Food Chemistry 202 (2016) 324–333

Contents lists available at ScienceDirect

Food Chemistry

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

Elaboration of microparticles of carotenoids from natural and synthetic sources for applications in food

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ARTICLE INFO

Article history:
Received 26 June 2015
Received in revised form 15 December 2015
Accepted 29 January 2016
Available online 4 February 2016

Chemical compounds studied in this article:
β-Carotene (PubChem CID: 5280489)
Chitosan (PubChem CID: 21896651)
Carboxymethylcellulose (PubChem CID: 6328154)
Sodium tripolyphosphate (PubChem CID: 24455)

Keywords:
Microencapsulation
Release profile
Chitosan
Carboxymethylcellulose
Sodium tripolyphosphate

ABSTRACT

Carotenoids are susceptible to isomerization and oxidation upon exposure to oxygen, light and heat, which can result in loss of color, antioxidant activity, and vitamin activity. Microencapsulation helps retain carotenoid stability and promotes their release under specific conditions. Thus, the aim of the study was to encapsulate palm oil and β-carotene with chitosan/sodium tripolyphosphate or chitosan/carboxymethyl cellulose and to assess the performance of these microparticles in food systems by analyzing their release profile under simulated gastric and intestinal conditions. Encapsulation efficiency was greater than 95%, and the yield of microparticles coated with chitosan/sodium tripolyphosphate was approximately 55%, while that of microparticles coated with chitosan/carboxymethylcellulose was 87%. Particles encapsulated with chitosan/carboxymethylcellulose exhibited ideal release behavior in water and gastric fluid, but showed low release in the intestinal fluid. However, when applied to food systems these particles showed enhanced carotenoid release but showed low release of carotenoids upon storage.

1. Introduction

Carotenoids are one of the most widespread pigment groups in nature and are responsible for the yellow, orange or red color of fruits, leaves, and flowers (Rodriguez-Amaya, 1997). Synthetic β-carotene, with a molecular structure identical to natural β-carotene, was first synthesized in the 1950s using β-ionone, derived from acetone or butadiene, as the precursor (Britton, Liaaen-Jensen, & Pfander, 1996).

Natural β-carotene can also be obtained from filamentous fungi, yeasts, micro-algae, and bacteria using biotechnological processes, or by extraction from plant sources. Using natural sources of carotenoids such as palm oil is distinctly advantageous as it has not only β-carotene but also other carotenoids, albeit in lower concentrations, which contribute to the health benefits associated with carotenoids (Dufossé et al., 2005).

Some carotenoids are precursors of vitamin A, if they have a chemical structure with at least one β-ionone ring linked to a chain of 11 carbons, as in the case of β-carotene which has greater pro-vitamin A potential compared to either α-carotene or β-cryptoxanthin (Rodriguez-Amaya, 1997).

In addition to such pro-vitamin activity, some carotenoids also show high antioxidant potential because the presence of conjugated double bonds enables these molecules to accept electrons from reactive species, thus neutralizing free radicals. However, the presence of conjugated double bonds also makes these compounds susceptible to isomerization and oxidation in the presence of oxygen, light, and heat, which then lead to loss in color, antioxidant property, and vitamin activity (Gonnet, Lethuaut, & Boury, 2010; Rodriguez-Amaya, 1997). Microencapsulation is a process that increases the stability of these compounds by physically isolating them from environmental conditions using wall materials (Matsuno & Adachi, 1993).

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http://dx.doi.org/10.1016/j.foodchem.2016.01.140
0308-8146/

Wall materials are usually biodegradable polymers that may be natural, modified natural or synthetic. The natural polymers are less costly compared to other polymers when used for microencapsulation (Liu, Jiao, Wang, Zhou, & Zhang, 2008). In addition to protecting the core material, microencapsulation enables the controlled release of the active substance(s) in specific parts of the body (Jingou et al., 2011). Though a single polymer can be used for microencapsulation, in most cases single encapsulating agent does not possess all the properties of an ideal wall material, and thus a combination of polymers is recommended (Matsuno & Adachi, 1993).

Encapsulation through the interaction between the different wall materials can be achieved by different methods, such as complex coacervation and ionic gelation. Complex coacervation requires an interaction between oppositely charged polymers, and this method has been used for the encapsulation of vitamin E (Alencastre et al. (2006)), essential oils (Jun-Xia, Hai-Yan, & Jian, 2011), phenolic compounds (Belščak-Cvitanović et al., 2011; Zou et al., 2012), iron (Araújo, 2011), etc. Ionic gelation uses a multivalent anionic salt as the cross linker that promotes complexation with a positively charged polymer, and this method has been used for the encapsulation of ascorbyl palmitate (Yoksan, Jirawutthiwongchai, & Arpo, 2010), oregano essential oil (Hosseini, Zandi, Rezaei, & Farahmandghavi, 2013), vegetable oils (Barreto, 2008), and lutein (Arunkumar, Prashanth, & Baskaran, 2013).

Food applications of encapsulated materials include providing supplements, changes in solubility, taste, texture and color, and acting as sources of antioxidants and antimicrobials. Several processes of nano- and micro-encapsulation have been patented by companies for use in foods or as dietary supplements (Foladori & Invernizzi, 2007); however, only a few studies relating to the application of encapsulated compounds in foods have been reported, e.g., application of microcapsules containing lycopene in cakes (Rocha, Fávaro-Trindade, & Grosso, 2012), linseed oil in bread (Gallardo et al., 2013), curcumin in ice cream (Sousdaleff et al., 2013) and yogurt (Mangolim et al., 2014), and red bell pepper pigments in yogurt (Gomes, Petito, Costa, Falcão, & Araújo, 2014).

Among the vegetable oils consumed in the world, palm oil has the highest concentration of carotenoids (Zambiazi, 1997). Thus encapsulating this oil will prevent carotenoid degradation during processing and storage and will facilitate the incorporation and consumption of carotenoids from food products.

Given the above, the aim of the study was to prepare microparticles containing synthetic β-carotene and palm oil with chitosan/carboxymethylcellulose as wall material by the complex coacervation method and with chitosan/sodium tripolyphosphate by the ionic gelation method, and to characterize these microparticles with respect to encapsulation efficiency, morphology, and thermal behavior. Furthermore, the aim was also to assess the release profile of the microencapsulated compounds under simulated human gastric and intestinal conditions, apply these microparticles in two food systems, namely bread and yogurt, to assess their release profile and stability during storage and their release profile upon exposure to simulated gastric and intestinal conditions.

2. Material and methods

2.1. Materials

The core materials used were trans β-carotene in powder form with 97% purity (Sigma Aldrich Brazil Ltd, São Paulo, SP, Brazil), soybean oil (Soya, Bunge Alimentos S.A., São Paulo, SP, Brazil), and pure palm oil (Hemmer, Cia Hemmer Indústria e Comércio, Blumenau, SC, Brazil).

The encapsulating agents used were medium molecular weight chitosan (190–310 KDa, Sigma Aldrich Brazil Ltd, São Paulo, SP, Brazil), sodium carboxymethylcellulose (Synth, LabSynth, Diadema, SP, Brazil), and sodium tripolyphosphate (Synth, LabSynth, Diadema, SP, Brazil).

2.2. Methods

2.2.1. Content of carotenoids from core material

Quantification of carotenoid in palm oil (natural source) was performed by dissolving 0.1 g of sample in 25 mL of hexane, and it was subsequently read spectrophotometrically (Jenway 6705 UV/Vis) at 450 nm. The quantitation was based on a calibration curve obtained using standard β-carotene ($y = 0.2191$, $x = -0.0045$, $R^2 = 0.9996$) and results were expressed as μg of β-carotene g^{-1} of sample (Rodriguez-Amaya, 1997).

Pure β-carotene (synthetic source) was added to soybean oil, and the carotenoid content of this oil was also evaluated in a similar way.

2.2.2. Preparation of microparticles

Encapsulation was performed according to the method described by Zou et al. (2012), with a few modifications.

2.2.2.1. Wall materials. Chitosan (5 mg mL^{-1}) was dissolved in 0.1 M hydrochloric acid and neutralized with 0.2 M sodium hydroxide solution till pH 5.6. The final volume of 1000 mL was made up with distilled water.

Carboxymethylcellulose (CMC, 5 mg mL^{-1}) and sodium tripolyphosphate (TPP, 5 mg mL^{-1}) were dissolved in distilled water.

2.2.2.2. Core materials. Palm oil containing 1302.38 μg β-carotene g^{-1}, and soybean oil containing 1235.04 μg β-carotene g^{-1} were used as core materials.

2.2.2.3. Encapsulation. Complex coacervation is characterized by the occurrence of phase separation due to electrostatic attraction between two oppositely charged macromolecules such as chitosan (positive charge) and carboxymethylcellulose (negative charge). For encapsulation 100 mg of the core material (palm oil or soybean oil with β-carotene) was added to 50 mL of chitosan solution; the solution was homogenized using an ULTRA-TURRAX device (T18 Basic IKA) at 13500 rpm for 5 min; 50 mL of sodium carboxymethylcellulose dispersion was added, and stirred further for 3 min. The pH of the complex coacervation was 6.2. The dispersions were centrifuged (Eppendorf 5430 R) at 7000*g* for 10 min. The supernatant was used for encapsulation efficiency analysis and the precipitate containing palm oil microparticles or β-carotene microparticles was lyophilized and stored at −75 °C.

The process of ionic gelation uses a multivalent ionic salt as the cross linker that promotes complexation with a positively charged polymer. In this study, chitosan (positively charged) and sodium tripolyphosphate (negatively charged) were used. The procedure for encapsulation by ionic gelation was identical to that of coacervation and used 50 mL of sodium tripolyphosphate (5 mg mL^{-1}) instead of sodium carboxymethylcellulose. The pH of the ionic gelation was 7.7.

2.2.3. Encapsulation efficiency (EE)

The supernatant (mentioned in Section 2.2.2) was made to a volume of 100 mL, and the precipitate was washed with distilled water. The supernatant (10 mL) was mixed with 10 mL of hexane and homogenized on ULTRA-TURRAX (T18 Basic IKA) at 13000 rpm for 20 s. The phases were separated, and the nonpolar phase was collected, and spectrophotometrically read (Jenway