%) in ethanol (R^2 = 0.9984) from absorbances at 425 nm using a spectrophotometer (Libra S22, Biochrom, Cambridge, UK) (Silva et al., 2017).

Table 1
Composition of the agglomerated cornstarch produced with different formulations.

Formulation	Cornstarch % w/w	Maltodextrin solution % w/w	Lyophilized liposomes % w/w
F_4	89	7	4
F_7	86	7	7
F_{10}	83	7	10

2.4. Production of the cornstarch enriched with curcumin-loaded lyophilized liposomes using wet agglomeration with high shear

Wet agglomeration was carried out with high shear using a multiprocessor (Walita R17625, Philips, São Paulo, SP, Brazil) with an impeller rotation frequency of 1300 rpm (21.7 Hz), using the method of Toniazzo, Galeskas, Dacanal, and Pinho (2017). Cornstarch (100 g) and curcumin-loaded lyophilized liposomes were mixed with agitation, whereas the maltodextrin solution (30% w/v) was dripped over the mixture for 10 min with the aid of a peristaltic pump (Cole Parmer 7528-30, Vernon Hills, IL, USA). The wetted agglomerated cornstarch was then dried for 24 h at 60 °C in a convection oven. Finally, all the samples were stored in glass desiccators containing silica gel (25 °C, < 10% relative humidity) for a maximum of 70 d. The enriched cornstarches were produced using three different mass percentages of curcumin-loaded lyophilized liposomes, as can be seen in Table 1.

2.5. Characterization of the cornstarch enriched with curcumin-loaded lyophilized liposomes

2.5.1. Morphology

The morphology of the curcumin-loaded lyophilized liposomes, maltodextrin and enriched cornstarches was analyzed using scanning electron microscopy (SEM) using a TM-300 (Hitachi, Tokyo, Japan). The powders were placed on double-faced carbon tape and fixed to aluminum stubs. Images were captured using a voltage of 15 kV and a 500x magnification (for the pure ingredients) or a 1000x magnification (for the enriched cornstarches).

2.5.2. X-ray powder diffraction

X-ray diffraction analyses were done using a MiniFlex600 (Rigaku, Tokyo, Japan) with a copper anode tube with λ = 1.5418 Å and a graphite monochromator in the diffracted beam. The scanning angle ranged from 5° to 40° for 2θ, in steps of 0.2° and a 2°/min rate. Crystallinity percentages were determined using a deconvolution method based on Gaussian profile functions using Origin® 2017 software (OriginLab, Northampton, MA, USA).

2.5.3. Fourier transform infrared (FTIR) spectroscopy

FTIR spectroscopy analyses were done using a PerkinElmer FT-IR Spectrometer (Waltham, MA, USA). Briefly, the powders (10 mg) were added to KBr pellets and spectra were obtained in the wavenumber region between 4000 and 400 cm^{-1} with a resolution of 4 cm^{-1} with 20 scans. Data were analyzed using Spectrum One software version 5.3.1. software (Perkin-Elmer).

2.5.4. Moisture content and water activity

Moisture content analysis of cornstarches (500 mg) were done to constant weight using a moisture analyzer (MB35, Ohaus, Greifensee, Switzerland) using infrared radiation from a halogen source. Water activity was determined using an Aqualab analyzer (3TE, Decagon Devices, Pullman, WA, USA).

2.5.5. Moisture adsorption isotherms

The equilibrium moisture content of the powders was determined using a gravimetric static method (Labuza, 1985). Samples with known