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Proposal for fractionating Brazilian ginseng extracts: Process intensification approach

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ABSTRACT

Brazilian ginseng (*Pfaffia glomerata*) roots contain several bioactive compounds, including beta-ecdysone with several therapeutic effects, saponins with surface activity and fructooligosaccharides (FOS) with prebiotic effects. Regarding this rich composition, a two-step intensified extraction process that uses the same solid-liquid extraction apparatus was developed to obtain and fractionate the bioactive compounds from Brazilian ginseng roots using ethanol and water as the extraction solvents. The intensified process was compared to a conventional extraction process using water as the solvent. The beta-ecdysone and saponins were mainly concentrated in the ethanolic extract obtained in step 1 of the intensified extraction process, while the FOS was isolated in the aqueous extract obtained in step 2. The effect of pressure (0.1, 5 and 10 MPa) on the extraction yield, beta-ecdysone content, saponin content, water surface tension reduction rate and FOS content was evaluated using the analysis of variance (ANOVA) statistical method. The highest beta-ecdysone content (5.6%, d.b.) was obtained at ambient pressure (0.1 MPa), while the highest saponin content (55%, d.b.) was obtained at 5 MPa. However, the extracts that had better surface activity were also obtained at ambient pressure. The total FOS content obtained at ambient pressure was 7.9% (d.b.). The kinetic study showed that suitable process times for the first and second steps were 38 and 110 min, respectively.

1. Introduction

New perspectives for the manufacturing chains are focused on improving the use of raw materials using green technologies to increase productivity without affecting product quality (Vardanega et al., 2015; Zabot et al., 2015). This focus meets the process intensification concept, which includes initiatives that increase the production capacity within a given equipment volume, decrease the energy consumption per ton of product and reduce the residue formation (Stankiewicz and Moulijn, 2000). In this context, extraction processes that result in several value-added compounds from a vegetal matrix using the same equipment or process improvements that increase its efficiency are considered intensified processes (Shirsath et al., 2012).

Recently, an intensified process was developed to obtain turmeric oil, and an extract rich in curcuminoids from the turmeric (*Curcuma longa* L.). The turmeric oil was obtained using supercritical fluid extraction, and the extract was obtained by pressurized liquid extraction. Both processes were performed sequentially with the same equipment (Carvalho et al., 2015; Osorio-Tobón et al., 2014). A similar intensified process was performed to obtain a volatile oil, and an extract rich in phenolic compounds from rosemary leaves (*Rosmarinus officinalis*) (Zabot et al., 2015). This strategy is also suitable for obtaining bioactive compounds from Brazilian ginseng (*Pfaffia glomerata*) roots (BGR).

BGR contain secondary metabolites, such as beta-ecdysone and saponins, that have pharmacological and medicinal properties (Freitas et al., 2004; Nakamura et al., 2010; Neto et al., 2004, 2005; Rates and Gosmann, 2002). Saponins also possess surfactant activity (Bitencourt et al., 2014; Vardanega et al., 2014) and can be used to obtain stable emulsions (Rosa et al., 2016; Santos et al., 2013b). Furthermore, inulin-type fructans, also named fructooligosaccharides (FOS), were found in the BGR (Caleffi et al., 2015; Vardanega, 2016). FOS plays an important role in the human

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body and is considered to be a prebiotic compound because it is a non-digestible carbohydrate and it acts in the host by selectively stimulating the growth and/or activity of some microorganisms in the colon (Roberfroid et al., 2010).

Studies have been developed to separately evaluate different processes to obtain the BGR fractions containing one of the compounds of interest, such as supercritical fluid extraction and pressurized liquid extraction to obtain beta-ecdysone-rich extracts (Debien and Meireles, 2014; Debien et al., 2015; Leal et al., 2010), low pressure solvent extraction to obtain saponin-rich extracts from the BGR (Vardanega et al., 2014) and a sequential extraction process using supercritical CO_2, ethanol and water to fractionate the saponins from the BGR (Bitencourt et al., 2014). However, to our knowledge, there is no study about the extraction of FOS from BGR.

Based on these aspects, the aim of this study was to propose an intensified process for Brazilian ginseng roots processing that uses the same solid-liquid extraction equipment to obtain extracts rich in beta-ecdysone, saponins and FOS, sequentially using ethanol and water as the solvents. The effect of pressure on the intensified extraction was also studied and was compared to a conventional extraction process using water as the extraction solvent.

2. Materials and methods

2.1. Raw material

Brazilian ginseng roots (BGR) were collected and prepared as described by Vardanega et al. (2014). The BGR were washed and dried at 313 K for 5 days in a forced air circulation dryer. The dried BGR (9.2% moisture) were comminuted in a pulse mill (Marconi MA 340, Piracicaba, Brazil) and the larger particles were also comminuted in a knife mill (Tecnal, model TE 631, Piracicaba, Brazil) for 2 s at 18,000 rpm. The milled BGR were separated according to size in Tyler series sieves (W. S. Tyler, Wheeling, IL). The mean diameter of the particles (8.0 μm) was determined using the ASAE method (ASAE, 1998). The BGR were stored in a freezer (Metalfrio, model DA 420, São Paulo, Brazil) at 263 K.

2.2. Intensified extraction process

2.2.1. Equipment

The apparatus used to develop the intensified extraction process is shown in Fig. 1 and it consists of an HPLC pump (Thermoseparation Products, model ConstaMetric 3200 P/F, Florida, USA), a 6.57-mL extraction cell (Waters, serial # 4501374824-10, Pittsburg, USA) containing a sintered metal filter at the bottom and upper parts, an electrical heating jacket and a back pressure regulator (Tescom, 26-1761-24-161, ELK River, USA). A more detailed description of the unit can be found elsewhere (Santos et al., 2012).

2.2.2. Extraction procedure

The intensified extraction (IE) was performed in two steps in a sequential manner. Ethanol was used as the extraction solvent in the first step, and water was used in the second step. The effect of the following pressures were evaluated: 0.1 MPa (ambient pressure) (IE-0.1), 5 MPa (IE-5) and 10 MPa (IE-10). For each experiment, 4.5 g (wet basis) of the BGR were used. The bed height (H_B) was 2.0 cm, and the diameter (D_B) was 1.6 cm, resulting in an H_B/D_B ratio of 1.25. The flow rate and temperature were defined as 2.0 mL min^{-1} and 333 K, respectively, for both steps. A S/F value (kg of solvent/kg of raw material, wet basis) of 50 was defined to assure that all of the extractable compounds were obtained. First, ethanol was pumped to reach a S/F of 50; the second step was started immediately after obtaining the ethanolic extract by replacing the extraction solvent with water. Then, water was pumped to achieve a S/F of 50. A comparative extraction (CE) process using only water as the solvent at 333 K and ambient pressure was performed until a S/F of 50 was reached for comparison purposes. To remove the solvent, the ethanol was eliminated from the ethanolic extracts in a rotaevaporator (LS Scientific, LSRE-52CS-BA, Lagos, Nigeria) at

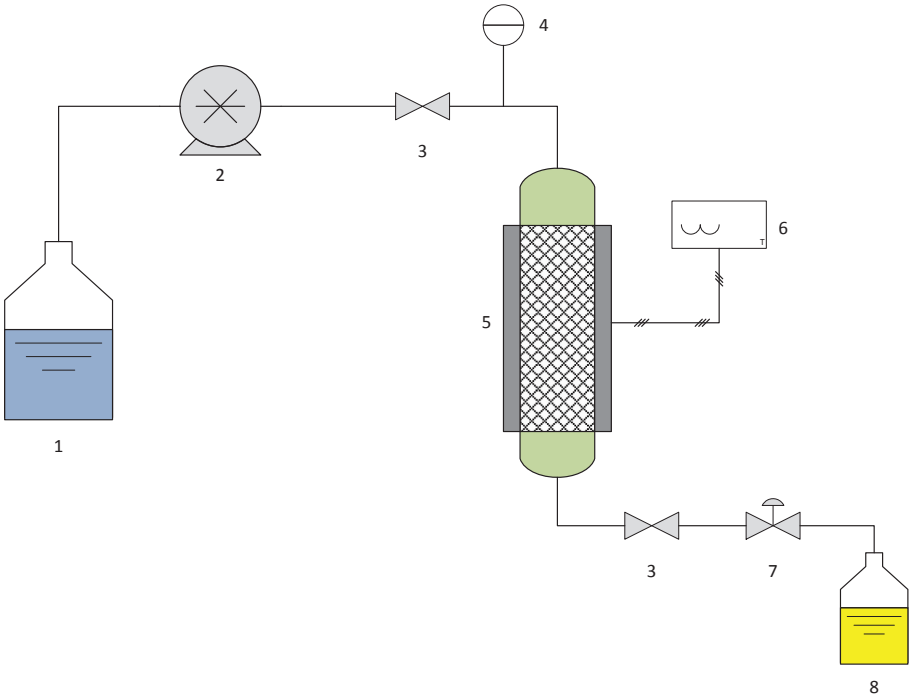

Fig. 1. The experimental apparatus used for the intensified extraction. (1) Solvent reservoir, (2) Liquid pump, (3) Blocking valves, (4) Pressure gauge, (5) Extraction vessel equipped with a jacket for heating, (6) Temperature controller, (7) Back pressure regulator, and (8) Extract collecting vessel.