LWT - Food Science and Technology 50 (2013) 569–574

Contents lists available at SciVerse ScienceDirect

LWT - Food Science and Technology

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

Olive oil and lemon salad dressing microencapsulated by freeze-drying

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ARTICLE INFO

Article history:
Received 11 April 2012
Received in revised form
31 July 2012
Accepted 7 August 2012

Keywords:
Food emulsion
Maltodextrin
Arabic gum
Lemon juice
Olive oil

ABSTRACT

An instantaneous food emulsion was formulated containing olive oil and lemon juice using combinations of polymers, such as Alginate (ALG), Arabic gum (AG), Maltodextrin (MD) and Carboxymethyl cellulose (CMC) and freeze-dried, aiming at the development of a new microencapsulated product. The characterization of particle size, the surface analysis by scanning electron microscopy, the X-ray diffraction and the differential scanning calorimetry were performed with the emulsions that showed a good oil encapsulation. Mixtures of maltodextrin and arabic gum showed the lowest average values of particle size. Moreover, these samples presented rounded shapes and some depressions shown by scanning electron microscopy and proved to be an amorphous material by X-Ray Diffraction. The glass transition temperatures of samples C (12.5 g/100 g MD and 7.5 g/100 g AG), 146.60 °C, and D (10 g/100 g MD and 8.5 g/100 g AG), 147.54 °C, were similar, because the type of polymers was similar. This study shows that it is possible to microencapsulate emulsion oil in water (1:1) by freeze-drying to use an instant sauce salad.

1. Introduction

The molecular gastronomy is the study of chemical and physical processes that occur during food preparation. Considering this, new methods and techniques can be created or improved. The microencapsulation is a process in which tiny particles or droplets are surrounded by a coating, or embedded in a homogeneous or heterogeneous matrix, to form small capsules (Gharsellaoui, Roudaut, & Chambin, 2007). Most edible oils are chemically unstable and susceptible to oxidative deterioration, especially when exposed to oxygen, light, moisture, and high temperature. That oxidative degradation results in a loss of nutritional quality and a development of undesired flavors, affecting shelf stability and sensory properties of the oil (Velasco, Dobarganes, & Márques-Ruiz, 2003). Therefore, the encapsulation by freeze drying of salad dressing, which is composed of olive oil and lemon juice, aims to increase the stability of this food by decreased activity of water, contributing to the reduction of weight and density of the product and reducing costs in transportation and storage. Lyophilization is carried out using a simple principle of physics called sublimation. Sublimation is the transition of a substance from the solid to the vapor state, without first passing through an intermediate liquid phase. The process of lyophilization consists of freezing the food so that the water in the food becomes ice, under a vacuum, sublimating the ice directly into water vapor and draw off the water vapor. Once the ice is sublimated, the foods are freeze-dried and can be removed from the machine. Emulsions as salad dressings can quickly lose stability, differently from dry presentation that allows an increase in shelf life, retaining the functional and nutritional compounds for longer time; besides, it is easier to commercialize (Fonseca, 2008; Profiqua, 2002). Food emulsions are compositionally complex; their droplets are stabilized within different extents by proteins, small-molecule surfactants (emulsifiers), and polysaccharides (Dickinson, 2010). The alginates are natural polymers that are widely regarded as biocompatible and non-toxic (Thevenet, 1988). Carboxymethyl cellulose (CMC) as a typical hydrocolloid, has no direct influence on the taste and flavor of foodstuffs, but at the same time has a significant effect on gel formation, water retention, emulsifying and aroma retention. In the food industry CMC is used as a stabilizer, binder, thickener, suspending and water-retaining agent, in ice-cream and other frozen desserts, sauces and creams (Hegeduši, Herceg, & Rimac, 2000). Maltodextrins are widely used in food emulsions as stabilizers (Chronakis & Kasapis, 1995) and their addition is mainly performed in materials that are hard drying (Sablani, Shestha and Bhandari, 2008). Arabic gum is a complex heteropolysaccharide with a highly ramified structure, with the main chain formed by D-galactopyranose units (Bemiller & Whistler, 1996). It has been used

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http://dx.doi.org/10.1016/j.lwt.2012.08.005

as an encapsulating agent in microencapsulation by spray drying because of its good emulsification capacity and low viscosity in aqueous solution (Gabas, Telis, Sobral, & Telis-Romero, 2007). The microencapsulation process transforms oils into easily-handled solids and protects them from oxidation through a solid wall that acts as a physical barrier limiting the diffusion of oxygen (Gharsellaoui, Roundaut, Chambin, Volley, & Saruel, 2007). To evaluate the procedure encapsulation, techniques with laser diffraction were chosen in this work in order to measure the size of particles formed, along with the scanning electron microscopy to visualize the surface of the tablet, the X-ray diffraction to evaluate the amorphous material and differential scanning calorimeter to determine the glass transition temperature (Tg) values. Thus, this work aimed to characterize a microencapsulated food emulsion based on olive oil and lemon juice, obtained by freeze-drying to use an instant sauce salad.

2. Materials and methods

2.1. Materials

Arabic gum, carboxymethyl cellulose and alginate were purchased from VETEC® LTDA, (Rio de Janeiro, Brazil) and maltodextrin, with dextrose equivalent of ca 20, was obtained as a gift sample from PluryQuimica®, (São Paulo, Brazil). The olive oil Portucale (Vienes® Fátima, Portugal) and lemon were purchased in a local supermarket (Cofrutagem Araquara LTDA, São Paulo, Brazil).

2.2. Oil-in-water emulsions preparation

The polysaccharides were slowly dispersed in a lemon juice (10 ml), followed by the addition of olive oil (10 ml) and homogenized for 40 s at 9600 rpm by using an Ultra-Turrax T25 homogenizer (IKA Instruments, Germany) equipped with a dispersing tool. Different concentrations of hydro-soluble polymers used to prepare the samples are summarized in Table 1. These samples were stored in round plastic pots with 3.6 cm diameter and 2.0 cm in height, frozen at −50 °C for 24 h and then freeze-dried in equipment of Enterprise® for approximately 18 h. The samples remained with the same form of the container. Only the samples with all oil encapsulated in container were characterized.

2.3. Particle size

A little amount of a tablet was dissolved in 10 ml of propan-2-ol, for 30 s in an ultrasonic, model Ultra Clean 800A Unic®. After, the particle size was measured by laser diffraction (SALD-2201, SHIMADZU®) obtaining particles with a refraction index of 1.70–0.20. Analyzes were performed in duplicate and the mean, the mode and the cumulative distribution d [25], d [50] and d [75] was obtained. The cumulative distribution d [25], d [50] and d [75] are size values corresponding to the cumulative distribution at 25%, 50% and 75%, respectively. Thus, the d [25] represents a size value below which 25% of the cumulative distribution is present.

Table 1
Emulsion compositions, considering olive oil–lemon juice (1:1) microencapsulated.

	Emulsions			
Polymers (g/100 g)	A	B	C	D
Alginate (ALG)		2.5		
Gum arabic (GA)			7.5	8.5
Maltodextrin^{DE10} (MD)	10	12	12.5	10
Carboxymethyl cellulose (CMC)	1.0			

2.4. Surface analysis by scanning electron microscopy (SEM)

Images of samples and polymers (in a powder presentation and coated by a gold blade) were recorded using a Scanning Electron Microscope (model JSM5800LV-JEOL®, Japan), operated at 20 kV electron bean acceleration voltage. These images were magnified 5.000 and 10.000 times.

2.5. X-ray diffraction (XRD)

X-ray diffraction measurements were performed in a diffractometer X'Pert PRO (PANalytical), and data were collected over an angular range of 10–100°, at a count rate of 1 s per step of 0.05°.

2.6. Differential scanning calorimetry (DSC) and moisture

A differential scanning calorimeter (Diamond DSC, Perkin Elmer) was used to determine the glass transition temperature (Tg) values. Samples, with approximately 5.0 mg, were enclosed in hermetically sealed aluminum pans just before analysis and then loaded into the equipment at room temperature. The DSC curves were obtained in the temperature range of −20 to 200 °C and the samples were heated at 20 °C/min under the inert N_2 atmosphere. The Tg was measured by the peak half height of samples.

2.7. Centesimal composition

The Centesimal Composition was the determination of humidity, ashes, protein by Kjeldahl and lipid by Bligh Dyer method (Instituto Adolfo Lutz, 1985). Carbohydrate content was calculated by subtracting humidity rate, ashes, protein and lipid from a 100 g sample. Total Energetic Value was calculated based on Atawer conversion factors, which considers 4 kcal/g of protein, 4 kcal/g of carbohydrate and 9 kcal/g of lipid (Lima, Silma, Trindade, Torres, & Manchini-Filho, 2007).

3. Results and discussion

In order to trap the amount of oil contained in this type of emulsion, the proportions of polymers reported in this study were taken from a preliminary study. First, the polymers used by Silva, Rocha-Leão, and Coelho (2010) were tested in order to trap the emulsion, but despite the author have been able to maintain stable emulsion, the proportions used were not sufficient to microencapsulate the oil. Other combinations of polymers such as modified starch, arabic gum, maltodextrin, dextrin, vicilina, alginate, carboxymethyl cellulose and beta-cyclodextrin, were tested and many defects were seen in the samples after being freeze-dried. The most recurrent defects seen were: 1) the unencapsulated oil, because of insufficient amount of polymer, 2) samples with intense yellow color, in which occurred fast oxidation and exposure to oil, 3) freeze-dried sample showing spongy layer, probably owing to emulsion separation of phase during the freezing, and 4) the presence of holes in the middle of sample. After making a screen to discover the lowest ratio of polymers to encapsulate the emulsion shown in Table 1, the studies of characterization continued for discovering the best type of polymer to be used. The polymer proportions shown in Table 1 are those that kept the state solid product without suffering collapse after freeze-drying. The tablet had the format of the container in which it was dried; this was round with a diameter of 3.5 cm. The process of freeze drying involves removing water from food without using high temperature, the food is quickly frozen producing smaller ice crystals; then the sublimation process occurs, ensuring food sensorial characteristics, without degradation of substances (Pegg & Shahidi, 2007).