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Effect of wall materials on the spray drying efficiency, powder properties and stability of bioactive compounds in tamarillo juice microencapsulation☆

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ABSTRACT

Hydro- and lipo-soluble bioactive compounds, such as anthocyanins and carotenoids, in tamarillo juice were microencapsulated using different wall materials, such as maltodextrin (MD), n-octenyl succinic anhydride modified starch, from waxy maize for high load encapsulation (OSA 1), low viscosity gum Arabic alternative (OSA 2), resistant maltodextrin (RMD) and gum Arabic (GA). The wall materials were characterized according to their physicochemical and functional properties, molecular weight distribution and encapsulation efficiency using X-ray diffractometry. The tamarillo powders obtained after spray drying were evaluated for their physicochemical and thermal properties, phenolic content, flavonoid content, antioxidant capacity and storage stability. Although there were significant differences in terms of the encapsulation efficiencies of the wall materials, yield, physical properties and storage stability of the spray dried powders, all of the wall materials successfully encapsulated the hydro- and lipo-soluble bioactive compounds. The viscosity, amorphous region and molecular weight of the wall material had positive influences on the encapsulation efficiency, powder properties and storage stability of the encapsulated tamarillo juice. The storage stability of the powders depended on their water activity, hygroscopicity and glass transition temperature (Tg). The tamarillo powders showed greater anthocyanin and carotenoid degradation in the presence of light at 25 °C compared to the powders stored in the dark at 4 °C. GA and OSA 1 resulted in the highest encapsulation efficiency for both the hydro- and lipo-soluble bioactive compounds, while OSA 1 and MSB showed the greatest storage stability. Reductions in the antioxidant activity, phenolic content and flavonoid content during storage will contribute to the degradation of anthocyanins and carotenoids.

1. Introduction

Tamarillo is a fruit well known as a good source of hydro- and lipo-soluble bioactive compounds, such as anthocyanins and carotenoids, that act as natural colorants and natural antioxidants. Tamarillo is reported to contain three types of anthocyanins and seventeen types of carotenoids [1]. The levels of anthocyanins and carotenoids that were found in tamarillo fruits were 8.5 and 4.4 mg/100 g of fresh weight, respectively. The major anthocyanin is delphinidin 3-rutinoside, while the major carotenoid in tamarillo is β-cryptoxanthin [2]. These bioactive compounds degrade easily in the presence of oxygen, moisture and heat; however, they can be retained in foods more effectively when encapsulated. Converting tamarillo juice into powder using an encapsulation technique is common practice for the production and stabilization of natural colorants and other bioactive compounds. Spray drying has a low heat stress; therefore, it is a good technique to retain heat sensitive bioactive compounds such as vitamins, antioxidants and natural pigments. Although spray drying is a good method for drying, encapsulation has the disadvantage of the limited number of natural biopolymers available as wall materials [3].

The selection of a wall material for encapsulation depends on the solubility of the bioactive compound of interest, which is either hydro- or lipo-soluble. A commonly used wall material for liposoluble bioactive compounds is gum Arabic, and for hydrosoluble compounds, it is maltodextrin. Wall materials such as hydrolysed starch, OSA starch and soluble starch are also favorable for spray drying because of their high solubility in water, low viscosity, bland taste, colorless solutions and low cost [3]. For hydrophobic bioactive compounds, a hydrophobic material must be used to encapsulate, while a hydrophilic material is

☆ *Abbreviations:* Maltodextrin DE10, n-octenyl succinic anhydride modified starch from waxy maize for high load encapsulation, n-octenyl succinic anhydride modified starch from waxy maize for low viscosity gum Arabic alternative, resistant maltodextrin and gum Arabic were identified as MD, OSA 1, OSA 2, RMD and GA, respectively.

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typically used as a wall material for the microencapsulation of hydrophilic bioactive compounds [4]. The spray drying of hydrosoluble bioactive compounds, such as anthocyanins from black carrots [5], *Garcinia indica* Choisy [6], purple sweet potatoes [7], and acai [8], has been investigated previously using maltodextrins with different dextrose equivalent (DE) values, gum Arabic or modified starch. Meanwhile, the wall materials used for spray drying liposoluble bioactive compounds, such as carotenoids from watermelon [9], gac [10], sweet potato [11] and cantaloupe [12], were maltodextrins with different DE values, gum Arabic and waxy starch. In the case of tamarillo, the type of wall material required for encapsulation must encapsulate both hydro- and lipo-soluble bioactive compounds.

To the best of our knowledge, there is no available information on an appropriate wall material for the encapsulation of tamarillo with both hydro- and lipo-soluble bioactive compounds. Since the coating behaviour of each wall material is different, the suitability of a wall material for encapsulation needs to be evaluated because of the lack of knowledge about the compatibility of the encapsulate and coating properties. Thus, this study attempted to characterize the wall materials for tamarillo microencapsulation and to evaluate the spray drying efficiency, powder properties and stability of hydro- and lipo-soluble bioactive compounds in tamarillo powders. This study will provide knowledge on the selection of wall materials to produce fruit powder containing both hydro- and lipo-soluble bioactive compounds and other related research.

2. Materials and methods

2.1. Materials

N-octenyl succinic anhydride modified starch from waxy maize for high load encapsulation (OSA 1) and n-octenyl succinic anhydride modified starch from waxy maize for low viscosity gum Arabic alternative (OSA 2) were provided by Ingredion Incorporated, USA. Fibersol-2 or resistant maltodextrin (RMD) and gum Arabic (GA) were purchased from Archer Daniels Midland Company, USA and R&M Chemicals (Essex, UK), respectively. Maltodextrins DE10 (MD) was purchased from San Soon Seng Food Industries Sdn. Bhd., Malaysia. GA and MD were used as controls in this study. Standards used in this experiment were purchased from Sigma Chemical Co. (St. Louis, MO, USA). All other chemicals used in the experiments were of analytical grade and purchased from Mercks (Darmstadt, Germany).

2.2. Physicochemical and functional characterization of wall materials

The moisture content and dry matter were determined using an oven drying method based on the AOAC Method 934.01 [13] to a constant weight. The solubility of the wall materials was determined using a method from Sciarini et al. [14]. The supernatant was homogenized and centrifuged before drying to a constant weight. The pH of 20% of the wall materials in distilled water was determined using a pH meter (Mettler-Toledo SevenEasy pH, Switzerland). The viscosity of 20% of the wall materials in distilled water was determined using a rapid viscometer (Brookfield DV II, USA). The water holding capacity (WHC) and oil holding capacity (OHC) of the wall materials were determined using a method from Robertson et al. [15]. The sample emulsion was stirred and left at room temperature for 1 h before centrifugation and the residue obtained was weighed. The emulsifying activity (EA) and emulsion stability (ES) of the wall materials were evaluated using a method from Chau et al. [16]. The sample emulsion was homogenized and centrifuged before heated at 80 °C for 30 min in an oven.

2.3. X-ray diffraction spectroscopy

The crystallinity of powder was determined using a method described by Cano-Chauca et al. [17]. X-ray diffractograms of the wall materials were obtained using an X-ray diffractometer (Shimadzu 6000 V2.6, Japan). Each sample was tightly packed into the sample holders, and the diffraction data were collected over an angular range from 4 to 30° (2θ). The X-ray patterns were visually compared with the peak characteristics of a theoretical diffractogram.

2.4. Specific refractive index increment (dn/dc) measurements

Dn/dc is the rate of change of the refractive index versus the concentration of a solution. The dn/dc is an optical property of a polymer solution that is essential for accurate determination of the molecular weight using a light scattering technique. The dn/dc of the wall materials was measured using a BI-DNDC differential refractometer (Brookhaven Instruments Crop., Holtsville, NY, USA). Stock solutions (2 mg/mL) were prepared using 0.1 M sodium nitrate containing 0.5 g/L sodium azide. Dilutions were made from the stock solution, and five different concentrations were injected manually into the refractometer to determine the dn/dc value.

2.5. High pressure size exclusion chromatography (HPSEC)

The molecular weight distribution was measured using a method described by Kittisuban et al. [18] with some modification. The molecular weight distribution of each wall material was determined using an HPSEC coupled with multiple detectors: an Optilab rEX differential refractive index (RI) detector ($\lambda = 658$ nm), a ViscoStar-II differential pressure viscometer detector (VS) and a Dawn Heleos multi-angle laser light scattering (LS) detector ($\lambda = 661$ nm) (Wyatt Technology, Santa Barbara, CA, USA). One PL aquagel-OH MIXED-H 8 μm column (Agilent Technologies, Santa Clara, CA, USA) with a resolving range of 100 to 10,000,000 g/mol was used. The mobile phase consisted of a 0.1 M sodium nitrate solution containing sodium azide (0.5 g/L) and was filtered using 0.22 μm polyvinylidene difluoride (PVDF) filters. The flow rate was 0.5 mL/min, and the analyses were performed at room temperature. The wall materials (1.5 mg/mL) were dissolved in the mobile phase and filtered through a 0.22 μm PVDF syringe filter prior to analysis. The performance of the HPSEC system was checked using a monodisperse dextran standard (25 kDA). The data collected from the three detectors were evaluated using Wyatt Technology ASTRA software 5.3.4.14.

2.6. Preparation of spray dried tamarillo powders

Tamarillo fruits were obtained from three growers in the Cameron Highlands, Malaysia. The maturity of the fruits was between 21 and 24 weeks. The tamarillo fruits were washed and drained before being sent through a centrifugal juice extractor (Santos Juicer #28, France). The juices were packed in aluminum laminated polyethylene pouches and stored at −20 °C prior to use. Before spray drying, the tamarillo juices were thawed to room temperature and diluted with distilled water to 12° Brix. The wall material (20% w/w juice) was then added to the tamarillo juice, and the mixture was homogenized (DIAX 900 Heidolph homogenizer, Schwabach, Germany) for 10 min at 9500 rpm and filtered using a 100 μm mesh filter screen. Spray drying was performed using a pilot scale spray drier (Niro A/S, GEA, Germany) with an air flow rate of 900 m^3/min, a rotary atomizer with a speed of 15,000 rpm, an inlet air temperature of 150 °C, an outlet air temperature of 70 °C and a feed rate of 8 rpm. The spray drying conditions were selected after investigating the effect of the inlet air temperature (140–170 °C), outlet air temperature (60–90 °C) and feed rate (6–10 rpm) on the powder properties of tamarillo powders. The spray dried tamarillo powders produced were collected for the following study.