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Formation of solid lipid microparticles from fully hydrogenated canola oil using supercritical carbon dioxide

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ABSTRACT

A simple and green process based on supercritical carbon dioxide (SC–CO_2) technology was used to produce solid lipid microparticles from fully hydrogenated canola oil (FHCO). The effects of pressure (122, 211 and 300 bar) and nozzle diameter (0.1, 0.3 and 14.3 mm) on the particle size, morphology, thermal properties and polymorphism were investigated. Then, the essential oil loading capacity of the particles obtained under optimum conditions was evaluated. Smaller nozzle diameter (0.1 mm) and lower pressure (122 bar) produced spherical particles with narrower particle size distribution and a mean diameter of 1.27 μm. Melting point of all particles decreased to 65 °C from 69 °C for the FHCO. The main polymorphic form of the particles obtained at lower pressures and larger nozzle diameters was α, whereas it was β at higher pressures and smaller nozzle diameters. Essential oil loading efficiency of 96% was achieved when FHCO blended with 40% essential oil was used as the feed material. SC-CO_2 process is a promising technology for solid lipid particle formation as delivery vehicles for bioactives.

1. Introduction

Interest in the development of functional foods incorporating bioactives has been on the rise in order to produce health-promoting food products. However, many of those bioactives are water insoluble, i.e. lipophilic, which limits their incorporation into food and beverage formulations. In addition, many bioactives are sensitive to heat, light and oxygen and degrade easily during processing and storage. Therefore, inclusion of lipophilic bioactives into foods to develop products with improved quality, shelf life and nutritional value is challenging. Encapsulation of bioactives in solid lipid particles can be used as a method to overcome the difficulties associated with the incorporation of lipophilic bioactives. Solid lipid particles are one of the well-known lipid-based delivery systems for bioactives and drugs (Scalia et al., 2015; Yada et al., 2014; Tamjidi et al., 2013). However, conventional production methods such as high pressure homogenization, microemulsion method, and oil-in-water emulsion precipitation have several disadvantages such as coarse particles with a broad particle size distribution, degradation of the product due to thermal and mechanical stress, or the contamination of the particles with petroleum-based toxic solvents, limited bioactive loading capacity and scale up issues (Weiss et al., 2008). Therefore, a simple, efficient and green method that eliminates the use of organic solvents is needed to produce a variety of lipid particles as encapsulation systems for lipophilic bioactives.

There is a growing interest in particle design using supercritical carbon dioxide (SC–CO_2) technology, targeting applications in the pharmaceutical, nutraceutical, food, cosmetic and specialty chemicals industries (Hakuta et al., 2003; Weidner, 2009) as a method of "green" manufacturing due to the well-known advantages of CO_2 in terms of its nontoxicity, non-flammability, availability in large quantities, tunable solvent properties, and moderate critical temperature and pressure (31.1 °C and 7.4 MPa) (Ciftci et al., 2012). Significant developments in the supercritical fluid processing of fats and oils have led to advanced methods, opening up new application areas such as nanoparticle formation, encapsulation and crystal design (Temelli, 2009; Moreno-Calvo et al., 2014; Weidner, 2009). A variety of supercritical processes, including supercritical antisolvent (SAS), rapid expansion of supercritical solutions (RESS) and precipitation from gas saturated solutions (PGSS) have been developed to produce a range of particles loaded with drugs and bioactives (Jung and Perrut, 2001). In SAS, the bioactive substance and a carrier are first dissolved or suspended in an organic solvent, which are then precipitated upon contact with SC-

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CO_2, acting as an antisolvent. SC-CO_2 acts as a solvent in RESS, where a bioactive compound is dissolved in the SC-CO_2 and then the solution is rapidly depressurized through a nozzle, resulting in the formation of fine particles. In PGSS, SC-CO_2 acts as a solute, which is dissolved in the liquid material and then the expanded liquid phase is sprayed and depressurized through a nozzle. Despite these developments, there are only a few reports focusing on the micronization of solid lipids using supercritical fluid technology and more specifically targeting the encapsulation of food-related bioactives. Current fat micronization methods to produce fat powder are based on spray drying. Fat powder is used in the food industry for various applications such as infant formulas, sauces, soups, bakery products and creams. An alternative method for powder fat formation and encapsulation of bioactives for food applications would be welcomed. Lubary et al. (2011) reported milk fat micronization using the supercritical melt micronization process, which is similar to PGSS. In this process, the molten fat is saturated with CO_2 under pressure followed by spraying. Fats and oils can dissolve up to 30% CO_2 depending on temperature and pressure as well as the lipid composition (Weidner, 2009). Solubilization of such high levels of gas has a significant impact on the physical properties of the CO_2-saturated lipid phase with a substantial reduction in viscosity, melting point and interfacial tension, which makes spraying and expansion of the mixture through a nozzle possible (Weidner, 2009). Thus, nano- and micro-sized particles can be formed depending on the process parameters and nozzle diameter. PGSS was successfully used to encapsulate lavandin oil in poly-(ε-caprolactone) (Varona et al., 2013), to produce lipid microparticles containing bioactive molecules functionalized with polyethylene glycol (Vezzu et al., 2010) and to prepare microcomposites of theophylline and hydrogenated palm oil with an encapsulation efficiency of up to 35% (Rodrigues et al., 2004). In another study, Paz et al. (2012) achieved β-carotene encapsulation efficiency of 30–60% in soybean lecithin using a PGSS-drying process. Chattopadhyay et al. (2007) reported the production of solid lipid nanoparticle suspensions using supercritical fluid extraction of emulsions (SFEE). In this process, first an oil-in-water (O/W) type of emulsion is formed using an organic solvent containing the dissolved solute, and then the emulsion is contacted with SC-CO_2, which acts as an antisolvent while extracting the solvent at the same time.

Recently, Ciftci and Temelli (2014) reported the melting point depression of solid lipids (fully hydrogenated canola oil, tristearin, monostearin, stearic acid, trilaurin, lauric acid, cocoa butter, and coconut oil) upon saturation with high pressure CO_2. The present study takes advantage of the melting point depression to produce solid lipid particles that can be loaded with bioactives using the PGSS process. These solid lipid particles can then be used to incorporate lipophilic bioactives into various food systems. Decreased melting point can protect the heat labile bioactives by avoiding the use of higher temperatures during processing. As well, the solid lipid matrix provides good biocompatibility for human consumption, controlled release of the bioactive, increased physical stability and increased protection of the bioactive from environmental conditions (Weiss et al., 2008).

The main objective of this study was to adopt the PGSS process for the production of solid lipid microparticles from fully hydrogenated canola oil (FHCO). A variety of liquid and solid lipids such as stearic acid, oleic acid, corn oil and carnauba wax has been evaluated as carriers for the delivery of bioactives as summarized by Tamjidi et al. (2013); however, FHCO has not been used for this purpose previously. FHCO is an inexpensive *trans*-free solid fat available in large volumes, and therefore, it is suitable for solid lipid microparticle production. As well, better understanding of the impact of processing parameters on the characteristics of the particles obtained is essential for further process development. Therefore, the specific objectives were: (a) to investigate the effects of process parameters such as pressure and nozzle diameter on the morphology, size, polymorphism and thermal properties of the particles obtained using FHCO, and (b) to load the FHCO particles obtained under the optimum conditions with spearmint essential oil as a model bioactive and to investigate the effect of spearmint oil concentration on the essential oil loading capacity. Spearmint essential oil was selected due to its antioxidant and antimicrobial activities (Scherer et al., 2013).

2. Materials and methods

2.1. Materials

FHCO was kindly provided by Canbra Foods Ltd. (Lethbridge, AB, Canada). The major fatty acids in FHCO consisted of 5.0% C16:0, 88.2% C18:0 and 3.3% C18:1 as determined previously (Ciftci and Temelli, 2014). Spearmint essential oil was obtained from Dale Thacker Specialty Crops (Bow Island, AB, Canada). CO_2 (purity of 99.8%, water level <3 ppm) was obtained from Praxair Canada Inc. (Mississauga, ON, Canada).

2.2. Production of lipid microparticles using supercritical carbon dioxide

Solid lipid microparticles were produced from FHCO using the particle formation unit shown in Fig. 1. The particle formation unit was designed and custom built in our laboratory. The unit consisted of a 60 mL high-pressure vessel, a high pressure circulating pump, rupture disc, pressure gauge, depressurization valve and a sample collection vessel. The high pressure vessel was heated with a flexible band heater, and all tubing, valves, rupture disc and gauge were heated with a rope heater wrapped around them. The temperature of the heaters was controlled with digital temperature controllers.

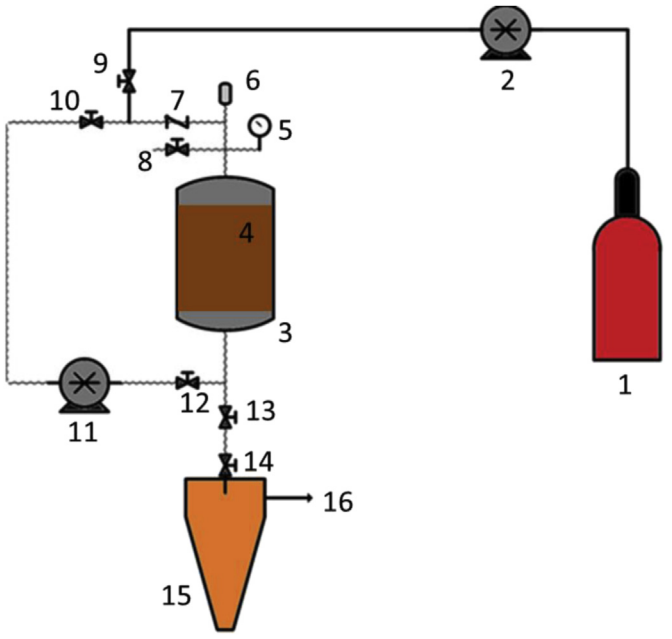

Fig. 1. Schematic diagram of the particle formation unit. (1) CO_2 cylinder, (2) high pressure CO_2 pump, (3) high pressure vessel, (4) heating band, (5) pressure gauge, (6) rupture disc, (7) check valve, (8) sample inlet valve, (9, 10) shut-off valves, (11) high pressure circulating pump, (12, 13) shut-off valves, (14) depressurization valve, (15) sample collection vessel, (16) CO_2 exhaust.