Food Research International 85 (2016) 26–35

Contents lists available at ScienceDirect

Food Research International

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

Replacing modified starch by inulin as prebiotic encapsulant matrix of lipophilic bioactive compounds

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ARTICLE INFO

Article history:
Received 5 March 2016
Received in revised form 6 April 2016
Accepted 9 April 2016
Available online 14 April 2016

Keywords:
Prebiotic wall material
Encapsulation efficiency
Origanum vulgare L.
X-ray diffraction
Thermogravimetric analysis
Thymol entrapment

ABSTRACT

The purpose of this work was to replace modified starch (SF) by inulin (IN), a prebiotic carbohydrate, during emulsification assisted by ultrasound. Oregano extract was encapsulated using five proportions of IN and SF as wall materials. The effect of such substitution on the microparticle characteristics was evaluated. Attempting to contribute with the increasing demand for prebiotic consumption, mixing one part of SF with three parts of IN (1:3, mass basis) yielded encapsulation efficiency equal to 66 ± 1% and the largest thymol retention: 84 ± 9%. Besides the entrapment of thymol, high amount of other compounds present in oregano extract could be entrapped in the polymeric matrix: 92 ± 1%. Reduction of the microparticles sizes when increasing the proportion of inulin was also observed. Comprising such results and those presented for powder morphology, surface extract, particle size distribution, X-ray diffraction and thermal stability, the proportion 1:3 (SF:3IN) is a favorable prebiotic encapsulant matrix for encapsulating oregano extract and retaining target bioactive compounds.

1. Introduction

Several vegetal sources have extractable substances that are industrially desirable in the food, chemical, cosmetic and pharmaceutical fields. One of these sources is oregano (*Origanum vulgare* L.), which contains thymol, carvacrol, gamma-terpinene, alpha-terpinene, alpha-terpineol, linalool, terpinen-4-ol, sabinene and beta-phellandrene, among others (Borgarello, Mezza, Pramparo, & Gayol, 2015; Ruben, Valeria, & Ruben, 2014). Antioxidant and antimicrobial activities of oregano extract are mainly associated with the presence of thymol and carvacrol (Asensio, Grosso, & Juliani, 2015; Falco, Roscigno, Landolfi, Scandolera, & Senatore, 2014), which are efficient against bacterial strains and lipid oxidation. Oregano extract has been used as flavoring agent in food and beverages, as well as in the manufacture of fungicides and insecticides (Kordali, Cakir, Ozer, Cakmakci, Kesdek, & Mete, 2008).

Some sensitive compounds are unstable when exposed at light and oxygen ambient. Thus, the encapsulation of bioactive compounds is a technology used for protecting them into a physical entrapment formed by a homogeneous or a heterogeneous matrix (Costa, Duarte, Bourbon, Pinheiro, Serra, Martins, et al., 2012). Oregano extract, for instance, was encapsulated in chitosan nanoparticles by a two-step process: oil-in-water (o/w) emulsification and ionic gelation (Hosseini, Zandi, Rezaei, & Farahmandghavi, 2013). In another study, gum Arabic and modified starch were used in the encapsulation of oregano essential oil (Botrel, Borges, Fernandes, Viana, Costa, & Marques, 2012). It is seen diverse wall materials from a wide variety of natural and synthetic polymers used for encapsulation processes. However, the wall materials present different actions of protection and influence particle characteristics and retention/release of active substances. Regarding the human intake, some polymers present high energetic content, opposing the consumption of prebiotic substances. As concept introduced by Gibson & Roberfroid (1995), prebiotics are nondigestible food ingredients that beneficially influence the host by stimulating the growth and/or activity of a limited number of bacteria species resident in the colon, and thus improve host health. This definition has been revised along the time, but the main features have mostly been retained.

The great interest in the development of prebiotics is aimed at nondigestible oligosaccharides. Some of the prebiotics are the inulin-type fructans, because they provided evidence of their ability to change the gut flora composition after a short feeding period based on results from in vitro studies and human subjects (Kolida & Gibson, 2007). Inulin is a versatile fructooligosaccharide generally extracted from chicory (Pandey, Soccol, Selvakumar, Soccol, Krieger, & Fontana, 1999) that is applied in the stabilization of proteins and modified drug delivery (Mensink, Frijlink, Maarschalk, & Hinrichs, 2015) or converted in other functional ingredients by inulinases (Mazutti, Skrowonski, Boni, Zabot, Silva, Oliveira, et al., 2010a; Mazutti, Zabot, Boni, Skovronski, Oliveira, Luccio, et al., 2010b).

Although inulin has several applications in diverse areas, including in the food area for decades, the use of inulin as wall material in the

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http://dx.doi.org/10.1016/j.foodres.2016.04.005
0963-9969/

food field is a few exploited. Wall materials commonly used in the encapsulation of bioactive compounds are gums, modified starches, whey proteins and dextrins (Chranioti, Nikoloudaki, & Tzia, 2015; Khazaei, Jafari, Ghorbani, & Kakhki, 2014; Silva, Gomes, Hubinger, Cunha, & Meireles, 2015a; Zandi, Mohebbi, Varidi, & Ramezanian, 2014). However, such substances do not present functional activities as inulin does. Looking for the substitution of gums, maltodextrin or starches by prebiotic materials, some researchers are studying the use of inulin. Recently, Fernandes, Borges, & Botrel (2014a) evaluated the effects of the partial or total replacement of gum Arabic by inulin on the characteristics of rosemary essential oil microencapsulated by spray-drying. Saénz, Tapia, Chávez, & Robert (2009) also reported the ability of inulin for microencapsulation of bioactive compounds from cactus pear fruit. Thus, the use of inulin can favor the application of oregano extract in functional foods.

Accompanying the global changes and demands, the market of ingredients and additives supplied to the food industry is recently overcoming the challenge of substituting synthetic substances by natural compounds. In the past few decades, considerable efforts have been devoted to developing functional products that promote healthiness and well-being. One of these recent efforts in the food field stands for using functional encapsulating matrices with the objective to protect bioactive compounds. Based on this trend of consuming compounds with functional activities, this scientific study proposes the substitution of a high caloric substance (modified starch) by a prebiotic carbohydrate with low content of calories (inulin) for the encapsulation of bioactive compounds. The objective was to evaluate the main characteristics of microparticles containing oregano extract formed with five proportions of modified starch and inulin as wall materials, aiming to use inulin to a feasible extent. The main characteristics assessed were: extract entrapment efficiency, encapsulation efficiency, surface extract, thymol retention, particle size distribution, micrographs of microparticles, X-ray diffraction and thermal stability of microparticles.

2. Material and Methods

2.1. Obtaining oregano extract by supercritical fluid extraction

Supercritical fluid extraction has been increasingly applied to obtain several bioactive compounds (Moraes, Zabot, & Meireles, 2015; Zabot, Moraes, Carvalho, & Meireles, 2015). Then, oregano extract was obtained with supercritical CO_2 at 50 °C and 20 MPa (Diaz, 2010) using the SFE-2×1L equipment described in previously studies (Zabot, Moraes, & Meireles, 2014; Zabot, Moraes, Petenate, & Meireles, 2014). Firstly, dry oregano leaves (acquired from local market) were comminuted until mean diameter size of 0.9 ± 0.1 mm. After, 480 g of milled leaves was loaded into the extraction vessel of 1 L. The extraction bed was pressurized with CO_2 and maintained at static extraction during 20 min. Dynamic extraction started with constant CO_2 flow rate of 28 g/min during 120 min, indicating a solvent mass to feed mass (S/F) ratio of 7. The extract was collected and immediately stored for further applications. Five extractions assays were performed aiming to reach sufficient extract for the emulsification step.

2.2. Wall materials

Wall materials used for encapsulating oregano extract were as follows: Snow-Flake (SF) E6131 chemically modified starch (Ingredion Brasil Ingredientes Industriais Ltda., Mogi Guaçu, SP, Brazil) (SF wall material) and chicory inulin (IN) Orafti®GR (with a degree of polymerization higher than 10) supplied by Beneo-Orafti (São Paulo, Brazil) (IN wall material). Silva, Gomes, Hubinger, Cunha, & Meireles (2015a) reported the characterization of these wall materials used in this study.

2.3. Emulsions of oregano extract

The emulsion characteristics were evaluated after partially replacing the modified starch (SF) for inulin (IN). The procedure was accomplished as follows: SF was added to ultra-pure water supplied by a Milli-Q Advantage water purifier system (Millipore, Bedford, USA) and the biopolymer suspension was prepared 24 h before the emulsification process. The suspension was maintained static at approximately 25 °C during 24 h to ensure the complete saturation of the molecules of SF. After this period, IN was dissolved in ultra-pure water at 90 °C and mixed with SF according to the defined concentration (Table 1) for each essay. After saturation, 20 wt.% of oregano extract (relative to the mass of total solids) was added to the suspension, that is, 5 g of extract/100 g of emulsion. The concentration of total solids in the emulsion (emulsifying + extract) was 25 g/100 g of emulsion.

Aliquots of 40 mL of the suspensions were sonicated using a 13 mm diameter and 19 kHz ultrasonic probe (Unique, Disruptor, 800 W, Indaiatuba, Brazil) for obtaining the emulsions. The probe height contacting the emulsions was standardized to 40 mm (Silva, Gomes, Hubinger, Cunha, & Meireles, 2015a). For ultrasonication, the same equivalent energy density reported by Silva, Zabot, & Meireles (2015c) was used, representing a nominal power of 760 W during 7 min. The experimental runs were carried out in duplicate, accounting 10 runs.

2.4. Characterization of emulsions

2.4.1. Droplet size distribution

Droplet size distribution and mean diameter of the emulsion droplets were determined by light scattering technique using laser diffraction (Mastersizer 2000 Malvern Instruments Ltd, Malvern, UK). The mean diameter was calculated based on the mean diameter of a sphere of similar area, superficial mean diameter ($D_{[3,2]}$), as Equation (1). Polydispersity index (PDI) was calculated as Equation (2). All samples were analyzed in triplicate, using the wet method, with dispersion in water and refractive index of 1.52.

$$D_{[3,2]} = \frac{\sum_{i=1}^{k} n_i \cdot D_i^3}{\sum_{i=1}^{k} n_i \cdot D_i^2} \quad (1)$$

$$PDI = \frac{D_{90} - D_{10}}{D_{50}} \quad (2)$$

Where: D_i is the mean diameter of the droplets; n_i is the number of droplets; and D_{10}, D_{50} and D_{90} are the particle diameters at the 10th, 50th and 90th percentile of particles undersized, respectively.

2.5. Powder formation by freeze-drying

Immediately after formed, the emulsions were frozen in aluminum plates at -40 °C for 3 h and then subjected to freeze-drying (FD) process. Drying was performed in a freeze-dryer system (Liobras, L 101, Sao Carlos, Brazil). The dried emulsions were converted into fine powders through maceration. The experimental runs were performed in duplicate.

2.6. Particle characterization

2.6.1. Moisture

Moisture content of microparticles obtained after partially replacing modified starch by inulin was determined gravimetrically in a forced circulation oven at 105 °C until reaching constant weight (AOAC, 1997).