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Microencapsulation of palm oil by complex coacervation for application in food systems

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ABSTRACT

This study aimed at microencapsulating palm oil, containing high carotenoid content, with chitosan/xanthan and chitosan/pectin, using the complex coacervation method, followed by atomization and lyophilization. The DSC technique was used to confirm the encapsulation. The atomized microparticles had spherical shape and irregular size, and the lyophilized microparticles had irregular shape and size. Lyophilization resulted in lower carotenoids losses, and higher yield and encapsulation efficiency. In addition, the release profile in both water and gastrointestinal fluid was satisfactory. Prior to their application in food, a greater percentage of carotenoids was released in the fluid that simulates gastrointestinal conditions; however, the compounds were degraded after their release. In this case, the chitosan/pectin microparticles showed the best release profile. After processing, the release was lower and the released compounds were not degraded. Thus, the chitosan/xanthan microparticles showed the best potential for practical application, particularly, in yogurt preparation.

1. Introduction

The palm fruit (*Elaeis guineensis*), is obtained from native palm trees in Africa and also cultivated in Central America, South America, and Asia known as "dendezeiro,". Two types of oils can be extracted from this fruit. Palm kernel oil is derived from the almond of the fruit and palm oil is derived from its mesocarp (De Oliveira, Ribeiro, & Kieckbusch, 2015).

Palm oil has high content of bioactive compounds, particularly carotenoids, which contribute to its stability and nutritional value (Mustapa, Manan, Mohd Azizi, Setianto, & Mohd Omar, 2011). Carotenoids are a group of pigments that exhibit varying colors ranging from yellow to red. Its chemical structure comprises of eight isoprene units, thus forming a system of conjugated double bonds. This system enables them to receive electrons from reactive species, neutralizing free radicals. Therefore, carotenoids have antioxidant properties (Rodriguez-Amaya, 1997).

Furthermore, some carotenoids such as β-carotene, α-carotene, and β-cryptoxanthin are precursors of vitamin A. These carotenoids have at least one β-ionone ring that is linked to a chain of 11 carbons, making them susceptible to isomerization and oxidation by O_2, light, and high temperatures (Rodriguez-Amaya, 1997). This possibly results in loss of color and reduced antioxidant effect and vitamin A activity (Gonnet, Lethuaut, & Boury, 2010).

The microencapsulation technique provides stability to these compounds and releases them in a controlled manner under specific conditions. The encapsulating materials used in this technique may also protect sensitive compounds by physically isolating the compounds (Matsuno & Adachi, 1993).

In general, proteins, lipids, and carbohydrates can be used as encapsulating materials. However, the choice of material depends on its non-reactivity with the compound to be encapsulated, of encapsulation method, as well as an ideal release mechanism (Shahidi & Han, 1993).

Depending on the encapsulation method used, one or more encapsulating materials can be used. In general, two or more encapsulating materials are used because a single material does not possess all the ideal properties for the process (Matsuno & Adachi, 1993).

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Complex coacervation is a method that uses a combination of encapsulating agents. In this method, electrostatic attraction is caused between a minimum of two oppositely charged macromolecules. In addition, other weak interactions such as hydrogen bonding and hydrophobic interactions possibly contribute to the complex formation (Ach et al., 2015). This method is widely used for encapsulating lipophilic compounds, such as essential oil (Peng et al., 2014), vegetable oil (Yang, Gao, Hu, Li, & Sun, 2015), fish oil (Wang, Adhikari, & Barrow, 2014), and palm oil (Marfil, Vasconcelos, Pontieri, & Telis, 2015).

In general, the wall materials used are biodegradable polymers that may be natural, natural modified, or synthetic. Compared to other polymers used for microencapsulation, natural polymers are less expensive (Liu, Jiao, Wang, Zhou, & Zhang, 2008). Chitosan, a polysaccharide, is widely used as the wall material. It is also known as a polycationic polymer due to the presence of amino groups in its structure, which interacts with negatively charged groups such as carboxylic groups present in the chemical structures of xanthan gum and pectin molecules (Chellat et al., 2000).

The interaction between these groups can form polyelectrolyte complexes that are capable of entrapping a range of compounds. The stability of these complexes is possibly affected by pH; therefore, the release of the encapsulated compounds can be controlled by pH, similar to those occurring in the gastrointestinal tract of the human body. Other factors such as temperature and enzymatic action may also weaken the chemical bonds (Nordby, Kjoniksen, Nyström, & Roots, 2003; Mendes, De Oliveira, De Castro, & Giordano, 2011).

Microparticles can be added to different food products for masking flavor and aroma as well as changing texture and color. In addition, they can be added as antimicrobials and antioxidants. However, only a few studies have addressed their application in food industry. Thus far, studies have been conducted on flaxseed oil microencapsulation in bread (Gallardo et al., 2013) and application of lycopene microcapsules in cakes (Rocha, Fávaro-Trindade, & Grosso, 2012).

Therefore, this study aimed at microencapsulating palm oil, containing high carotenoid content, using the complex coacervation method and chitosan/xanthan gum and chitosan/pectin as wall materials. Subsequently, the encapsulation efficiency and internalizing efficiency of the carotenoids, the morphology, thermal behavior, and release profile of carotenoids in water and in a fluid that simulates gastrointestinal environment was evaluated. Furthermore, the application of microparticles in bread and yogurt preparation was studied and the release profile of encapsulated compounds was evaluated when the fortified foods were exposed to fluids that simulate gastrointestinal conditions.

2. Material and methods

2.1. Materials

Pure palm oil (Hemmer, Cia Hemmer Indústria e Comércio, Blumenau, SC, Brazil) was used as the encapsulated material. Medium molecular weight chitosan (C) 190–310 KDa, with deacetylation degree of 75–85% (Sigma, Sigma Aldrich Brazil Ltda, São Paulo, SP, Brazil), xanthan gum (X) (Sigma, Sigma Aldrich Brazil Ltda, São Paulo, SP, Brazil), and pectin (P) (CP Kelco, CP Kelco Brazil, Limeira, SP, Brazil) were used as the encapsulating agents.

2.2. Methods

2.2.1. Carotenoid content from palm oil

Palm oil (1 mg) was dissolved in 25 mL hexane and evaluated using a spectrophotometer (Jenway 6705 UV/Vis.) at 450 nm. It was quantified based on a calibration curve of standard β-carotene ($y = 0.2191 \times -0.0045$, $R^2 = 0.9996$). The results are expressed in μg of β-carotene.g^{-1} of sample (Rodriguez-Amaya, 1997).

2.2.2. Microparticle elaboration

The encapsulation was performed according to the method described by Rutz, Borges, Zambiazi, Da Rosa, and Da Silva (2016). Palm oil/wall material ratio was determined from preliminary tests and previous data. The same procedure was performed for the wall materials.

Chitosan (5 mg.mL^{-1}) was dissolved in hydrochloric acid solution (0.1 M). After adjusting the pH to 5.6 with 0.2 M sodium hydroxide, the desired volume was achieved by dilution with distilled water. Oil palm (100 mg; 1327.08 μg of β-carotene.g^{-1}) was added to 50 mL chitosan solution. The homogenization was performed using an Ultra Turrax device (T18 Basic IKA) at 13,500 rpm for 5 min.

Subsequently, the encapsulation by the complex coacervation method was performed by adding a dispersion of 50 mL xanthan gum (5 mg.mL^{-1}) or pectin (5 mg.mL^{-1}). After the addition of the second wall material, the solution was continuously stirred for another 3 min.

One part of the sample was frozen at −75 °C and lyophilized (lyophilizer Terroni-Enterprise II), and the other part was subjected to atomization in a pneumatic type dual fluid atomizer (Mini Spray Dryer LM MSDI 1.0 LABMAQ, Brazil) with a 1.0-mm diameter feeding nozzle. The feed of the dryer was carried out using a peristaltic pump with a flow rate of 0.60 L.h^{-1}. The temperature of the drying air was 140 °C, the air pressure was 3.5 kg f.cm^{-2}, and the atomizing airflow rate was 40.5 L.h^{-1}.

2.2.3. Internalizing efficiency

Internalizing efficiency (IE) is defined as the percentage of carotenoids present in the microparticles that were actually encapsulated (Rutz et al., 2013). Distilled water (5 mL) and 5 mL hexane were simultaneously added to 30 mg of the sample. The total carotenoid content of the microparticles was quantified by homogenizing the sample in Ultra Turrax (T18 Basic IKA) at 13,000 rpm for 1 min, followed by centrifugation (Fanem BL-206) at 1057*g* for 3 min.

The carotenoid content on the surface was quantified by homogenizing 30 mg sample with 5 mL hexane in a vortex mixer (Biomixer QL-901) for 1 min, followed by centrifugation (206 Fanem-BL) at 1057*g* for 3 min. After the phase separation, the non-polar phase was collected and evaluated using a spectrophotometer (Jenway 6705 UV/Vis.) at 450 nm. The quantification process was performed as per the protocol described previously (Section 2.2.1).

The IE was calculated using Eq. (1), where *Ct* represents the total carotenoids present in the microparticles and *Cs* is the carotenoid content present on the surface of the microparticles.

$$\% IE = \frac{Ct - Cs}{Ct} \times 100 \quad (1)$$

2.2.4. Encapsulation efficiency

Encapsulation efficiency (EE) is defined as the percentage of carotenoids encapsulated in relation to the carotenoid content initially added (Somchue, Sermsri, Shiowatana, & Siripinyanond, 2009). It was calculated using Eq. (2), where *Ct* is the total carotenoids of the microparticles, *Cs* is the carotenoid content on the surface of the microparticles (both evaluated as described in Section 2.2.3), and Ca is the initial content of carotenoids.