FOOD AND BIOPRODUCTS PROCESSING 97 (2016) 63–75

Contents lists available at ScienceDirect

Food and Bioproducts Processing

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

Effect of type of encapsulating agent on physical properties of edible films based on alginate and thyme oil

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ARTICLE INFO

Article history:
Received 3 July 2015
Received in revised form
16 October 2015
Accepted 23 November 2015
Available online 1 December 2015

Keywords:
Alginate
Thyme oil
Encapsulating agents
Films
Emulsions
Physical properties

ABSTRACT

Active edible coatings incorporating antimicrobial agents such as thyme oil are studied to improve the shelf-life of fresh foods. The type of encapsulating agents used to microencapsulate the active compound could affect both, emulsions and film's physical properties. The aim was to study the effect of different encapsulating agents (trehalose, β-cyclodextrin and Tween 20) on physical and antimicrobial properties of alginate/thyme oil emulsions and films. Physical and antimicrobial characterization of film-forming emulsion and films were developed. Results showed differences in rheological behavior, particle size and stability of emulsions by encapsulating type. The highest stability of emulsions containing Tween 20 (instability of 0.69%) was attributed to more interactions between components (observed by FTIR) and the lowest particle size, but other samples also showed high stability (instability <5%). The stability of emulsions did not correlate with films microstructure indicating that interactions amount components changed when solvent was evaporated. Films containing trehalose and β-cyclodextrin had less color and opacity than Tween 20, which is an advantage by industrial applications by coating due cannot affect the organoleptic characteristics of a fresh product. In conclusion, it is important the choice of encapsulating agent in order to use for developing active coatings with potential for applications in fresh food.

1. Introduction

Food quality and safety are the major concerns in the food industry, especially on preventing chemical and microbiological deterioration, as well as the exposure of labile biomolecules (vitamins, essential oils, colorants) to extreme humidity and temperature conditions, which leads them to degradation (Ponce Cevallos et al., 2010). For this, the interest in development of novel ways to prolong the shelf life of food products has increased in the last years. Edible films as a solid sheet can be applied on the surface of the food systems and can be a good alternative to improve food quality by serving as selective barriers to moisture transfer, carbon dioxide permeability, oxygen uptake, lipid oxidation, and losses of volatile aromas and flavors (Kester and Fennema, 1986; Di Piero et al., 2011; Lacroix and Vu, 2014).

These films are thin layers of edible material such as protein, polysaccharide and lipid. Among polysaccharides, alginate, is widely used to produce films (Rojas-Grau et al., 2007; Córdova et al., 2015), which can be used to carry active

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http://dx.doi.org/10.1016/j.fbp.2015.11.001

ingredients, for example antioxidant and antimicrobial agents (Tapia et al., 2008). Studies have focused on the incorporation of natural active compounds in edible film since they may widen the functionality protecting the product from microbial spoilage and thus extend their shelf-life (Mchugh and Senesi, 2000; Alboofetileh et al., 2014; Karagöz et al., 2010; Peretto et al., 2014).

Essential oils and their constituents have been frequently used as flavors agents in foods and are categorized as Generally Recognized as Safe (GRAS) (López et al., 2007; Hill et al., 2013). Essential oils rich in phenolic compounds have been reported to have a wide spectrum of antimicrobial activity. Among these, clove, oregano, rosemary, sage vanillin and oils have been found to be effective antimicrobial agent (Holley and Patel, 2004; Burt, 2004), specifically thyme and oregano essential oils have been pointed out to possess better antimicrobial potential for meat applications because carvacrol, thymol, c-therpinene and *p*-cymene are the principal constituents of oregano and thyme essential oils (Burt, 2004; Solomakos et al., 2008). However these compounds are highly volatile, insoluble in water due to their lipophilic nature and may suffer degradation reactions in the presence of ambient oxygen and light as well as under the action of moderate temperatures (Beyki et al., 2014).

One way to allow their incorporation into matrices is through an encapsulation process which can be classified as: physical-chemical processes (coacervation, emulsion evaporation), chemical processes (gelling, complexation) and physical processes (fluidized bed, extrusion, spray drying, spray cooling), where the emulsion evaporation has been used as release system for a lot of ingredients and functional foods and is probably the release of lipids system most used in the food industry (Esparza and Irache, 2011). For the preparation of emulsion is possible to use different encapsulating agents such as cyclodextrins, tween 80 and 20, trehalose, maltodextrin, pectin, capsul (Drusch et al., 2006; Álvarez-Cerimedo et al., 2008; Ponce Cevallos et al., 2010; Karunasawat and Anprung, 2010; Aguiar et al., 2012; Hill et al., 2013), due to their ability to facilitate the formation, improve the stability, and produce desirable physicochemical properties in oil-in-water emulsions.

Cyclodextrins are cyclic oligosaccharides where the central cavity is hydrophobic, while the rims of the surrounding walls are hydrophilic (Del Valle, 2004; Gibara et al., 2015). However, much of the interest in cyclodextrin arises precisely from their ability to encapsulate hydrophobic molecules with suitable size inside their annulus to form inclusion complexes and then to alter physical, chemical, and biological properties of the encapsulated guest molecules (Ponce Cevallos et al., 2010). In the same way, trehalose appears to be very promising for microencapsulation because it possesses a uniquely high glass transition temperature and remains in the glassy state at temperatures higher than other sugars and has a greater capability of stabilizing proteins, lipids or carbohydrates (Drusch et al., 2006). Besides, it has been reported that edible hydrocolloid films prepared with trehalose could act as very effective barriers to gases, especially to CO_2, showing a higher barrier compared to similar films obtained with glycerol as plasticizer (Giosafatto et al., 2014).

Drusch et al. (2006) who used 10% trehalose concentration, reported that encapsulation in a trehalose matrix offers protection for the microencapsulated fish oil for a certain period of time and Hill et al. (2013) utilized beta-cyclodextrin to encapsulate different essential oils (*trans*-cinnamaldehyde, eugenol, cinnamon bark, and clove bud extracts) mixed in an aqueous solution in a 1:1 molecular ratio. He reported that such essential oil inclusion complexes could be useful antimicrobial delivery systems with a broad spectrum of application in food systems where Gram-positive and negative bacteria could grow.

Little work has been done on the effect of encapsulating agent on physical properties of films. However, the type of encapsulating molecules could affect both, emulsions and film physical properties. The aim of the present work was to study the effect of different encapsulating agents on physical properties of alginate/thyme oil emulsions and films. Both systems were investigated in order to obtain information that can be used for developing coatings with potential for applications in fresh food.

2. Materials and methods

2.1. *Composition and preparation of samples*

Thyme oil was purchased from commercial local company (Ambar, Santiago, Chile); Sodium alginate (G5706401), trehalose dihydrate [D-glucopyranosyl-(1-1)-Dglucopyranoside] (S-518) and sorbitol (20/60 DC) were provided by Blumos S.A. (Santiago, Chile); calcium carbonate was purchased from Winkler Ltda. (Santiago, Chile); β-cyclodextrin and Tween 20 were obtained from Merck S.A. and Sigma Aldrich Co. (Santiago, Chile), respectively. All reagents were used without any further purification.

Film-forming emulsions were prepared by mixing 100 g of aqueous phase and 0.5% (wt/wt) of thyme oil phase. In all cases, aqueous phase was prepared with sodium alginate 1% (wt/wt), sorbitol 1% (wt/wt) and calcium carbonate 0.02% (wt/wt). Three different encapsulating agents were used: trehalose (0.2; 0.4; 0.6 and 0.8% wt/wt), β-cyclodextrin (0.1; 0.15; 0.2 and 0.25% wt/wt), and Tween 20 (1; 1.5; 2 and 2.5% wt/wt). Emulsions were prepared by magnetic stirred moderately at 40 °C until the solutes were completely dissolved. Then, thyme oil was incorporated using an homogenizer (Trhistor Regler TR50, Germany) at 5000 rpm for 1 min. Films were obtained from film-forming emulsions by casting method at 40 °C for 20 h in forced air oven (Wiseven DaihanScientific WOF-105, Korea). Film control with thyme oil was prepared without any encapsulating agent. Films were stored in desiccators for 24 h prior to analysis.

2.2. *Film-forming emulsion characterization*

2.2.1. *Flow behavior*

Flow measurements were carried out in a controlled stress rheometer (CarriMed, CSL2 100, TA Instruments, UK), using parallel-plate geometry (40 mm diameter; 1.5 mm gap). Measurements were run at 25 ± 1 °C and after loading the sample, it was allowed to stand for 3 min to stabilize and reach the desired temperature. At least two batches of each composition were prepared and each batch was measured in triplicate, using a fresh sample for each measurement.

Curve flows were obtained by recording shear stress values, which was increased from 0 to 250 Pa and decreased from 250 to 0 in 240 s. Experimental data from ascending flow curves of