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Encapsulation of citral in formulations containing sucrose or trehalose: Emulsions properties and stability

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ABSTRACT

The performance of formulations for citral encapsulation by spray drying was assessed. Focus was put on the emulsions properties and stability. Formulations contained sucrose or trehalose with or without maltodextrin and a modified starch as emulsifier. Viscosity, molecular mobility, microstructure, droplet size distribution, and stability were analyzed before and after spray-drying. Instability processes operated in the emulsions bulk, being flocculation the main one. Droplet size and viscosity influenced the emulsions stability. An increase in viscosity attained by the addition of MD delayed the global destabilization processes. Emulsion properties and stability were similar between the formulations containing sucrose or trehalose. Also, there were no differences regarding aroma retention or sensorial perception of citral. The mixture of maltodextrin and trehalose presented higher T_g values allowing to maintain the glassy state of the powder in broader temperature and relative humidity conditions. Trehalose presented a promising performance as an ingredient in the carrier formulation to encapsulate citric flavors.

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1. Introduction

Microencapsulation of flavors is of great importance in the flavoring and food industries. This process is carried out in order to obtain a dry flavor powder, which is easy to handle due to the advantage of the solid state. Flavor encapsulation can be accomplished by a variety of methods such as spray-drying, spray-chilling or spray-cooling, freeze-drying, extrusion, coacervation and molecular inclusion, among others. The choice of appropriate microencapsulation technique depends on the end use of the product and the conditions involved in the manufacturing process (Madene et al., 2006). The two major industrial processes are spray-drying and extrusion (Beristain et al., 1996; Goubet et al., 1998; Yamamoto et al., 2011), although freeze-drying (Yang et al., 2011) and coacervation (Patrick et al., 2013) are also used.

The most common way to perform the microencapsulation of flavors is by spray drying, which is the transformation of a feed from a fluid state (solution, dispersion, emulsion) to a dried particulate form. This technology results suitable; not only in providing protection against degradative reactions and preventing the loss of flavor, but also in giving the controlled release of flavors.

The manufacturing and storage processes, packaging materials and ingredients present in foods often cause modifications in overall flavor by reducing aroma intensity or producing off-flavor components. On the other hand, many factors linked to aroma (soluble or not in water) affect the global quality of the food: (a) the physico-chemical properties; (b) the matrix affinity, since some flavors are more stable in carbohydrates based matrix carriers and others are more stable in lipid-based coatings; and (c) the concentration and interactions of volatile aroma molecules with food components (Landy et al., 1995).

Emulsions are complex two-phase systems, made by droplets dispersed in a continuous phase. They are inherently unstable due to the difference in specific gravity between these two phases. Specifically, oil in water emulsions (o/w)

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constitute colloidal systems in which small lipid droplets are dispersed in an aqueous medium and form the basis of many kinds of foods. The conversion of two separate immiscible liquids into an emulsion, or the reduction of the size of the droplets in a preexisting emulsion, is achieved by homogenization processes. Conventional methods to prepare emulsions rely on stirring equipment, colloid mills, homogenizers, ultrasonics or microfluidizers (Charcosset, 2009).

The retention during spray drying of flavors, insoluble in water (Soottitantawat et al., 2003) can be largely influenced by the emulsion droplets size. Soottitantawat et al. (2003) have found that a smaller emulsion size is needed for the higher retention of non soluble flavors (for a certain range of sizes).

For technologists, the ability to control the properties and stability of food emulsions is a key factor in the development of better products. In this contribution, focus was put on the encapsulation of citral flavor (non soluble in water). The choice of components for the carrier matrix was based on obtaining a stable powder capable of efficiently encapsulate citral. In this sense, a modified starch was incorporated as an emulsifier, due to its ability of lowering the surface tension at the oil–water interface and imparting viscosity to the aqueous continuous phase (Prochaska et al., 2007). The addition of maltodextrin (MD) to the formulation provides a low cost, bland polysaccharide that is not particularly surface-active; and its main stabilizing action in oil-in-water emulsions is believed to be through viscosity modification or even gelation of the aqueous continuous phase surrounding the oil droplets when the used concentration is appropriate. Also, the high glass transition temperature (T_g) of low dextrose equivalent MD provides good physical stability to the dried powders (Bhandari and Hartel, 2005). Regarding the spray drying process, it is well known that the type of carriers affects the flavor retention, for this reason, disaccharides, mainly sucrose, are sometimes included in commercial formulations to improve retention characteristics (Menting et al., 1970). Concerning the powder stability, sucrose has some negative characteristics as it has a relatively low T_g, which has shown to affect the storage stability of orange and strawberry spray-dried flavors (Busso Casati et al., 2007). On the other hand, trehalose could be used as an alternative to use in microencapsulation, as it shows higher T_g values compared to sucrose (Komes et al., 2003). Komes et al. (2003) observed an improvement in aroma retention in dehydrated strawberry purees in the presence of trehalose compared to sucrose. In a previous work, no significant differences in the perception of citral flavor in emulsions containing trehalose or sucrose were observed (Sosa et al., 2011). So, the potential use of trehalose as a component of the carrier formulation for citral encapsulation was assessed in the present contribution.

Therefore, the objective of this work was to comparatively determine the performance of four sugar based formulations for citral encapsulation by spray-drying. Emulsions attributes and stability were analyzed before, and after the spray-drying process (upon hydration of the powder with water).

2. Materials and methods

2.1. Materials

Sucrose and trehalose, of analytical grade, were purchased from Merk (Germany) and Cargill Inc. (Wayzata, MN, USA), respectively, and Maltodextrin (MD) with a dextrose equivalent of 12 was from Givaudan S.A. (Argentina). The commercial modified starch used as emulsifier was Capsul (Gelfix, Argentina). The dispersed phase was constituted by citral "Extra Fino" kindly given by Saporiti Sabores (Argentina). Deionized water was used in all cases.

2.2. Emulsions preparation

Premixtures were prepared by completely dissolving in water, the starch and either sucrose or trehalose, or a mixture (1:1) of MD plus S or MD plus T. These mixtures constituted the continuous phases of the emulsions, and after adding the required citral amount, will constitute forward in the text the so called S, T, SMD and TMD formulations. In all cases, the total mass of dry solids was 40 g per 100 g of emulsion. The exact mass of each dry component in the formulations is detailed in Table 1. 200 mL of each pre-emulsion was obtained by mixing the ingredients in a Griffin & George (Loughborough, UK) stirrer. This operation was carried out for 2 min, at 25 °C and 750 rpm. Emulsions formation was completed by submitting the pre-emulsion to a high speed blender, Sorvall OMNI MIXER 17106-OMNI Corporation International, Waterbury (CT, USA), operating at 16,000 rpm for 10 min. The temperature increase was avoided by keeping the sample into an ice bath during stirring.

One set of samples were immediately spray-dried. These samples were destined to the study of reconstituted emulsions (RE). The remaining samples constituted the so called fresh emulsions (FE), which were immediately analyzed.

2.3. Spray-drying

The emulsions were spray-dried using a laboratory scale device, Mini Spray Dryer Büchi B290 (Flawil, Switzerland). The operational conditions of the drying process were: the co-current flow method, inlet air temperature 175 ± 3 °C, outlet air temperature 83 ± 3 °C, flow rate 8 mL/min, air pressure 3.2 bar, two-fluid nozzle diameter 1.5 mm. Once obtained, the powders were collected into sealed PVDC bags and then stored at −18 °C until analysis. The powders moisture (% in dry basis) was determined in duplicate by the Karl Fischer method, using a Mettler Toledo DL 31 titrator (Schwerzenbach, Switzerland). After spray-drying, the powders a_w values ranged between 0.098 and 0.183 with standard deviations lower than 0.005. The obtained moisture values were 4.30 ± 0.14, 4.44 ± 0.07, 3.82 ± 0.22, and 4.69 ± 0.10 for S, T, SMD, and TMD powders, respectively. Dry powders were dissolved in deionized water to give the RE (keeping the original solids content 40%).

2.4. Viscosity determination

Apparent viscosity of emulsions was determined from the respective flow curves, whose points were collected with a cone and plate DV-LVT Brookfield viscometer (Middleboro, MA, USA) using a stainless steel cone 41, at room temperature 25 ± 1 °C. A shear rate range between 0.5 and 200 s^{-1} was applied (Kawakatsu et al., 2001). The sample volume was 2 mL. All the determinations were made at least in duplicate.

2.5. Distribution and size of emulsion droplets

The droplet size of emulsions was measured by light scattering using a Mastersizer 2000 device with a dispersion unit Hydro 2000MU (Malvern Instruments Ltd., UK). Immediately

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