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HPLC-DAD-ESI-MS^2 analytical profile of extracts obtained from purple sweet potato after green ultrasound-assisted extraction

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ABSTRACT

Ultrasound pre-treatment (UAE) was applied to assist the extraction of valuable compounds (polyphenols (especially anthocyanins), and proteins) from purple sweet potato (PSP). Under optimum conditions (ultrasound time (40 min); supplementary hot extraction (80 °C) up to 120 min; pH: 2.5; ethanol concentration: 58%), the highest concentrations of polyphenols (3.877 mg/g), anthocyanins (0.293 mg/g), and proteins (0.753 mg/g) were found, with minimal specific energy consumption (8406 J/mg).

Moreover, anthocyanin and non-anthocyanin polyphenols in PSP extract from optimized extraction temperature were identified using HPLC-DAD-ESI-MS^2. The major identified anthocyanins were peonidin-3-caffeoyl-*p*-hydroxybenzoyl sophoroside-5-glucoside, peonidin-3-(6″-caffeoyl-6‴-feruloyl sophoroside)-5-glucoside, cyanidin-3-caffeoyl-*p*-hydroxybenzoyl sophoroside-5-glucoside, whereas the major identified non-anthocyanin molecules were quinic acid, chlorogenic acid, caffeic acid, and chlorogenic acid-3-glucose. The amount of the predominant anthocyanin and non-anthocyanin compounds from PSP extract obtained after UAE was higher than that extracted after conventional solvent extraction. The results obtained in this work demonstrated the efficiency of UAE for the recovery of anthocyanins from PSP.

1. Introduction

Sweet potatoes constitute an important natural source of dietary fibers, minerals, vitamins, and antioxidants, which can be used for human and animal consumption (Teow et al., 2007).

This plant is one of the most promising economic crops due to its high yielding and strong resistance capabilities for using in various environments, soils, and temperatures. Todays, sweet potato is considered as the seventh most important food crop in the world. In China, sweet potato accounts for 90% of worldwide sweet potato production; >117 million tons is produced annually (Qiongying et al., 2015). In particular, purple sweet potatoes (PSP) contain high level of polyphenols (e.g. anthocyanins), similar to that accumulated in white, yellow, or orange potatoes (Wang, Yu, & Song, 2011). Valuable compounds in PSP extracts have been shown to display a remarkable spectrum of biological activities, such as hepatoprotective (Hwang, Choi, Choi, Chung, & Jeong, 2011), antioxidant activity (Zhang et al., 2010), and memory enhancing properties (Lu et al., 2012). Moreover, PSP colorants are highly sought by food industry as low-cost crop, due to their lack of toxicity, unique color, and nutritional benefits (Jansen & Flamme, 2006). Thus, currently there is growing worldwide interest in both the fresh market and processing industries in developing the use of PSP.

In the line of PSP feedstock valorization, it is of paramount importance to find novel methodologies to improve the extraction yield of PSP colorants. Numerous methods (e.g. pure water extraction and conventional solvent extraction) have been described in the literature to extract polyphenols from PSP (de Aguiar Cipriano, Ekici, Barnes, Gomes, & Talcott, 2015), observing low

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extraction yields. High temperature as 80 °C was applied for valuable compounds (anthocyanins) extraction with high yields (Bridgers, Chinn, & Truong, 2010). Although conventional solvent extraction (CSE) techniques are the most common, there is a need to develop alternative techniques and methods to increase efficiency and selectivity, meantime avoid or minimize the use of toxic solvents (e.g. methanol).

Recently, the potential of numerous non-conventional extraction techniques such as ultrasound-assisted extraction (UAE), supercritical fluid extraction (SFE), and microwave-assisted extraction (MAE) have been evaluated to overcome the issues encountered for CSE, and increase the extraction yields (Bursać Kovačević et al., 2016; Galanakis, 2012; Koubaa et al., 2015). Among these technologies, UAE has been demonstrated as an interesting tool to recover polyphenols from plant food materials, due to its cheapness and low equipment and maintenance cost compared to other non-conventional techniques (Roselló-Soto et al., 2015).

Enhancing the extraction efficiency by using ultrasound is mainly attributed to the cavitational effect generated by the ultrasonic waves (Wang, Sun, Cao, Tian, & Li, 2008). Cavitation is the result of creation, growth, and implosion of gas bubbles generated during ultrasonic treatment. These bubbles collapse on the surface of plant material and release high pressure and temperature, which generate shock waves and lead to micro-fractures formation. Ultrasound also exerts mechanical effect that allows better penetration of extracting solvent into the sample matrix, which increases the contact surface area between solid and liquid phases (Koubaa et al., 2016). Increased mass transfer and significant disruption of cell walls come as a result of these combined effects. It has been also reported in literature that ultrasonic waves could be associated with some chemical effects, which are rather undesirable due to the changes in chemical composition, possible degradation of targeted compounds, and the production of free radicals within the gas bubbles (Paniwnyk, Beaufoy, Lorimer, & Mason, 2001). Moreover, it is also important to optimize acidity and extraction time for valuable compounds recovery (Putnik, Bursać Kovačević, & Dragović-Uzelac, 2015). Therefore, the extraction conditions such as time, temperature, ultrasonic power, and frequency must be accurately determined.

Developing low cost tailor-made products is nowadays required to meet the new trends in food and bioprocess technologies. This development depends on the production costs estimated during the design and the scale up of processing equipment, usually by numerical models that have proven their efficiency in reducing the processing time and costs (Roselló-Soto et al., 2015). Among the different modeling approaches, response surface methodology (RSM) has been widely used in the optimization of food processes as it is a valuable tool to investigate the interaction between factors and to quantitatively depict the effects of given parameters on their measured responses (Baş & Boyacı, 2007).

Although previous studies have evaluated the potential of UAE to extract total phenolic compounds (TPC) and total anthocyanins (TA) from PSP (Cai et al., 2016), there is a lack of information about the polyphenol profiles, which is important to target the extract's application. For instance, it has been shown that the bioavailability of polyphenols differ according to their total amount and their composition (D'Archivio, Filesi, Varì, Scazzocchio, & Masella, 2010). In this line, this work was devoted 1) to evaluate and optimize UAE conditions, pH, and ethanol concentration in order to improve the recovery of polyphenols, and especially anthocyanins, as well as proteins from purple sweet potatoes, compared to conventional solvent extraction, and 2) to identify the polyphenols' composition in both extracts (with and without UAE), using HPLC-DAD-ESI-MS^2.

2. Materials and methods

2.1. Plant material

Fresh purple sweet potatoes (PSPs) (E-Shu No.8) were kindly provided by the Institute of Food Crops, Hubei Academy of Agricultural Sciences (China), and were stored at 4 °C until use. Prior to extraction, entire PSP root was washed with cold water, and was cut into disks of 4 cm diameter and 0.5 cm thickness (25 ± 0.5 g). PSP disks were used for conventional solvent extraction (CSE) and UAE tests.

2.2. Chemicals and reagents

Folin-Ciocalteu (FC) reagent was purchased from Sigma-Aldrich (St. Louis, MO, USA). Analytical grade ethanol, gallic acid, sodium carbonate, HPLC grade methanol, and acetic acid were purchased from Tianjin Kermel Chemical Reagent Co., Ltd. (Tianjin, China).

2.3. Valuable compounds extraction from PSP

2.3.1. Ultrasound-assisted extraction (UAE)

UAE experiments were carried out using an ultrasonic bath SB-3200 DTS (produced by Ningbo Scientz Biotechnology Co. LTD, bath frequency 45 kHz, power input 178 W). The set-up allowed the control of time and temperature. Ultrasound treatments were applied to the mixture of PSP and solvent at a solid/liquid ratio of 1:20 (25 ± 0.5 g PSP and 500 g hydroalcoholic solution) at room temperature (25 ± 4 °C) in a water bath for 10, 25 and 40 min. The increase of temperature was strictly monitored, and tests were conducted at 40 ± 4 °C, 60 ± 4 °C and 80 ± 4 °C. Once the planned temperatures were achieved, they were kept constant up to 120 min (supplementary extraction). The temperature was controlled using a water bath and thermocouples.

A solution of 4% hydrochloric acid was added dropwise to obtain an extraction solution with a pH ranging from 1 to 4 (as described below using RSM), which was in the pH range of maximum anthocyanins' color stability (Lapornik, Prošek, & Golc Wondra, 2005). After reaching the extraction time, the resulting extract was first filtered with double gauze, and then centrifuged at 4000 rpm for 10 min. The supernatant was subjected to evaporation in a rotary evaporation system at 37 °C and a vacuum degree of 50 mbar to remove the remaining ethanol, and the volume was finally concentrated to 100 ml containing water and solutes. The resulting extract was stored at −20 °C until needed for analysis.

2.3.2. Conventional solvent extraction (CSE)

CSE (without ultrasound assistance) was carried out under the optimal conditions (pH, temperature, and ethanol concentration) found using RSM optimization of UAE process, for the sake of comparison.

2.4. Chemical analyses and calculations

2.4.1. Anthocyanin analysis

2.4.1.1. HPLC-DAD-ESI-MS^2 analysis. Anthocyanin profiles of PSP extracts were obtained using high performance liquid chromatography (HPLC) (with diode array detector (DAD)) coupled to mass spectrometry (MS) (with electrospray ionization interface (ESI)) (HPLC-DAD-ESI-MS^2). Accela series HPLC instrument (Thermo Fisher, San Jose, CA, USA) coupled with Accela LTQ XL mass spectrometer (Thermo Fisher, San Jose, CA, USA) equipment were used for the analysis. HPLC instrument was composed of two Accela 600 pumps, Accela auto-sampler, and Accela PDA detector. Chromatographic separation was carried out using a