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Carotenoid stability in high total solid spray dried emulsions with gum Arabic layered interface and trehalose–WPI composites as wall materials

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ABSTRACT

The present study investigated the ability of spray dried single layer (SL) and layer-by-layer (LBL) high total solid emulsions with carbohydrate (trehalose) and non-carbohydrate (WPI) solids to stabilize carotenoids upon storage at 35 °C, 50 °C, and 65 °C. Carotenoid loss followed first order loss kinetics, and increased with increasing storage temperature. Rapid initial first order loss followed by a second, less rapid first order loss was observed. Storage of the systems above the T_g reduced carotenoid loss in the initial first order loss. The loss of carotenoids in LBL system was more temperature dependent initially but SL system was more temperature dependent in the second first order loss step. LBL system showed slower loss rate of carotenoids in the initial first order loss step and at 65 °C in the second step. Carotenoid retention was significantly higher in LBL system upon storage at 65 °C. *Industrial relevance:* Although layer-by-layer (LBL) technique has been known to produce emulsions with better stability towards environmental stresses, few have reported the application of LBL technique using systems with high total solids. The application of LBL technique on emulsion with high total solids and subsequent spray-drying of the emulsion in this manuscript will provide useful information to the food and pharmaceutical industries. The possibility to spray-dry such systems with high total solids producing high quality powders would be feasible to the industry as it greatly reduces production cost. The present study also reports on the carotenoid loss kinetics of dehydrated concentrated systems with carbohydrate and non-carbohydrate mixtures as wall materials and compares the ability of single layer (SL) and LBL systems in preventing the loss of the encapsulated carotenoids.

1. Introduction

Studies have found that the application of layer-by-layer (LBL) interfacial structures in LBL emulsions can result in emulsions with higher stability towards environmental stresses such as heat treatment, variations in pH, freeze–thaw cycles, lipid oxidation and ionic strength (Aoki, Decker, & McClements, 2005; Gharsallaoui et al., 2010; Güzey & McClements, 2006; Lim, Griffin, & Roos, 2014; Ogawa, Decker, & McClements, 2003a). The higher stability of LBL system is due to the thicker and denser interfacial layer of the particles, higher steric repulsion, as well as lower van der Walls attraction strength (Benjamin, Silcock, Leus, & Everett, 2012; Gu, Decker, & McClements, 2005; Harnsilawat, Pongsawatmanit, & McClements, 2006; Moreau, Kim, Decker, & McClements, 2003). The thicker interfacial layer of LBL systems can increase the stability of the oil particles towards disruptions providing better protection towards the encapsulated materials (Güzey & McClements, 2006; McClements, 1999). LBL emulsion can be produced via electrostatic attraction between a charged primary layer with an oppositely charged secondary layer present in the continuous phase (Lim & Roos, 2015; Moreau et al., 2003). Whey protein isolate (WPI) was used as the primary layer and gum Arabic as the secondary layer in this study. Due to its ability to be positively or negatively charged by changing the pH of the aqueous phase, protein is widely used as the primary layer in LBL systems (Gharsallaoui et al., 2010; Gu, Decker, & McClements, 2004; Klein, Aserin, Ishai, & Garti, 2010). The isoelectric point (pI) of β-lactoglobulin is 5.2 (Bryant & McClements, 1998) while α-lactalbumin is 4.1 (Weinbreck, de Vries, Schrooyen, & de Kruif, 2003). As gum Arabic has a pKa value of approximately 2.2, gum Arabic will be negatively charged above pH 2.2 (Weinbreck, Tromp, & de Kruif, 2004). Generally, the use of proteins as emulsifiers results in small oil droplets in emulsions with poor stability towards environmental stresses. On the other hand, polyelectrolytes produced oil droplets with better stability towards environmental stresses but were incapable of producing small oil droplets unless used in excess quantities (McClements, 2003).

Carotenoids are commonly found in fruits and vegetables with β-carotene, a highly lipophilic carotene having the highest vitamin A activity and rate of conversion to vitamin A among the provitamin A carotenoids (Grune et al., 2010). β-Carotene is also capable of showing antioxidant and anticancer properties, and may prevent heart diseases (Albanes, 1999; Bendich & Olson, 1989; Omenn et al., 1996).

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Degradation of β-carotene occurs mainly through oxidation (Simpson, 1985) and isomerization (Sweeney & Marsh, 1971). On the contrary, lutein, due to the presence of hydroxyl groups in its molecular structure is categorized as xanthophyll. Lutein is commonly found in green vegetables and can minimize light-induced skin damage (Ribaya-Mercado & Blumberg, 2004) as well as prevents cataracts and macular degeneration (Kachik, Bernstein, & Garland, 1997). Chronic and degenerative diseases in humans can be reduced with the consumption of food products consisting of bioactive compounds (van Dokkum, Frølich, Saltmarsh, & Gee, 2008). The low bioavailability of carotenoids as crystals or within protein complexes in fruits and vegetables reduces adsorption in the gastrointestinal tract during digestion (Williams, Boileau, & Erdman, 1998). Nonetheless, the use of carotenoids with oil can improve the bioavailability of carotenoids as oil increases carotenoid adsorption (van Het Hof, West, Weststrate, & Hautvast, 2000). Therefore, the incorporation of carotenoids in the lipid phase of an oil-in-water (O/W) emulsion can enhance its bioavailability. The lipid phase can be emulsified with an emulsifier containing an aqueous phase followed by the addition of glass forming hydrophilic component and dehydration of the emulsion to produce a continuous phase of the dried formulation (Drusch, Serfert, Van Den Heuvel, & Schwarz, 2006; Ramoneda, Ponce-Cevallos, Buera, & Elizalde, 2011; Spada, Noreña, Marczak, & Tessaro, 2012; Mehanna, Alwattar, & Elmaradny, 2015).

Microencapsulation utilizes wall materials to entrap core materials protecting it from environmental stresses to provide longer shelf life and enable controlled release of the core materials (Shahidi & Han, 1993). Microencapsulation is commonly achieved through spray drying in the food industry (Ré, 1998). Spray drying is also straightforward and economical giving high quality powders with a long shelf life. Maltodextrins, trehalose, milk proteins, corn syrup and modified starch are frequently used as wall materials in spray drying (Desobry, Netto, & Labuza, 1997; Drusch et al., 2006; Hogan, McNamee, O'Riordan, & O'Sullivan, 2001; Liang, Huang, Ma, Shoemaker, & Zhong, 2013; Mehanna et al., 2015; Shaw, McClements, & Decker, 2007). Crystallization of the wall materials in powders can lead to degradation of released carotenoids containing a lipid phase as a result of direct exposure to environmental stresses (Buera, Schebor, & Elizalde, 2005). Nevertheless, studies have shown that mixtures of sugars and proteins can delay the crystallization rate of the sugar component (Haque & Roos, 2004; Jouppila & Roos, 1994). Our earlier studies also showed that the application of LBL interfacial structures with trehalose, and the mixture of trehalose and maltrodextrin (DE 10) as wall materials reduced the loss of carotenoids upon storage (Lim & Roos, 2016; Lim et al., 2014). The objectives of the present study were to obtain single layer (SL) and LBL powders by spray drying emulsions with high total solid content as well as to determine the ability of the SL and LBL powders having carbohydrate (trehalose) and non-carbohydrate (WPI) mixture as wall materials to protect carotenoids upon storage in the vicinity of the glass transition temperature (T_g) of the carbohydrate. Data on carotenoid loss kinetics and the ability of SL and LBL systems in preventing the loss of encapsulated carotenoids in spray dried systems stored in closed containers were reported, providing important information to food and pharmaceutical ingredients and formulations. The application of concentrated systems having carbohydrate and non-carbohydrate mixture as wall materials for spray drying will be beneficial for the industries as there are few reports on the use of such a system.

2. Materials and methods

2.1. Materials

Sunflower oil was Musgrave Excellence™ (Spain), whey protein isolate (WPI, Isolac) was from Carbery Food Ingredients (Ballineen, Ireland), gum Arabic (Sigma Aldrich G9752) from Sigma Aldrich (Stenheim, Germany), trehalose (crystalline dehydrate) from Hayashibara Shoji Inc. (Japan), all-trans-β-carotene (crystalline Type I, synthetic, >93% (UV)) from Sigma-Aldrich (U.S.A.), and lutein (Marigold) from Shaanxi Sciphar Biotechnology Co. Ltd. (China). All other chemicals were purchased from Sigma-Aldrich, Inc. (Dublin, Ireland) and they were of analytical grade.

2.2. Emulsion preparation

The emulsions were prepared using methods modified from our earlier study (Lim & Roos, 2015, 2016). The emulsions prior to spray drying were SL and LBL which consisted of 18.2% sunflower oil and carotenoids, 18.18% trehalose, 9.09% WPI, and water (adjusted to pH 3.5 using citric acid) and with and without gum Arabic (0.27%), respectively. WPI was dispersed in deionized water (19.36%, *w*/w, in water) and the dispersion was allowed to hydrate for 2 h to enhance its hydration. The dispersion was adjusted to pH 3.5 using citric acid solution (10% *w*/w). β-Carotene (0.05%, *w*/w, of oil) and lutein (0.05%, *w*/w, of oil) were dissolved in sunflower oil and mixed with a Silverson mixer (Model AXR, Silverson Machines Ltd., Chesham, UK) at 50 °C. A Silverson mixer was used to mix the oil phase (2 parts of sunflower oil) and water phase (1 part of water used initially for the dispersion of WPI) at the minimum speed for 60 s to obtain pre-emulsion. The pre-emulsions were subsequently homogenized at room temperature using a two-stage valve homogenizer (APV-1000, APV Homogenizer Group, Wilmington, MA, USA) for 3 cycles at 240 bar (200 bars for the first stage and 40 bars for the second). A mixture of trehalose and WPI (ratio of 21:8) was used as wall material. Trehalose (46.41%, *w*/w) was dissolved in deionized water and citric acid solution mixture (40.21% water and 59.79% citric acid solution) using a Silverson mixer at 50 ± 1 °C. WPI (30.77%, *w*/w) was dispersed in deionized water, stirred with a rod, and allowed to hydrate for 2 h to ensure complete hydration. The trehalose solution and WPI dispersion were then mixed together using the Silverson mixer and the pH was adjusted to pH 3.5 using citric acid solution. To obtain SL emulsion, the emulsion was mixed with the trehalose and WPI mixture at a ratio of 1:2.14 for 30 min. Gum Arabic (5.66%, *w*/w, in water) was dissolved in deionized water and stirred for 2 h. The gum Arabic solution was adjusted to pH 3.5 with citric acid solution. LBL emulsion was obtained by mixing the gum Arabic solution with the emulsion at a ratio of 1:6 at room temperature for 30 min using a Silverson mixer. The emulsion with gum Arabic as the secondary layer was then mixed with the trehalose and WPI mixture at a ratio of 1:1.83 for 30 min. The SL emulsion was added with a similar amount of water instead of gum Arabic solution to achieve the same final weight as the LBL emulsion.

2.3. Spray drying and sample packaging

The emulsions were dehydrated using a single stage Niro 25 spray dryer (GEA Niro Production Minor, Soborg, Denmark) with inlet and outlet temperatures set at 185 °C and 85 °C, respectively equipped with a rotating disk atomizer at 24,000 rpm. The powder solids consisted of 39.79% oil with carotenoids, 39.75% trehalose and 19.87% WPI as their main components and they were rapidly cooled to room temperature, sealed in plastic bags, and stored at room temperature to prevent water uptake and physico-chemical changes prior to analysis. The water contents after spray drying were less than 3% (Table 1) and water activities were 0.14 ± 0.01a_w for SL and 0.14 ± 0.01a_w for LBL powders, respectively. The powders (2 g) were then transferred into 10 mL clear glass vials (Schott, Müllheim, Germany). The samples in vials were hermetically sealed with a vacuum using a freeze-drier (Lyovac GT 2, Steris®, Hürth, Germany) and packaged in plastic pouches (PA/PE 90, Fispar, Leamore Warehouse, Dublin, Ireland) using a vacuum sealing system (Polar 80, Henkelman Vacuum Systems, Hentogenbosch, Netherlands) in duplicates. The vacuum in the packages represents isolation of the systems from surrounding atmosphere and served as an indicator for leakage