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Wild blueberry polyphenol-protein food ingredients produced by three drying methods: Comparative physico-chemical properties, phytochemical content, and stability during storage

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ABSTRACT

Particulate colloidal aggregate food ingredients were prepared by complexing wheat flour, chickpea flour, coconut flour and soy protein isolate with aqueous wild blueberry pomace extracts, then spray drying, freeze drying, or vacuum oven drying to prepare dry, flour-like matrices. Physico-chemical attributes, phytochemical content and stability during storage were compared. Eighteen anthocyanins peaks were identified for samples. Spray dried matrices produced with soy protein isolate had the highest concentration of polyphenols (156.2 mg GAE/g) and anthocyanins (13.4 mg/g) and the most potent DPPH scavenging activity (714.1 μmoles TE/g). Spray dried blueberry polyphenols complexed with protein were protected from degradation during 16 weeks at 4 °C and 20 °C. Soy protein isolate more efficiently captured and stabilized wild blueberry pomace phytochemicals than other protein sources. Overall, spray drying the blueberry extracts complexed with protein proved to be an environment-friendly strategy to produce stable functional ingredients with multiple applications for the food industry.

1. Introduction

In the United States, several cultivated and wild species of blueberry represent together a major fruit commodity with a total annual production valued in approximately US$ 890 million in 2014 (USDA. Economic Research Service, 2016). The antioxidant and health relevant bioactive compounds in different blueberry species are well documented (Prior et al., 2010; Yousef et al., 2013), but wild blueberries (*Vaccinium angustifolium* Aiton) have shown consistently higher levels of phenolics, anthocyanins, and antioxidant capacity and more complex phytochemical profiles when compared to cultivated blueberries (Grace, Esposito, Dunlap, & Lila, 2014; Kalt et al., 2001). Wild blueberries have also demonstrated a wide collection of health-relevant bioactivities such as anti-diabetic (Grace et al., 2009), anti-hypertensive (Kalea, Clark, Schuschke, Kristo, & Klimis-Zacas, 2010), and anti-inflammatory effects (Esposito, Chen, Grace, Komarnytsky, & Lila, 2014).

Considerable amounts of pomace consisting of residual pulp, seeds and skin are generated during the industrial juicing process. There is growing evidence that these by-products of the fruit industry contain large residual amounts of bioactive compounds with health-related activities (Struck, Plaza, Turner, & Rohm, 2016) which has motivated the development of environmentally-friendly uses for waste pomaces (Roopchand, Kuhn, Krueger, et al., 2013).

Despite their diversity of health benefits, phytoactive components from fruits and pomace can be rapidly degraded with exposure to light, heat and oxygen, and anthocyanins in particular are known for their susceptibility to pH change, among other factors (Patras, Brunton, & O'Donnell, 2010). Therefore, processing techniques that stabilize these compounds and extend their shelf life while maintaining their bioactive properties would broaden opportunities for fruit processors, and potentially expand markets for fruit-based products.

Previously, we introduced an efficient strategy which used protein-rich flours to sorb and complex polyphenols from fruit extracts, while excluding sugar and fat, to create stable dry granular functional ingredients which provide a highly bioavailable delivery system for bioactive compounds (Ribnicky et al., 2014;

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Roopchand et al., 2012; Schneider, Esposito, Lila, & Foegeding, 2016). The drying step of this process has significant influence on the cost-effectiveness, functionality and bioactive potential of the final ingredient, however. Lyophilization (freeze-drying) is a laboratory standard for most effectively retaining bioactivity of phytochemical compounds, but it is a time-intensive and costly method (Karam, Petit, Zimmer, Djantou, & Scher, 2016). Vacuum oven drying is a relatively simple and popular technique, but it can also be expensive for large scale production and some degradation of ephemeral, heat sensitive phytochemicals can occur (Motevali, Minaei, Khoshtaghaza, & Amirnejat, 2011). We have recently shown that spray drying fruit pulps and pomace extracts increases their shelf-life while maintaining organoleptic and biofunctional properties (Borges et al., 2016). This technique is one of the most popular drying methods in the food industry, but its use as an efficient means to produce polyphenol-protein matrices has not been fully investigated. Interestingly, proteins can be used as carriers during the spray drying of fruit materials, and the final spray-dried material is less sticky and has higher product recoveries when compared to conventional polysaccharide-based drying aids (Bazaria & Kumar, 2016; Bhusari, Muzaffar, & Kumar, 2014). In addition, lower concentrations of proteins are required in order to accomplish an efficient spray drying process, as opposed to large amounts of conventional drying carriers, such as maltodextrin (Fang & Bhandari, 2012; Jayasundera, Adhikari, Howes, & Aldred, 2011).

In this study we compared the spray drying technique to freeze drying and vacuum oven drying to produce particulate food ingredients by complexing four different protein-rich food matrices (wheat flour, chickpea flour, coconut flour and soy protein isolate) with wild blueberry pomace extracts. The physico-chemical and bioactive properties of the polyphenol-protein matrices were evaluated as well as their stability during storage at different temperatures.

2. Materials and methods

2.1. Blueberry pomace

The wild blueberry (*Vaccinium angustifolium* Aiton) pomace used in this study was provided by Wyman's of Maine (Milbridge, ME, USA) and consisted of several batches of pomace produced as juicing by-products in 2015. The lyophilized material contained residual pulp, skin and seeds, and was kept frozen at −20 °C until use.

2.2. Production of blueberry polyphenol-protein matrices

Concentrated blueberry extracts were prepared from freeze dried blueberry pomace blended with 50% ethanol at 80 °C for 2 h, followed by filtration under vacuum through cheesecloth and centrifugation at 4000 rpm for 20 min. After ethanol evaporation in a rotary evaporator under vacuum at 40 °C under constant rotation, the resulting extract was mixed with each protein source. Four different protein sources were tested: organic whole wheat flour (Wh, 14% protein, 3% fat, Arrowhead Mills, Boulder, CO); chickpea flour (Ch, 20% protein, 7% fat, Bob's Red Mill, Milwaukie, OR), coconut flour (Co, 20% protein, 16% fat, Now Foods, Bloomingdale, IL) or soy protein isolate (SPI, 90% protein, 0.6% fat, ADM, Decatur, IL). The dry weight of the concentrated blueberry extract (10.1 ° Brix) was determined by freeze drying 4 ml aliquots of liquid extract. A calculated amount of each protein source (wheat flour, chickpea flour, coconut flours or soy protein isolate) was added to the liquid extract in order to standardize to a final concentration of 10% (w/v) total phenolics in the resulting aggregate matrices.

2.2.1. Drying

Three different drying strategies (freeze drying [FD], vacuum oven drying [VO], and spray drying [SD]) were used to produce the dried matrices, resulting in 12 experimental blueberry polyphenol-protein matrices: Wh-FD (wheat flour + blueberry, freeze dried); Ch-FD (chickpea flour + blueberry, freeze dried); Co-FD (coconut flour + blueberry, freeze dried); SPI-FD (soy protein isolate + blueberry, freeze dried); Wh-VO (wheat flour + blueberry, vacuum oven dried); Ch-VO (chickpea flour + blueberry, vacuum oven dried); Co-VO (coconut flour + blueberry, vacuum oven dried); SPI-VO (soy protein isolate + blueberry, vacuum oven dried); Wh-SD (wheat flour + blueberry, spray dried); Ch-SD (chickpea flour + blueberry, spray dried); Co-SD (coconut flour + blueberry, spray dried); SPI-SD (soy protein isolate + blueberry, spray dried).

Three independent drying batches were produced on different days, and results represent the average and standard deviation of samples. The freeze dried samples were produced in a lab-scale freeze drier (Labconco, FreeZone12, Kansas City, MO, USA) for 48 h. Drying conditions were 0.2 mBar condenser pressure and −45 °C drying temperature. The vacuum oven dried samples were obtained by spreading approximately 1-cm thick layers of the blueberry polyphenol-protein mixture on aluminum foil plates, and placing in a vacuum oven (Isotemp model 285A, Fisher Scientific, Pittsburgh, PA, USA) set at 80 °C. After 24 h, the dry flakes were ground (IKA, A11, Wilmington, NC, USA) and stored in a closed glass container at −20 °C. Spray dried samples were produced with a spray dryer (B-290, Buchi Labortechnik AG, Switzerland) with a 1.5 mm nozzle under the following conditions: 15 mL/min of feed flow controlled by peristaltic pump (corresponding to 50% of pump rate), inlet temperature of 190 °C and outlet temperature of 85–90 °C. The feed mixture (blueberry pomace extract mixed with protein) was kept under constant magnetic stirring at 20 °C. Nitrogen was used in co-current flow (100% aspirator rate, 35 mm Q flow, actual gas volume flow of 538 L/h). The operating conditions were defined by previous experiments (data not shown). After drying, the samples were collected from the collection chamber and immediately sealed, weighed and stored at −20 °C to prevent subsequent moisture uptake. The resulting dried blueberry polyphenol-protein matrices were used for further analysis. The schematic procedure for producing the blueberry polyphenol-protein is shown in Supplementary Fig. 1.

2.3. Spray drying yield (solids recovery)

The production yield of spray dried samples expressed as solids recovery was determined according to Daza et al. (2016). It was calculated as the ratio between the total solids content in the final matrix and the total solids content in the feeding mixture, and expressed as percentage (%).

2.4. Physico-chemical properties of blueberry polyphenol-protein matrices

2.4.1. Moisture and water activity

The moisture was calculated with 1–2 g of each treatment, which was dried in a vacuum oven at 70 °C until reaching constant weight (AOAC, 1990; met. 934.06). The water activity of the samples was measured using an Aqualab water activity meter (Decagon, Pullman, WA).