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Microencapsulation by spray drying of a lycopene-rich tomato concentrate: Characterization and stability

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ABSTRACT

The objective of this study was to evaluate the effect of different encapsulating agents (maltodextrin, whey protein isolate and the modified starch Capsul®) on the physicochemical properties and lycopene stability of a tomato concentrate microencapsulated by spray drying. Different formulations were produced according to an experimental mixture simplex-lattice design, where the independent variables were the concentrations of each encapsulating agent. Physical properties (moisture content, solubility and hygroscopicity), lycopene concentration and the antioxidant capacity of particles immediately after drying, as well as lycopene stability during storage, were analyzed as responses. Particles presented initial lycopene content between 333 and 494 μg/g. The powders produced with maltodextrin and modified starch presented the highest concentrations of this carotenoid and greater antioxidant capacity. These two responses showed high degree of correlation with each other. Maltodextrin and modified starch also led to lower lycopene degradation rates during storage and, therefore, these encapsulating agents were considered the most suitable for the tomato concentrate encapsulation.

1. Introduction

In recent years several studies have indicated that the regular consumption of lycopene-rich foods is associated with the reduction and prevention of chronic cardiovascular diseases and some types of cancer (Cicero, Fogacci, & Colletti, 2017; Kulczyński, Gramza-Michałowska, Kobus-Cisowska, & Kmiecik, 2017; Singh, Sharma, Ghosh, Park, & Jeong, 2017), leading to a growing interest in this carotenoid. Among the vegetables that contain lycopene in their composition, tomato is considered one of the major sources, with values varying between 20 and 100 μg/g (Demiray, Tulek, & Yilmaz, 2013; Thompson et al., 2000).

Lycopene ($C_{40}H_{56}$) is an acyclic carotenoid with 13 double bonds of which 11 are conjugated. This configuration is responsible for scavenging and stabilizing reactive oxygen species (ROS), in particular singlet oxygen ($^1O_2$) (Rao, Ray, & Rao, 2006). However, when free from its food matrix, lycopene is easily subjected to degradation during storage, mainly in the presence of oxygen (Goula & Adamopoulos, 2005; Shu, Yu, Zhao, & Liu, 2006). Thus, the industry is constantly looking for alternatives to protect this carotenoid during storage and/or processing.

Microencapsulation is a technique generally used to protect bioactive compounds from adverse environmental external conditions. It can improve their stability, increasing shelf life and allowing their release in a given environment under controlled conditions. Several methods, including complex coacervation (Silva, Favaro-Trindade, Rocha, & Thomazini, 2012), alginate gelation (Marquis, Alix, Capron, Cuenot, & Zykwinska, 2016), electrodynamic atomization (EHDA) (Mehta et al. 2017) and electrospraying (Rasekh et al., 2017; Zhang et al., 2017), have been reported aiming at encapsulation for food and pharmaceutical applications. Spray drying is the most common technique used for food encapsulation, due not only to its efficiency, but also to the possibility of continuous production and easy scale-up and industrialization (Peanparkdee, Iwamoto, & Yamauchi, 2016; Gharsallaoui, Roudart, Chambin, Voilley, & Saurel, 2007).

Particles produced by spray drying are matrix-based, i.e, the core is entrapped within a continuous network of a polymeric matrix, and their main advantage is the easy reconstitution (Matalanis, Jones, & McClements), which is important for applications in liquid and pasty foods, or in instantaneous powders. Spray drying has been largely used to encapsulate carotenoids such as norbixin (Tupuna et al., 2018) and astaxanthin (Ahmed, Li, Fanning, Netzel, & Schenk, 2015; Montero,

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Calvo, Gómez-Guillén, & Gómez-Estaca, 2016), phenolic compounds from bayberry (Cheng et al., 2017) and propolis (Busch et al., 2017) and vitamins such as B12 (Carlan, Estevinho, & Rocha, 2017), α-tocopherol and ascorbic acid (Nesterenko, Alric, Silvestre, & Durrieu, 2014). Rocha, Favaro-Trindade, and Grosso (2012) reported that microencapsulation by spray drying of lycopene resulted in a better storage stability when compared to the free lycopene. The authors applied the particles in a cake formulation and observed that they were able to release the lycopene during preparation of the studied system and to color it homogenously. Choudhari, Bajaj, Singhal, and Karwe (2012) evaluated the stability of lycopene during extrusion cooking of rice flour and observed that, at different extrusion conditions, the lycopene microencapsulated by spray drying showed better color retention in the extrudates than the free lycopene.

Different encapsulating materials have been used for food products. Maltodextrin, which is obtained from partial hydrolysis of starch, is the most common encapsulating agent, with advantages such as low cost and good protection against oxidation (Goula & Adamopoulos, 2012), but with no emulsifying ability. Whey protein isolate is also being used due to its countless beneficial properties such as amphiphilic characteristics, capacity of self-association and interaction with many substances, high molecular weight and flexible molecular chain, in addition to other interesting properties such as solubility, viscosity and emulsifying power (Madene, Jacquot, Scher, & Desobry, 2006). The octenylsuccinate-modified starch, known as OSA starch, is a starch added of the lipophilic component octenylsuccinate anhydride, which provides amphiphilic properties and increase of the starch emulsifying capacity. So, the combination of maltodextrin with other amphiphilic materials can be a promising alternative to encapsulate lipophilic compounds, such as lycopene, improving its protection and retention in the particles. Although some works have reported the use of maltodextrin (Goula & Adamopoulos, 2012) and OSA starch (Choudhari et al., 2012; Rocha et al., 2012) in the microencapsulation of lycopene by spray drying, there is no report on the effect of the mixture of these two materials, as well as on the use of whey protein isolate as encapsulating agents.

In this sense, the objective of this study was to evaluate the effect of three different encapsulating materials (maltodextrin, whey protein isolate and the modified starch Capsul®) on the physicochemical properties and lycopene stability of a tomato concentrate microencapsulated by spray drying.

2. Material and methods

2.1. Material

Italian tomatoes purchased in the local market (Rio de Janeiro, Brazil) were used as raw material. Tomatoes were chosen for this work because they are one of the main vegetable sources of lycopene.

Maltodextrin MOR-REX 2105® (Ingredion, Mogi-Guaçu, Brazil), whey protein isolate (Alibra, Campinas, Brazil) and the modified starch Capsul® (Ingredion, Mogi-Guaçu, Brazil) were used as encapsulating agents.

2.2. Preparation of tomato concentrate

The concentrate was obtained from the integration of reverse osmosis and diafiltration membrane processes. Initially, the tomato pulp was concentrated by reverse osmosis in a semi-pilot plate-and-frame system with flat, composite film membranes (HR 98 PP – DDS/Danish Separation System, Denmark), consisting of a polyamide selective layer on a polysulfone support, presenting 98% of nominal rejection to a 0.25 g/100 g NaCl solution, with permeation area of 0.396 m^2. The process was conducted in batch mode at 30 °C, with transmembrane pressure of 6000 kPa (Gomes, Costa, Campos, Couri, & Cabral, 2011). Next, sugars were removed from the concentrated pulp by diafiltration, using a tubular system with microfiltration membranes with porosity of 0.1 µm. The process was carried out up to the removal of soluble solids to values close to 2.0 °Brix, at 25 °C, with transmembrane pressure of 100 kPa. At the end of the process, a lycopene-rich concentrate was obtained, with reduced sugar content.

The concentrate was characterized for pH, soluble solid content and total titratable acidity, according to A.O.A.C. (2006). Lycopene content was determined according to the methodology described by Sadler, Davis, and Dezman (1990) and modified by Perkins-Veazie, Collins, Pair, and Roberts (2001). Antioxidant capacity was evaluated using the $ABTS^{+}$ radical scavenging spectrophotometric method described by Re et al. (1999).

2.3. Microencapsulation by spray drying

The encapsulating agents were added to the tomato concentrate in a ratio of concentrate's solids to encapsulating agent of 1:3, defined in preliminary tests as the amount needed for obtaining a reasonable amount of powder, since the initial solid content of the concentrate was only 2.8 g/100 g and the powder losses inside the drying chamber were around 50–60%. The mixture was homogenized using an Ultra-Turrax Ika T25 digital homogenizer (IKA, Staufen, Germany) at 10 000 rpm, for 3 min.

Microencapsulation was carried out in a Büchi 190 laboratorial spray dryer (Flawil, Switzerland), at the following operational conditions: compressed air pressure of 350 kPa, drying air flow rate of 700 L/h, feed flow rate of 34 mL/min and inlet and outlet air temperatures of 160 ± 2 °C and 80 ± 2 °C, respectively.

An experimental mixture simplex-centroid design (Table 1) was used to investigate the effect of encapsulating agents concentration on the responses: moisture content, solubility, hygroscopicity, lycopene content, lycopene degradation constant (k) and antioxidant capacity.

Results were adjusted to the following polynomial model:

$$y = \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{23} x_2 x_2 + \beta_{123} x_1 x_2 x_3 \quad (1)$$

Where β_n are the regression coefficients, y are the evaluated responses (moisture content, solubility, hygroscopicity, lycopene content, degradation constant (k) and antioxidant capacity) and x_1, x_2 and x_3 are the concentrations of maltodextrin, whey protein isolate and modified starch, respectively.

2.4. Physical properties

Moisture content was determined in a vacuum oven at 60 °C by gravimetric analysis until constant weight (A.O.A.C., 2006).

Powder solubility was determined by the method proposed by Cano-Chauca, Stringheta, Ramos, and Cal-Vidal (2005), which consists in the addition of 0.2 g of sample into a vessel containing 20 ml of distilled water, with high-speed magnetic stirring for 10 min, followed by centrifugation at 3000 × *g*, for 5 min. A 5 ml aliquot of the supernatant was

Table 1
Experimental design for the microencapsulation of tomato extract by spray drying.

Test	MD (g/100 g)	WPI (g/100 g)	MS (g/100 g)
1	100	0	0
2	0	100	0
3	0	0	100
4	50	50	0
5	0	50	50
6	50	0	50
7	33	33	33
8	33	33	33
9	33	33	33

MD = Maltodextrin; WPI = Whey Protein Isolate; MS = Modified Starch.