Food Research International 47 (2012) 38–44

Contents lists available at SciVerse ScienceDirect

Food Research International

journal homepage: www.elsevier.com/locate/foodres

The effect of adding oleic acid in the production of stearic acid lipid microparticles with a hydrophilic core by a spray-cooling process

Marilene D.M. Morselli Ribeiro [a,*], Daniel Barrera Arellano [a], Carlos R. Ferreira Grosso [b]

[a] Food Technology Department, Faculty of Food Engineering, State University of Campinas-UNICAMP, C.P. 6091, 13083-970, Campinas, SP, Brazil
[b] Food and Nutrition Department, Faculty of Food Engineering, State University of Campinas-UNICAMP, C.P. 6121, 13083-970, Campinas, SP, Brazil

ARTICLE INFO

Article history:
Received 17 June 2011
Accepted 24 January 2012

Keywords:
Lipid microparticles
Spray-cooling
Binary lipid mixtures
Release of hydrophilic core

ABSTRACT

The objective of the present work was to study the effect of adding oleic acid (OA) to the stearic acid (SA) used in the production of lipid microparticles (LP) by the spray-cooling process, the core material being a glucose solution, using lecithin as the surfactant. The fatty acid composition of the lipid raw materials was characterized. For the lipid mixtures the thermal behavior was determined by differential scanning calorimetry (DSC), and the solid fat content (SFC) and iso-solids diagrams also determined. The efficiency of encapsulation was determined in the LP produced, and also the particle size distribution, morphology and the release of the core material into the aqueous medium. The LP showed spherical, wrinkled forms. The addition of OA modified their crystallization pattern and aided in incorporation of the core material, resulting in a lower surface glucose amount of 2 to 7% for the mixtures as compared to 22% for pure SA. The encapsulation efficiency was high for the OA/SA mixtures (92%–96%) but only reached 75% when SA was used alone. The release profile was also modified, being greater for SA as compared to the mixtures containing OA.

© 2012 Elsevier Ltd. All rights reserved.

1. Introduction

Microencapsulation is used to coat substances in the liquid, solid or gaseous state with wall materials that protect them against adverse effects from the environment and to the environment in the case of dangerous or toxic core materials (Ré, 2000). Microcapsules are small particles containing active agents and coating materials including a variety of polymers, carbohydrates, fats and waxes. The applications of this technique have increased in the food industry, where the encapsulated materials (vitamins, oils, essences and others) are protected against moisture, oxidation, heating and other extreme conditions, avoiding chemical reactions with other components of the food formulations and also controlling release of the active principle of the microcapsule (Gibbs et al., 1999).

The control of the release can be used to increase the effectiveness of many encapsulated ingredients. This technique was first applied in the pharmaceutical industry (Savolainen et al., 2002) and then expanded to the food, agrochemical and veterinary medicine industries, others, with the advantage that the active ingredients can be released at controlled rates during prolonged periods of time. For example, aroma compounds and nutrients can be released during consumption, carbon dioxide released when an acid reacts with sodium carbonate during cooking, etc. (Pothakamury & Barbosa-Cánovas, 1995).

The use of lipids in the production of microparticles has been known for many years in the pharmaceutical industry, as well as the "spray-chilling" or "congealing" technique (Eldem et al., 1991). Currently the spray-cooling technique is receiving considerable attention, since it is a rapid, safe and reproducible physical process, allowing the easy adjustment of the particle size, where a solution, suspension or emulsion containing a core material in a molten lipid matrix is atomized into an environment at a temperature below the melting point of the mixture in use (Albertini et al., 2008), generally a cold chamber (cold air or liquid nitrogen).

During the solidification and crystallization process, lipids present an important phenomenon known as polymorphism, which is the capacity of a molecule to have more than one crystalline form depending on the arrangement of the crystal's nucleus (Sato & Ueno, 2005). Due specifically to this property, the solid lipid microcapsules, as well as the solid lipid nanoparticles (SLN) developed by high pressure homogenization (Liedtke et al., 2000) can show low encapsulation efficiency and expel the core during storage. Studies are underway to minimize this effect using nanostructured lipid carriers (NLC), which are mixtures of solid and liquid lipids. The liquid lipid modifies the crystallization kinetics, modifying the organization of the crystalline network, increasing the encapsulation capacity and decreasing expulsion of the core (Müller et al., 2002).

To aid incorporation of the core material in the lipid matrix, lecithin can be added as an emulsifier, this tensoactive compound has been widely used in foods. The acyl chains of the phospholipids show different sizes and degrees of unsaturation, influencing the

* Corresponding author. Tel.: +55 11 40725263; fax: +55 11 40725000.
E-mail address: morselliribeiro@uol.com.br (M.D.M. Morselli Ribeiro).

0963-9969/$ – see front matter © 2012 Elsevier Ltd. All rights reserved.
doi:10.1016/j.foodres.2012.01.007

lipid part and, consequently, the emulsion and dispersion systems (Nii & Ishii, 2004).

The best parameters of emulsion process were obtained from studies realized by Leonel, Chambi, Barrera-Arellano, Pastore, and Grosso (2010), as the amount of lecithin and velocity dispersion.

The aim of the present study was to produce solid lipid microparticles by the spray-cooling process, using mixtures of stearic acid (SA) and oleic acid (OA) in different proportions, containing lecithin as the emulsifier and a glucose solution as the core material and to evaluate the release behavior.

2. Material and methods

2.1. Raw materials

Commercial stearic acid (Braido, São Paulo, Brazil); commercial oleic acid (Braido, São Paulo, Brazil); 68% lecithin (Solae, Rio Grande do Sul, Brazil); anhydrous D-glucose (Synth, São Paulo, Brazil); enzymatic kit for the determination of glucose (Laborlab, SP); Tween 80 — polyoxyethylene sorbitan monooleate (Synth, São Paulo, Brazil); chloroform (F. Maia, São Paulo, Brazil).

2.2. Lipid mixtures

Binary mixtures were developed combining a saturated lipid (stearic acid — SA) with an unsaturated lipid (oleic acid — OA). The mixtures studied were (SA/OA): (1) 50/50, (2) 60/40, (3) 70/30, (4) 80/20, (5) 90/10 and (6) 100/0. The lipid mixtures were characterized by their fatty acid composition, thermal analysis and solids profile, and selected according to the melting point range, aspect and consistency adequate for the production of LP.

2.3. Emulsion preparation

Water in oil emulsions were prepared from the lipid mixtures with a 40% glucose solution in the proportion of 75/25 lipid/glucose solution, plus 5% soy lecithin (HLB = 4.0) as the emulsifier (Klein, 2008) w/w in relation to the total amount of lipid used.

The lipids and soy lecithin were heated to 70 °C, the glucose solution added, and the mixture homogenized for 5 min at 10,000 rpm in an Ultraturrax IKA T 18 Basic (Germany), maintaining the system in a water bath at the same temperature.

The stability of the emulsions was observed visually for 30 min in a 100 mL graduated cylinder in a water bath at the same temperature as the system, this being time enough to carry out the spray-cooling process. The stable emulsions were considered without separation and those that showed separation were disqualified in the time observed. The scale of the graduated cylinder was used to convert into percent stability.

2.4. Production of lipid microparticles by spray cooling

The lipid mixtures with (SA/OA) proportions of 70/30, 80/20, 90/10 and 100/0 were selected according to their melting points (Section 2.2), and the emulsion stability observed with respect to the aspect and viscosity for processing, with the objective of obtaining stable LP after spray cooling drying.

The emulsions were added by gravity to a tank connected directly to a double fluid atomiser with a nozzle diameter of 0.7 mm (Labmaq — SP, Brazil) and an air pressure of 1.25 kgf/cm^2 (air at room temperature), with the atomiser being carried out on the inside of a chamber cooled to 0 °C (Ultratorac, LKB — Bromma, Germany). For each lipid mixture (SA/OA) proportions of 70/30, 80/20, 90/10 and 100/0, the ideal temperature for adding it to the tank and the atomiser temperature were determined in independent processes, respectively as follow: 60 °C, 61 °C, 63 °C and 65 °C.

The LP produced were stored in closed containers at 4 °C, and analyzed with respect to their morphology, particle size, encapsulation efficiency, surface glucose and core release profile.

2.5. Fatty acid composition

The fatty acid composition of the lipid mixtures were determined by gas chromatography of the fatty acid methyl esters using the method Ce 1–62 of the American Oil Chemists' Society (1997). The fatty acid methyl esters were prepared using the methodology of Hartmann & Lago (1973).

An Agilent series 6850 CG System gas chromatograph was used, equipped with a flame ionization (FID) detector and a split injector. The components were separated in a LM-5 fused silica capillary column (L & M, polydiphenyldimethylsiloxane; 30 m in length; internal diameter of 0.25 mm; film thickness of 30 μm). Samples of 1.0 μL were injected with a split of 1:50, and the injector and detector temperatures were both 320 °C. The stripping gas was helium with a flow rate of 1.1 mL min^{-1}. The oven temperature was programmed to start at 180 °C for 5 min, followed by a gradient of 4 °C min^{-1} up to 300 °C, remaining at 300 °C for a further 35 min, giving a total run time of 70 min.

The qualitative composition was determined by a comparison of the retention times of the peaks with those of the respective fatty acid standards. The quantitative composition was determined by normalization of the area using the software Agilent GC Chemstation Plus, and expressed as a percentage of the mass.

2.6. Thermal behavior melting and crystallization curves obtained by differential scanning calorimetry

The melting and crystallization curves of the lipid mixtures were determined by differential scanning calorimetry using a Perkin Elmer model Diamond calorimeter (USA). The analyses were carried out with approximately 10 mg of sample contained in hermetic aluminum capsules in an inert atmosphere (N_2), using the method Cj 1–94 of the American Oil Chemists' Society (1998).

2.7. Solids profile

The solid profiles of the lipid mixtures were determined using the method Cd 16b-93 of the American Oil Chemists' Society (1999). Readings were taken in triplicate using a 20 MHz Maran Ultra Benchtop nuclear magnetic resonance spectrophotometer (USA) for the following temperatures: 10, 20, 25, 30, 35, 40, 45, 50, 55 and 60 °C.

2.8. Particle size distribution

The size of the LP was determined in a Jenaval-Zeiss optical microscope (East Germany), the images being registered by a digital camera, using an optovar of ×1.25 and objective of ×12.5. The software used to acquire the images was a Global LAB Image 2 and Scion Image (www.sciocorp.com). The samples were suspended in glycerol and the manually measured the diameters of 500 particles were analyzed for each sample. Two measures (horizontal and vertical) were performed and the average obtained in each particle, using transmitted light.

2.9. Particle morphology

The microstructure of the LP was observed using a Jeol JMS-T300 scanning electronic microscope (Tokyo, Japan), obtaining the images with a voltage acceleration of 10 kV and magnification of ×100 and ×350. The samples were fixed in aluminum stubs with double-faced copper tape, and covered with a fine layer of gold using a current of 40 mA for 180 s in a Baltzer evaporator (Baltec SCD50, Austria).

Download English Version:

https://daneshyari.com/en/article/4561939

Download Persian Version:

https://daneshyari.com/article/4561939

Daneshyari.com