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The effect of pigment matrix, temperature and amount of carrier on the yield and final color properties of spray dried purple corn (*Zea mays* L.) cob anthocyanin powders

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ABSTRACT

Spray drying is an economic technique to produce anthocyanin-based colorants. High pigments yields with minimum color degradation are desirable to maximize quality and profits. This study evaluated the impacts of purple corncob (PCC) anthocyanin extraction matrices (hot water, 40% ethanol, C18 purified), drying inlet temperature (130, 150, 170 °C) and amount of carrier (2%, 5%, 10% maltodextrin) on the yields and quality of PCC anthocyanin powders. Monomeric and polymeric anthocyanins, color properties (CIELch, haze), and pigments composition before and after spray drying were determined.

The yield and final color quality of spray dried PCC anthocyanins were affected ($p < 0.05$) by all parameters evaluated. The pigment matrix, inlet temperature, and carrier amount had biggest impacts on product water solubility, pigments degradation and yield, respectively. The optimal combination of hot water extracts spray dried with 5% maltodextrin at 150 °C gave the highest pigment yield (~90%) with good solubility with the least color loss.

1. Introduction

Color plays an important role in consumers' acceptance of foods. Natural food colorants have increased in popularity compared to artificial dyes because they may help enhance visual appeal while adding health-promoting phytochemicals. According to a 2013 natural color market report (Mintel & Leatherhead Food Research, 2013), the global sales of synthetic dyes stayed almost unchanged from 2007 ($548 million) to 2011 ($570 million), while the sales of natural colors increased dramatically from $465 million in 2007 to over $600 million in 2011, with an annual growth rate of over 7%.

First introduced in milk powders production, spray drying is a continuous operation widely used to encapsulate functional compounds. It is 30–50 times less expensive than freeze-drying (Gharsallaoui, Roudaut, Chambin, Voilley, & Saurel, 2007) and has been successfully applied in fruits and vegetables source colorants production such as acai, berries, pomegranate, purple sweet potato, black carrot (Fazaeli, Emam-djomeh, Ashtari, & Omid, 2012; Jimenez-Aguilar et al., 2011; Robert et al., 2010; Tonon, Brabet, & Hubinger, 2010). Quality of the final spray dried products can be affected by inlet/outlet temperature, choice of carrier, physical properties of the feed, nozzle conditions, among other factors (Mahdavi, Jafari, Ghorbani, & Assadpoor, 2014).

Purple corn (*Zea mays* L.), originated from Peru, is abundant in health beneficial anthocyanins that could be a competitive candidate as natural pigment source. Compared to famous anthocyanins rich food blueberry (anthocyanin content 1.3–3.8 mg/g FW), purple corn has higher anthocyanins level of 6.8–82.3 mg/g FW depending on the sections (Cevallos-Casals & Cisneros-Zevallos, 2003; Li et al., 2008). The purple corn cob (PCC), being low in sugars and rich in soluble fiber, is considered an ideal starting material for spray dry pigment production compared to other plant anthocyanin sources. Soluble fiber had been reported as a drying aid in encapsulation in spray dried cactus pear pigments (Saénz, Tapia, Chávez, & Robert, 2009). However, spray dried PCC pigments has not yet been investigated.

Most previous research discussed physicochemical properties of spray dried anthocyanins, such as moisture content, water activity, bulk density, particle size, glass transition temperature, solubility, antioxidant capability, storage stability (Ahmed, Akter, Lee, &

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Eun, 2010; Ersus & Yurdagel, 2007; Fazaeli et al., 2012; Ferrari, Germer, & DeAguirre, 2012b). The detailed chemical profiles of the pigments changes during spray drying process, like HPLC chromatogram, %polymeric color were rarely discussed. Polymeric color is a widely used index for anthocyanin degradation in aqueous extract, juice and wine (Choi, Kim, & Lee, 2002; Gao, Girard, Mazza, & Reynolds, 1997; Giusti & Wrolstad, 2001). Different from monomeric anthocyanins, the C-4 position of polymeric anthocyanin is covalently linked to other phenolic compounds, which makes polymeric anthocyanins resistant to bisulfite bleach (Berké, Chèze, Vercauteren, & Deffieux, 1998). These polymeric anthocyanins generally have higher hydrophobicity, and larger molecular weight compared to monomeric anthocyanins, which may affect their water solubility in a food matrix and bioavailability to humans (Gao et al., 1997; Hager, Howard, & Prior, 2008b). Polymeric anthocyanins are typically formed during thermal juice/puree processing and long-time storage, during which monomeric anthocyanins and other phenolic compounds condense and form polymeric anthocyanin (Choi et al., 2002; Hager, Howard, Prior, & Brownmiller, 2008a). Changes in polymeric anthocyanins have been well studied in thermal processing of anthocyanin-rich juice and puree (Brownmiller, Howard, & Prior, 2008; Hager et al., 2008b), but changes in polymeric anthocyanins in spray drying anthocyanins-rich materials have not yet been reported.

The objective of this study was to investigate the PCC pigments yield (monomeric anthocyanins), compare the PCC anthocyanins composition (%polymeric color and HPLC profiles), color properties (CIELch), water solubility (%haze) before and after spray drying, as impacted by choice of feeding pigment matrix, inlet/outlet spray drying temperature as well as the amount of carrier applied.

2. Materials and methods

2.1. Materials and reagents

Dried purple corn cob (*Zea mays* L.) particles were kindly provided by Agroindustrial S.A.C (Lima, Peru). Maltodextrin (DE = 16.5–19.5) were purchased from Sigma-Aldrich (St. Louis, MO). All solvents and other chemicals were purchased from Fisher Scientific (Fair Lawn, NJ, USA).

2.2. Spray drying conditions

All spray dry experiments were performed by using a laboratory scale mini spray dryer B-290 (BÜCHI Labortechnik AG, Switzerland). The PCC pigment solutions used for spray drying were prepared as described in 2.2.1. All extracted PCC pigments were in aqueous matrices before loading them into the spray dryer. Briefly, after adjusting the feeding monomeric anthocyanins concentration to around 0.35 mg/mL, 500 mL of PCC pigment extract was mixed with maltodextrin under magnetic agitation and heated to around 50 °C. Then the pigments solution was pumped through a peristaltic pump with drying nitrogen flow rate of 35 m^3/h for spray-drying. The feed pigment flow rate was 1.5 mL/min.

2.2.1. Pigments matrices

Anthocyanins extracts obtained from three different solvents with different degree of pigment purity were pumped into spray dryer to investigate the effect of pigment matrix on the final quality of spray dried PCC pigments product. The solvents used in this experiment were 40% (v/v) aqueous ethanol, 0.01% 6 N HCl acidified hot water (70 °C) and 70% (v/v) aqueous acetone. Extraction was performed at room temperature if not specified. Ground PCC powders were soaked into solvent for 1 h on a magnetic stir plate with a stir rate of 150 rpm. The extract was then filtered through Whatman No.4 filter paper using a Buchner funnel. For the 40% (v/v) aqueous ethanol extract, the solution was sent to rotary evaporator at 40 °C under vacuum to remove ethanol before spray drying. As for the 70% (v/v) aqueous acetone extract, the solution was partitioned with chloroform before sending the aqueous colored portion to rotary evaporator at 40 °C under vacuum to remove the remaining acetone (Rodriguez-Saona & Wrolstad, 2001). The aqueous pigments solution obtained was then passed through a C18 cartridge to remove non-phenolic compounds, and recovered with acidified (0.01% HCl) methanol (Rodriguez-Saona & Wrolstad, 2001). The methanol was evaporated in a rotary evaporator at 40 °C and the pigments re-dissolved in acidified water (0.01% HCl) to make them ready for spray drying. The total soluble solids of the PCC pigment extracts before adding maltodextrin were all <1%, measured by a handheld refractometer (Fisher Scientific, China). The amount of maltodextrin mixed into pigments was 25 g, 5% (w/v) of the pigment solution, inlet temperature was 150 °C and outlet temperature was 105 ± 1 °C. The obtained spray dried powders was solubilized into of distilled water to measure their quality.

2.2.2. Inlet/outlet temperatures

Three different inlet temperature settings, 130 °C, 150 °C, or 170 °C, were used to investigate the effect of inlet temperature on the final quality of spray dried PCC pigments product. The outlet temperature depends mainly on the inlet temperature. The PCC pigments used for this part of the experiment were all recovered from 0.01% 6 N HCl acidified hot water (70 °C). Similar to the process described previously, the spray dryer was pre-heated to the set inlet temperature and 5% (w/v, 25 g) of the maltodextrin was mixed into 500 mL of PCC pigments solution before pumping into the spray dryer. The obtained spray dried powders was solubilized back into distilled water for quality evaluation.

2.2.3. Amount of maltodextrin

Three different maltodextrin amounts, 2%, 5%, or 10% (w/v), were mixed with colored solution extracted by hot acidified water to investigate the effect of amount of maltodextrin on the final quality of spray dried PCC pigments product. Similar to the process described previously, 2%, 5%, 10% (w/v, 10 g, 25 g, 50 g, respectively) of the maltodextrin was mixed into 500 mL of PCC pigments solution. The spray dryer was pre-heated to 150 °C inlet temperature before pumping the pigments in for drying. The obtained spray dried powders was solubilized into distilled water for quality evaluation.

2.2.4. Reconstitution of pigmented solution from dried powders

The spray-dried purple corn anthocyanins powders were recovered from the collection vessel only, any particles deposited on the dryer chamber were discarded. Powders were weighted, and only 1/5 of the powders was solubilized back to 100 mL of distilled water. The colored solution obtained was used for pigment quality evaluation as "after spray-drying" sample.

2.3. Quality evaluation parameters

Quality evaluation of the PCC pigments included pigment yield based on monomeric anthocyanins, polymeric anthocyanins, color characteristics and %haze in the initial and reconstituted pigment solution. The PCC anthocyanins HPLC chromatograms before and after spray drying were also analyzed.

2.3.1. Yield of the pigments

The spray-dried yield of PCC pigments was calculated using the following equation.