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Physical and storage properties of spray-dried blueberry pomace extract with whey protein isolate as wall material

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ABSTRACT

In the food industry, attempts have been made to extract and encapsulate bioactives from pomace using organic solvents, e.g., ethanol. Ethanolic extracts contain high concentrations of bioactives, but encapsulation of them with whey proteins presents challenges arising from ternary phase equilibrium. This study aimed to prepare and characterize spray-dried powders made from blueberry pomace extract and whey proteins. The resulting microcapsules measured 48.5 μm in diameter, had 5% moisture content, and contained 1.32 mg cyanidin-3-*O*-glucoside (C3G), 2.83 mg gallic acid equivalents (GAE) and 48.52 nmol Fe (II) equivalents per gram powder. Sorption data obeyed Guggenheim–Anderson–De Boer isotherm. Storage tests revealed first-order degradation kinetics for monomeric anthocyanins, a two-fold increase in total phenolics and slight increase in antioxidant capacity. Exposure to light was comparable to storage at 37 °C, but slightly more severe in decomposing monomeric anthocyanins. The spray-dried encapsulated powder could be used as a suitable health-promoting food ingredient.

1. Introduction

The health-promoting properties of blueberries (*Vaccinium* sp.) have generated considerable interest after a report was published about its high antioxidant activity among 42 fruits and vegetables (Lee and Wrolstad, 2004). Blueberries were found to be a rich source of bioactive compounds such as anthocyanins and other flavonoids. Anthocyanins can be considered "signature" health compounds because of their greater abundance than the other flavonoids found in blueberries, and their capability to cross the blood–brain barrier (Kalt et al., 2008). Higher concentrations of anthocyanins and phenolics are found in the pomace, which is a common by-product of juice processing (Khanal et al., 2012; Lee and Wrolstad, 2004). Pomace was used as an ingredient in extruded products with health benefits *in vivo*, such as reduction of plasma cholesterol and abdominal fat (Khanal et al., 2009, 2012). Purified blueberry anthocyanin extracts were found to be more effective than whole berries in altering the development of obesity (Prior et al., 2010) and this could also be true with pomace. We previously compared several solvent systems in the extraction of anthocyanins from whole blueberries and pomace. Results showed that the ethanolic pomace extract possessed the highest amount of total monomeric anthocyanins and total phenolics (Flores et al., 2013).

Purified anthocyanins are labile compounds and susceptible to degradation in the presence of high pH, oxygen, heat, light and metallic ions, among others (Castañeda-Ovando et al., 2009). Hence, microencapsulation, such as by spray drying, can be carried out to impart protection and facilitate targeted release (Betz and Kulozik, 2011). Both ethanolic and aqueous extracts from blueberry pomace were successfully spray-dried, but the anthocyanin content from the aqueous extract was 10-fold lower than that of the alcoholic extract (Jiménez-Aguilar et al., 2011; Ma and Dolan, 2011). Elsewhere, ethanolic extracts were also used in spray drying of anthocyanin-rich extracts from other botanical sources (Burin et al., 2011; Ersus and Yurdagel, 2007). Food-grade ethanolic extracts may be further processed for human consumption.

Whey proteins are by-products of cheese manufacturing with significant commercial potential. They possess superior gelling and emulsification properties, and an amino acid profile suitable for protein fortification in beverages. Other health benefits associated with whey proteins include antimicrobial activity, inhibition of angiotensin-converting enzyme, and anticarcinogenic activity, among others (Chatterton et al., 2006). They can also be processed into pH-sensitive hydrogels or nanoparticles for the controlled release of bioactive compounds such as anthocyanins. Whey proteins can thus be used as alternatives to polysaccharide-based wall

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materials with relatively greater functionality (Betz and Kulozik, 2011; Betz et al., 2012; Gunasekaran et al., 2007; Oidtmann et al., 2012). Consequently, we compared the *in vitro* release properties of anthocyanin extracts spray-dried with either gum arabic or whey protein isolates (Flores et al., 2014). Results showed a rapid increase in phenolics content and antioxidant activity with gum arabic particles during simulated gastric digestion, followed by a drastic decrease in antioxidant activity after simulated intestinal digestion. In contrast, whey protein microcapsules promoted a gradual increase in phenolics content and maintained a high level of antioxidant activity throughout the entire digestion process. Thus, whey protein microcapsules could be utilized as an encapsulant for sustained-release of phenolics with high antioxidant activity. However, to the best of our knowledge, whey protein isolates have yet to be used as wall material in the spray drying of aqueous, alcoholic anthocyanin extracts. This could be due to the complex temperature and pH dependence of a ternary mixture of alcohol, water, and β-lactoglobulin, the major protein in whey (Abascal and Lencki, 2004).

The economic potential of encapsulating aqueous, alcoholic anthocyanin extracts with whey protein isolates includes greater concentration of bioactives and reduced energy costs due to the absence of a lyophilization step to remove the solvent. In this study, our main goal was to prepare spray-dried powder made with whey protein isolate and an aqueous, ethanolic extract from blueberry pomace. We also characterized the powder properties including particle size, moisture sorption isotherm, and encapsulation efficiency, and investigated physicochemical properties including total monomeric anthocyanin content, total phenolics content and antioxidant capacity as affected by storage conditions. Results of this study can be used in the development of health-promoting food ingredients.

2. Materials and methods

2.1. Materials

Ripe rabbiteye ("Powderblue" cultivar) blueberries were harvested from the Horticulture Research Farm of the University of Georgia in July 2013 and immediately frozen at −20 °C prior to processing. The berries were processed within two months. Whey protein isolate, containing at least 95% protein (*Bi*Pro®) was a kind gift from Davisco Foods International (Eden Prairie, MN). Chemicals used were reagent-grade and obtained from the Sigma–Aldrich Chemical Company (St. Louis, MO).

2.2. Methods

2.2.1. Processing of anthocyanin-rich extract

The berries were thawed between 4 and 6 °C and blanched in boiling water for 3 min. The juice was expressed using a commercial 2.5−L centrifugal Kuvings NJ-9310U juicer (Elk Grove Village, IL). The pomace was collected and extracted with 80% (v/v) aqueous ethanol at a mass:volume ratio of 1:10 (pomace:solvent). Extraction was performed at room temperature (22 °C) for 24 h and at an agitation rate of 300 rpm. The extraction flasks were covered with aluminum foil to protect against photodegradation. The mixture was filtered and the supernate was collected. The alcohol content of the supernate was measured using a Fisherbrand Model 11-590 alcohol hydrometer (Fisher Scientific, Pittsburgh, PA) and was found to be 67% (v/v). Next, the residual solvent was evaporated in vacuum for 30 min (Rotavapor R-124, Büchi Corp., Newcastle, DE) under the following conditions: 10 kPa total pressure, 40 °C bath temperature, 5 °C cooling water temperature, and 180 rpm agitation rate. The alcohol content of the resulting concentrate was found to be 12% (v/v) and the pH was 3.4.

2.2.2. Spray drying of the blueberry extracts (BBE)

Examination of the ternary diagrams developed for mixtures of ethanol–water-β-lactoglobulin showed that at a pH of 3.0 and temperature of 20 °C, a transparent liquid could be obtained at low ethanol and low-to-moderate β-lactoglobulin concentrations (Abascal and Lencki, 2004). Changes in pH during addition of whey protein were also considered. Results of our preliminary trials revealed an optimum mass ratio of 8:67:25 of ethanol:water:whey protein isolate to maximize the amount of both BBE and whey protein at 22 °C. The final pH of the ternary mixture was 6.8. Consequently, the ternary mixture was spray dried using a Model B-290 mini spray dryer (Büchi Corporation, Flawil, Switzerland) under the following process conditions: 6 mL/min peristaltic pump speed (corresponding to 20% pump rate), 160 °C inlet air temperature; 86–90 °C outlet air temperature; 100% aspirator rate (corresponding to a maximum air flow of 35 m^3/h), actual air flow rate of 0.667 m^3/h (40 mm Q flow), and a nozzle setting of 1 cleaning cycle/min. The powders were collected and stored in polypropylene bottles at −20 °C.

2.2.3. Particle size distribution

Particle size distribution was measured using a laser diffraction analyzer (Model LS 13 320, Beckman Coulter Inc., Fullerton, CA) under the following conditions: 30% pump speed, 10% obscuration rate, 10 s wait before the first run, 10 s sonication at power setting of 2 before the first run, and 50 s run time. Polarization intensity differential scanning (PIDS) was turned on. An optical model was developed with refractive indexes of 1.333 for the fluid (water) and 1.473 for the solid particles. Volume mean diameters ($D_{4,3}$), and cumulative mean diameter values corresponding to 10th and 90th percentile of the distribution (d_{10} and d_{90}) were reported.

2.2.4. Chemical analyses

All powders were dissolved at 0.01 g/mL for about 1 h in deionized water prior to the tests. Whey protein isolate served as control.

2.2.4.1. Total monomeric anthocyanin content (TMAC), total phenolics content (TPC) and ferric reducing antioxidant power (FRAP). The tests were conducted according to the procedures in our previous study (Flores et al., 2013). The pH differential method was used to measure the total monomeric anthocyanins. Results were calculated as mg of total cyanidin-3-*O*-glucoside (C3G) per gram of powder. The total phenolics content was measured using the Folin–Ciocalteu method and calculated as mg gallic acid equivalent (GAE) per gram of powder. Antioxidant activity was measured by FRAP and computed as nmol Fe^{2+} equivalents per gram powder.

2.2.4.2. Encapsulation efficiency. The method of Idham et al. (2012) was employed with modifications. Fifty milligrams of the spray-dried powder was dissolved in 3 mL of 95% (v/v) ethanol in test tubes, agitated for 1 min with a vortex mixer and centrifuged for 10 min at 3823 *g*. The supernate was assayed for surface TMAC as described earlier and reported as mg surface C3G/g powder. The encapsulation efficiency is defined as follows:

$$\%\ \text{Encapsulation efficiency} = \frac{\text{Total C3G/g} - \text{Surface C3G/g}}{\text{Total C3G/g}} \times 100\% \quad (1)$$

2.2.5. Moisture sorption isotherm

The integral method was employed to develop sorption isotherms. Powder samples were placed in tared 7-mL borosilicate