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Effect of drying technique and particle size of bilberry press cake on the extraction efficiency of anthocyanins by pressurized carbon dioxide extraction

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ABSTRACT

To improve the extraction efficiency of anthocyanins from bilberry press cake using pressurized carbon dioxide, the combined effect of drying technique and bilberry press cake particle size was assessed. Pressurized carbon dioxide using ethanol as co-solvent was compared with a simple and efficient solvent extraction using methanol. The press cake with large (>710 μm) size particles had a higher anthocyanins content (84 g/kg dry matter to 87 g/kg dry matter) than did the small (<710 μm) size particles (60 g/kg dry matter to 65 g/kg dry matter). Although, the large size particles contained more anthocyanins, more efficient anthocyanins extraction using pressurized carbon dioxide extraction was obtained with the small than the large size particles. The press cake dried by freeze-drying generated a powder with smaller particles and lower bulk density than either the microwave-assisted hot-air-dried or hot-air-dried powders. In comparison to methanol extraction, the most efficient anthocyanins extraction was obtained from the freeze-dried small size particles. This work showed that there is a potential to improve the extraction efficiency of anthocyanins extracted by pressurized carbon dioxide by selecting appropriate drying technology and particle size distribution of the press cake.

1. Introduction

The health benefits of berries are driving the consumption of fresh and processed berry products worldwide. Bilberries (*Vaccinium myrtillus*) are native to northern Europe and North America (TRI, 2001), and are particularly well known for their high content and large variety of anthocyanins, with up to sixteen anthocyanins structures identified (Paes, Dotta, Barbero, & Martínes, 2014).

Bilberry press cake is a byproduct from the production of bilberry juice having an alcohol-insoluble solid content of 370 g/kg fresh material, it is mostly composed of hemicellulose and cellulose (Aura et al., 2015). In addition, press cake is an important source of bioactive compounds such as anthocyanins and other polyphenols (Paes et al., 2014). Anthocyanins are water-soluble pigments making them desirable natural alternatives to synthetic colorings (Skrede & Wrolstad, 2002), and their antioxidant activity can prevent oxidative damage in foods (Viljanen, Kylli, Hubbermann, & Heinonen, 2005) and prevent ocular and vascular disorders, and diabetes mellitus (Pojer, Mattivi, Johnson, & Stockley, 2013; TRI, 2001). Laaksonen, Sandell, and Kallio (2010) reported that the main part of the bilberries' anthocyanins remain in the press cake after juice pressing. Despite the high nutrition value and economical interest in bioactive substances this byproduct is underutilized.

Solvent extraction of anthocyanins has been conventionally performed using organic solvents, such as methanol due to a high recovery (Francis, 1982). However, for food applications, alternative solvents are desired due to the non-food-grade status as well as human and environmental toxicity of solvents like for example methanol.

Carbon dioxide (pressurized or supercritical) combined with ethanol or ethanol/water mixtures has been investigated, as more environmentally friendly alternative with food grade status, to extract anthocyanins from various byproducts: bilberry press cake (Kerbstadt, Eliasson, Mustafa, & Ahrné, 2015), blueberry residues (Paes et al., 2014) and elderberry pomace (Seabra, Braga, Batista, &

Abbreviations: MWD, microwave-assisted hot-air drying; HAD, hot-air drying; FD, freeze-drying; TAC, total anthocyanins content; DM, dry matter.

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de Sousa, 2010).

Anthocyanins are sensitive to degradation (Skrede & Wrolstad, 2002), imposing stringent demands on byproduct handling before extraction. Byproducts are commonly dried before extraction for improved extraction efficiency, and the choice of drying conditions and technique has been showed to be important (Kerbstadt et al., 2015). Furthermore, the characteristics of the matrix, such as particle size (Laroze, Díaz-Reinoso, Moure, Zúñiga, & Domínguez, 2010) and structure (Türker & Erdoğdu, 2006), affect the extraction efficiency. The drying process causes differences in porosity and strength of the dried material (Krokida & Maroulis, 1997), that are likely to influence subsequent milling and extraction efficiency. Both freeze-drying (Cardoso et al., 2013) and hot-air drying (Paes et al., 2014; Seabra et al., 2010) are frequently used before extraction. Freeze-drying creates a porous structure due to the sublimation process and helps preserve temperature-sensitive compounds by avoiding high temperatures (Fellows, 2000; Krokida & Maroulis, 1997). Microwave drying is another possibility that was used by Kerbstadt et al. (2015) before anthocyanins extraction. Compared with air drying, microwave drying is faster and case hardening of the product surface is not formed creating a structural more homogeneous and porous product (Chua & Chou, 2006).

To our knowledge, the effect of various drying techniques and press cake particle size on anthocyanins extraction efficiency using high-pressure carbon dioxide has not been previously studied. The objective of this work was accordingly to assess the potential to improve the extraction efficiency of anthocyanins by pressurized carbon dioxide with ethanol co-solvent, compared with methanol extraction, by investigating the combined effects of three drying techniques (i.e. microwave-assisted hot air drying, hot air drying and freeze-drying) and two press-cake particle sizes (<710 μm or >710 μm).

2. Material and methods

A batch of bilberry (*Vaccinium myrtillus*) press cake was supplied by Svantes Vilt & Bär (Harads, Sweden). The press cake was produced by cold pressing without enzymatic treatment, had a moisture content of 747 g/kg ± 40 g/kg at delivery, and was stored in darkness at −40 °C until use. The press cake was thawed in darkness at 4 °C overnight (15 h) before drying, milling, fractionation, and extraction.

2.1. Drying bilberry press cake

Bilberry press cake was dried by freeze drying (FD), hot air drying (HAD) at 40 °C and 70 °C, and microwave assisted hot air drying (MWD) at 40 °C and 70 °C. The various drying techniques and temperatures were intended to create dried materials with different characteristics (in terms of structure, thermal degradation, and cell disruption) that might influence subsequent milling and extraction. With all techniques, the bilberry press cake was dried to a moisture content of approximately 200 g/kg. After drying, the material was stored in sealed polyamide/polyethylene plastic pouches, in darkness, at −20 °C until milling and extraction.

2.1.1. Freeze-drying

Approximately 60 g of frozen bilberry press cake was placed on the top shelf in a laboratory freeze dryer (ALPHA 1–2 SDplus; Christ, Osterode am Harz, Germany) connected to a vacuum pump (RZ 2.5; Vacuubrand, Wertheim, Germany). To avoid the influence of light, the freeze dryer was covered with aluminum foil. FD was performed for 420 min in four replicates. The samples from these four runs were mixed, and the resulting material had a moisture content of 215 g/kg ± 20 g/kg.

2.1.2. Hot-air drying

HAD was conducted in a hot-air oven (Garomat 142; Electrolux, Stockholm, Sweden), in which two trays (21 × 30 cm), each containing approximately 150 g of bilberry press cake, were placed on the middle shelf. The bilberry press cake was dried, in the absence of light, for 220 min at 40 °C to a moisture content of 198 g/kg ± 11 g/kg or for 90 min at 70 °C to a moisture content of 212 g/kg ± 50 g/kg The fan of the oven created an air velocity of 6.1 m s^{-1}. The hot air drying was done in one single run with two trays of material.

2.1.3. Microwave-assisted hot-air drying

MWD was performed according to the procedure described by Kerbstadt et al. (2015). In brief, 500 g of bilberry press cake was loaded in a microwave cavity (Tivox AB, Tidaholm, Sweden) connected to an air heating unit with a fan (Honeywell INU Control AB, Borås, Sweden), resulting in an air speed of 0.8 m s^{-1} in the middle of the cavity. A maximum microwave power of 1000 W was supplied by a MagDrive-1400 (Tivox AB, Tidaholm, Sweden) at a wavelength of approximately 0.12 m and a frequency of 2450 MHz. To keep the desired treatment temperature, the microwave power was regulated automatically by MagDrive v3.1 software (Tivox AB, Tidaholm, Sweden), dependent on the sample temperature as measured using fiber optic temperature probes (Neoptix Inc., Ville de Québec, QC, Canada). The bilberry press cake was dried in the absence of light for 190 min at 40 °C to a moisture content of 200 g/kg ± 30 g/kg or for 57 min at 70 °C to a moisture content of 200 g/kg ± 20 g/kg. MWD was conducted in duplicate.

2.2. Moisture content determination

The moisture content of the dried bilberry press cake was measured gravimetrically in a vacuum oven (Sanyo Gallenkamp, Loughborough, UK). Analysis was conducted in triplicate, for which approximately 3 g of sample were weighed in an aluminum dish and dried at 80 °C until constant weight.

2.3. Milling and particle size separation of dried press cake

The dried bilberry press cake was milled, from frozen condition, in a small-scale knife mill (model 2393; OBH Nordica, Stockholm, Sweden). Approximately 25 g of press cake were ground for 30 s. After milling, the powder was sieved in a vibratory sieve-shaker (Analysette 3; Fritsch, Idar-Oberstein, Germany), for 10 min at an amplitude of 1.5 mm and interval time of 10 s, to separate the particles into two fractions having particle size, i.e. <710 μm and >710 μm, to be used in the subsequent extraction step.

2.4. Characterizing the dried and milled press cake

Each dried and milled press cake was characterized in terms of particle size distributions, bulk density followed by imaging.

The particle size distribution of the milled press cake was analyzed using sieves of mesh sizes 500 μm, 710 μm, and 1250 μm. The same sieve-shaker and settings, as described in section 2.3, were used. The sieving was conducted in duplicate.

The bulk density (g/mL) of each fraction (i.e. <500 μm, 500–710 μm, 710–1250 μm, and >1250 μm) was determined by transferring each fraction to a 50-mL graduated cylinder for weight and volume measurement. The bulk density was measured as the poured bulk density without any tapping, as this corresponds to how the powder is loaded in the extraction basket. The measurements were made in duplicate.

The powder of each particle size fraction was visualized by