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Solid lipid microparticles produced by spray chilling technique to deliver ginger oleoresin: Structure and compound retention

Vivian Boesso Oriani [a,*], Izabela Dutra Alvim [b], Larissa Consoli [a], Gustavo Molina [c,d], Glaucia Maria Pastore [d], Míriam Dupas Hubinger [a]

[a] Laboratory of Process Engineering, Department of Food Engineering, School of Food Engineering, University of Campinas, CEP 13083-862 Campinas, SP, Brazil
[b] Cereal and Chocolate Technology Center — CEREAL CHOCOTEC, Food Technology Institute — ITAL, CEP 13070-178 Campinas, SP, Brazil
[c] Institute of Science and Technology, Food Engineering, UFVJM, Diamantina, Minas Gerais, Brazil
[d] Laboratory of Bioflavors, Department of Food Science, School of Food Engineering, University of Campinas, CEP 13083-862 Campinas, São Paulo, Brazil

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ABSTRACT

Ginger oleoresin (GO) is frequently used as a flavor ingredient in various foods. Besides flavoring, its bioactive compounds are known for antimicrobial and antioxidant properties. In this study, we investigated the formation and characterization of solid lipid microparticles (SLMs) loaded with GO by the spray chilling technique. Mixtures composed of palmitic acid with oleic acid or palm fat were used as carriers. DSC and X-ray diffraction showed that the particle crystalline structure was decreased by oleic acid. Retention of pungent and volatile compounds was higher than 96% and 75%, respectively, in the best formulations. FTIR spectroscopy revealed no chemical interaction between GO and carriers. The particles presented a spherical shape and rough surface, and GO was dispersed over the entire particles, as shown by confocal microscopy. High volatile and pungent compound retention have shown that spray chilling can be a very promising technique for the production of SLM loaded with GO.

1. Introduction

Ginger oleoresin (GO) is obtained from dried ginger rhizome by solvent extraction. The final product is a dark golden brown viscous oil that contains gingerols and shogaols, which are the compounds responsible for the typical ginger pungency, and have significant contribution in pharmacological products. Drying and heating convert gingerol molecules into shogaols, which are more pungent than gingerols and, because of that, shogaols at concentrations higher than gingerols indicate low quality for ginger products. Gingerols and shogaols consist of a series of similar molecules, but the most abundant are (6)-gingerol and (6)-shogaol. The native ginger oleoresin does not contain volatile compounds. Thus, ginger essential oil is usually added to the non-volatile resinous fraction to make commercial ginger oleoresin have a flavor profile close to the respective fresh spice (Balakrishnan, 2004). The predominant volatile compounds added in GO are classified as sesquiterpene hydrocarbons. Commercial GO is more attractive to the industry than fresh ginger because it is microbiologically safe and can be standardized for acceptable flavor levels (Balakrishnan, 2004; Singh et al., 2008; Huang, Chung, Wang, Law, & Chen, 2011; Kubra & Rao, 2012; Murthy, Gautam, & Naik, 2015).

In the liquid form, commercial GO may present difficulties in handling during product formulation, due to its high viscosity and volatility. Also, some of its compounds may be degraded or volatilized if exposed to heat and light during processing or storage. Encapsulation techniques have been used to overcome these problems, employing food grade materials to coat and carry oleoresin (Reineccius, 1989; Vaidya, Bhosale, & Singhal, 2006; Wang, Yuan, & Yue, 2015). The use of powdered GO is convenient for the food industry for many reasons, such as maintaining stability and protecting the compounds against the external environment such as light, heat and humidity. Furthermore, powders can have improved handling and flow properties in relation to the liquid form (Ré, 1998; Jafari, Assadpoor, He, & Bhandari, 2008).

There are several ways to convert oleoresins into powder. One of them is the spray chilling technique, that employs fatty acids, triacylglycerols, waxes or blends among these materials as carriers. The active material is first dispersed, emulsified or solubilized in the molten lipid matrix which is then fed into a heated nozzle and atomized into a chamber with temperature kept below the lipid melting point, where droplets solidify in contact with cooled air to form SLM. Spray chilling has many advantages when compared to spray drying or

* Corresponding author at: Laboratory of Process Engineering, Department of Food Engineering, School of Food Engineering, University of Campinas, P.O. Box 6121, CEP 13083-862, Campinas, SP, Brazil.
E-mail address: vivianboriani@yahoo.com.br (V.B. Oriani).

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other encapsulation techniques, such as not needing solvent evaporation, increased productivity, reducing cost and facilitating scale-up (Desai & Park, 2005; Okuro, Eustáquio De Matos, & Favaro-Trindade, 2013).

The use of lipids as encapsulating materials extends particle application possibilities, by exploring temperature or mechanical-triggered release (Madene, Jacquot, Scher, & Desobry, 2006).

Palm oil and its by-products are lipids of interest in the study of the formation of lipid particles. The palm fat is considered one of the cheapest vegetable oils to produce and refine (Gunstone, 2013). It is the only vegetable oil with almost 50–50 composition of saturated and unsaturated fatty acids, that present a semi-solid physical state (Mba, Dumont, & Ngadi, 2015). The refining of crude palm fat results in by-products such as palm fatty acid, comprising mainly free fatty acid with palmitic acid and oleic acid as the major components (Ping & Yousof, 2009). For production of solid lipid microparticles, blending high and low melting point lipids to act as carriers can result in a disorganized crystal structure that improves the active retention due to a modulation of lipid crystalline lattice (Jenning & Gohla, 2000; Müller, Radtke, & Wissing, 2002; Hu et al., 2005; Ribeiro, Arellano, & Grosso, 2012; Sartori, Consoli, Hubinger, & Menegalli, 2015).

In this context, our aim in this work was to enable the production and characterization of SLM loaded with a commercial GO by the spray chilling technique. A solid lipid (palmitic acid), responsible for ensuring the formation of solid particles at room temperature, and lipid modulators, with the function of modifying the crystal structure of the solid lipid matrix (liquid lipid (oleic acid) and semi-solid fat (palm fat)), were used as carrier agents. The microparticles were characterized in relation to volatile and pungent compound retention, crystalline structure, surface morphology and distribution of GO over SLM by the confocal technique. The behavior of lipid microparticles loaded with GO and the effect of the addition of lipid modulator in a solid lipid have not yet been studied.

2. Materials and methods

2.1. Materials

A commercial ginger oleoresin with 30% ginger essential oil addition was kindly donated by NATUREX (São Paulo, Brazil), and will be further referred as GO for simplification. As carrier agents, we used: palmitic acid (PA; VETEC, Rio de Janeiro, Brazil; melting point 63 °C; 98.8% palmitic acid determined by AOCS method; Ce 2-66, 2004), oleic acid (OA; VETEC, Rio de Janeiro, Brazil; melting point 8.2 ± 0.1 °C; 77.8% oleic acid and 11.6% linoleic acid determined by AOCS method; Ce 2-66, 2004) and palm fat (PF; Triângulo Alimentos, Itápolis, Brazil; melting point 44.9 ± 0.2 °C; 39.9% palmitic acid, 37.6% oleic acid, 10.2% linoleic acid and 8.2% stearic acid determined by AOCS method; Ce 2-66, 2004). All other reagents used were of analytical grade.

2.2. Methods

2.2.1. Selection of lipid carriers

Preliminary tests were conducted to define the lipid carriers. The miscibility of lipid mixtures in GO was used as criteria for selection. The GO concentration was kept at 10% (w/w) in all mixtures analyzed.

2.2.2. Production of SLM by spray chilling

Each lipid component and GO were weighed according to their respective proportion, as shown in Table 1. First, the lipids were heated to 85 °C by a temperature controlled water bath (Tecnal, TE-184, Piracicaba, Brazil) to assure complete melting. They were then added to GO and the mixture (0.050 kg) maintained under magnetic stirring. SLMs were obtained using a Büchi-B290 spray dryer set to the spray chiller mode (Büchi, Flawil, Switzerland). The mixture was fed into a heated double fluid atomizer with a nozzle diameter of 2.0 mm using a peristaltic pump, at a mass flow rate of 0.7 kg/h. SLMs were formed within a cooled chamber where the inlet air temperature was 7 °C. Atomizing air and cooling air flow rates were 1052 L/h and 35,000 L/h, respectively. At the end of the process, samples were collected and stored in closed containers and kept at 5 °C. Each experiment was performed in duplicate.

Table 1
Formulations and compositions of SLM loaded with ginger oleoresin.

Components	Role	Formulations				
		P1	P2	P3	P4	P5
		Composition, w/w				
Ginger oleoresin (GO)	Flavor	10	10	10	10	10
Palmitic acid (PA)	Carrier	90	85	75	85	75
Oleic acid (OA)	Carrier	–	5	15	–	–
Palm fat (PF)	Carrier	–	–	–	5	15

2.2.3. Characterization of SLM

2.2.3.1. Thermal analysis. Thermal analysis was performed by Differential Scanning Calorimetry, using a DSC 2920 Modulated thermal analyzer (TA Instruments, New Castle, Delaware, USA.). Samples were weighed (~5 mg) in hermetically sealed aluminum pans. Operation conditions were: heating to 80 °C and maintained for 5 min, cooled to −40 °C (10 °C/min) for 30 min, and then heated to 80 °C at 5 °C/min.

The crystallinity index (CI) was calculated using Eq. (1) by Freitas and Müller (1999), with some modifications.

$$CI(\%) = \left(\frac{\Delta H_{M,part}}{\Delta H_{M,PA} \times [PA]}\right) \times 100 \quad (1)$$

where $\Delta H_{M,part}$ and $\Delta H_{M,PA}$ are the melting enthalpy (J/g) of SLM with GO and with bulk PA, respectively; [PA] is the concentration of PA (g/100 g) in the matrix.

2.2.3.2. Crystalline structure. Samples were analyzed using the X-ray powder diffraction technique. A Philips X-ray diffractometer (Analytical, X Ray X'Pert-MPD, Almelo, Netherlands) was used to study the crystal structure. X-rays of $\lambda = 1.54056$ Å were generated by a Cu Kα source. The diffraction was measured in the 2θ range from 5 to 30° of 0.02°/s.

2.2.3.3. Particle size. The size distribution and mean volumetric diameter of the SLM were determined by the light scattering technique using laser diffraction in a Mastersizer 2000 (Malvern Instruments Ltd., Malvern, UK). Samples were dispersed in an aqueous solution containing 1.5% (w/w) Tween® 80, and then added into the dispersion unit of the equipment, which was filled with distilled water. The analysis was conducted at room temperature. The mean diameter was determined based on the average diameter of a sphere of the same volume (De Brouckere diameter — $D_{[4,3]}$). Size distribution was characterized by $D_{0.1}$, $D_{0.5}$ and $D_{0.9}$, which represent the diameter of accumulated distribution of 10%, 50% and 90% of total particles.

2.2.3.4. Fourier transform infrared spectroscopy-FTIR. Samples were ground with KBr and compressed by a hydraulic press, forming pellets that were used in a FTIR spectrometer (JASCO, 6100, Tokyo, Japan). Samples were analyzed in the 4000 to 400 cm^{-1} region, with a resolution of 4 cm^{-1} for 100 scans.

2.2.3.5. Morphology and GO distribution over SLM. The SLM morphology was investigated from images obtained in a scanning electron microscope (SEM) with Energy Dispersive X-ray Detection (LEO Electron Microscopy Leo 440i, Oxford — Cambridge, England). Microparticles were covered with a gold layer by a Polaron Sputter Coater (VG