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Sequential high pressure extractions applied to recover piceatannol and scirpusin B from passion fruit bagasse

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ABSTRACT

Passion fruit seeds are currently discarded on the pulp processing but are known for their high piceatannol and scirpusin B contents. Using pressurized liquid extraction (PLE), these highly valuable phenolic compounds were efficiently extracted from defatted passion fruit bagasse (DPFB). PLE was performed using mixtures of ethanol and water (50 to 100% ethanol, w/w) as solvent, temperatures from 50 to 70 °C and pressure at 10 MPa. The extraction methods were compared in terms of the global yield, total phenolic content (TPC), piceatannol content and the antioxidant capacity of the extracts. The DPFB extracts were also compared with those from non-defatted passion fruit bagasse (nDPFB). Identification and quantification of piceatannol were performed using UHPLC–MS/MS. The results showed that high TPC and piceatannol content were achieved for the extracts obtained from DPFB through PLE at 70 °C and using 50 and 75% ethanol as the solvent. The best PLE conditions for TPC (70 °C, 75% ethanol) resulted in 55.237 mg GAE/g dried and defatted bagasse, whereas PLE at 70 °C and 50% ethanol achieved 18.590 mg of piceatannol/g dried and defatted bagasse, and such yields were significantly higher than those obtained using conventional extraction techniques. The antioxidant capacity assays showed high correlation with the TPC ($r > 0.886$) and piceatannol ($r > 0.772$). The passion fruit bagasse has therefore proved to be a rich source of piceatannol and PLE showed high efficiency to recover phenolic compounds from defatted passion fruit bagasse.

1. Introduction

Piceatannol (3,3′,4,5′-trans-trihydroxystilbene) is a naturally occurring phenolic compound and a hydroxylated analogue of resveratrol, and both of these molecules display a wide spectrum of biological activities. Besides its antioxidative effects, piceatannol also exhibits potential anticancer properties, as suggested by its ability to suppress proliferation of a wide variety of tumor cells, including leukemia, lymphoma as well as cancers of the breast, prostate, colon and melanoma (Piotrowska, Kucinska, & Murias, 2012). Scirpusin B is dimer of piceatannol that shows strong vasorelaxant effect and prevents the postprandial elevation of blood glucose by improving the glucose metabolism (Matsumoto et al., 2014; Sano, Sugiyama, Ito, Katano, & Ishihata, 2011). Piceatannol and scirpusin B have been found in several plants, and relatively high amounts of those important phenolic compounds have been found in passion fruit seeds (Matsui et al., 2010; Sano et al., 2011).

In Brazil, the production of passion fruit is an important part of the fruit marketing. The most cultivated species in Brazil is the yellow passion fruit (*Passiflora edulis*), which is grown in 95% of orchards. Several passion fruit by-products are generated in the industrial processes during the pulp separation, and it is estimated that the residues from passion fruit processing juice reach 40–60% of the amount of processed fruits. About 90% of the passion fruit by-products are composed by rinds and bagasse (Malacrida & Jorge, 2012). The spoil of this material represents therefore a missed opportunity to mitigate environmental impacts and to generate new income sources by producing new products.

The production of polyphenols from natural sources has always attracted special interest. Proper and efficient extraction is perhaps the crucial goal for the isolation, identification and application of phenolic compounds (Garmus, Paviani, Queiroga, Magalhães, & Cabral, 2014), but the selection of the most sustainable extraction technique is difficult, since there are many extraction procedures that depend on numerous parameters. For example, supercritical fluid extraction (SFE) using CO_2 as the solvent is normally appropriated technique for the recovery of nonpolar compounds, whereas pressurized liquid extraction (PLE) is more suited for polar compounds, such as polyphenols (Wijngaard,

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Hossain, Rai, & Brunton, 2012). SFE has already been performed to recover oil from passion fruit bagasse, but this by-product has not been explored as a source of phenolic compounds via techniques that employ pressurized liquids, such as PLE.

For the extraction of bioactive compounds, PLE has been widely compared to other techniques such as maceration, ultrasound extraction, solvent partitioning or Soxhlet extraction, and has normally found to be superior. The main advantages of PLE have been related to higher extraction yields, the use of significantly less amounts of solvents, lower total extraction times, the possibility of process automation and the use of solvents classified as safe, such as ethanol, water or their mixtures (Herrero, Castro-Puyana, Mendiola, & Ibañez, 2013).

The aim of this study was therefore to explore the use of PLE using water–ethanol mixtures to extract piceatannol and scirpusin B from defatted passion fruit bagasse, and to compare the efficacy of PLE to that of conventional extraction methods.

2. Material and methods

2.1. Chemicals

Ethyl alcohol P.A., methyl alcohol P.A. (Synth, SP, Brazil), distilled water and CO_2 (White Martins, Brazil) with 99.0% purity were used as solvents. For the evaluation of total phenolic content Folin–Ciocalteu reagent and gallic acid were purchased from Sigma-Aldrich (SP, Brazil). For the chromatographic analyses, formic acid and methanol of HPLC grade were purchased from Merck S.A. (Rio de Janeiro, Brazil), deionized water was obtained from a Milli-Q (Millipore, Billerica, MA) purification unit and piceatannol standard was purchased from Sigma-Aldrich (SP, Brazil). For the evaluation of the antioxidant capacity, 2,2-diphenyl-1-picrylhydrazyl (DPPH), 6-hydroxy-2,5,7,8-etramethylchromane-2-carboxylic acid (Trolox), 2,4,6-tris(2-pyridyl)-s-triazine (TPTZ), 2,2′-azobis(2-methylpropionamidine) dihydrochloride (APPH) and fluorescein were purchased from Sigma-Aldrich (SP, Brazil).

2.2. Sample preparation

Passion fruit (*P. edulis* sp.) bagasse was provided by the company Sítio do Bello (Paraibuna — SP, Brazil). The bagasse was dried in an air circulation oven (Fanem, 320-SE, São Paulo, Brazil) for 30 h at 60 ± 2 °C. After drying, the passion fruit bagasse that presented moisture content of 0.035 ± 0.001 kg/kg on dry basis was ground in a knife mill (Marconi, MA340, Piracicaba, SP, Brazil), in order to homogenize and reduce the resistance to mass transfer during the extraction procedures.

The passion fruit bagasse was submitted to supercritical fluid extraction (SFE) using CO_2 as solvent in order to remove the nonpolar compounds from the raw material. The dynamic method was applied in SFE, which consists of continuous solvent flow through a fixed solid bed of sample particles placed inside the extraction column. Fig. 1 shows the extraction unit employed in this study, which was assembled in the laboratory of high pressure in food engineering (LAPEA, FEA, Unicamp, Brazil). For the SFE procedure, CO_2 was released from the cylinder, passed through a coil in a cooling bath (Marconi, MA-184, Piracicaba, Brazil) to be liquefied. Next, CO_2 was pumped (Maximator, M-111 CO_2, Nordhausen, Germany) to achieve the required pressure and passed through a coil in a heating bath (Marconi, MA-126, Piracicaba, Brazil) to reach the process temperature. Then the CO_2 flux entered the jacketed extraction column, flowed through the sample bed and dissolved the extractable compounds, left the bed and, after the depressurization in a micrometer valve (Autoclave Engineers, 10VRMM2812, Erie, USA) the extract was precipitated and CO_2 was quantified in a flow totalizer.

Approximately 40 g of sample was used, forming a fixed bed inside a 54.37 cm^3 (3.03 cm × 7.54 cm) stainless steel column. The SFE conditions were chosen in accordance to the results obtained in previous works, in which the best conditions to recover three distinct types of compounds (tocols, fatty acids, carotenoids) by a sequential process were identified (Viganó et al., 2016). The SFE process was divided into three sequential steps, the first was operated at 60 °C and 17 MPa, the second at 50 °C and 17 MPa and the third at 60 °C and 26 MPa. The

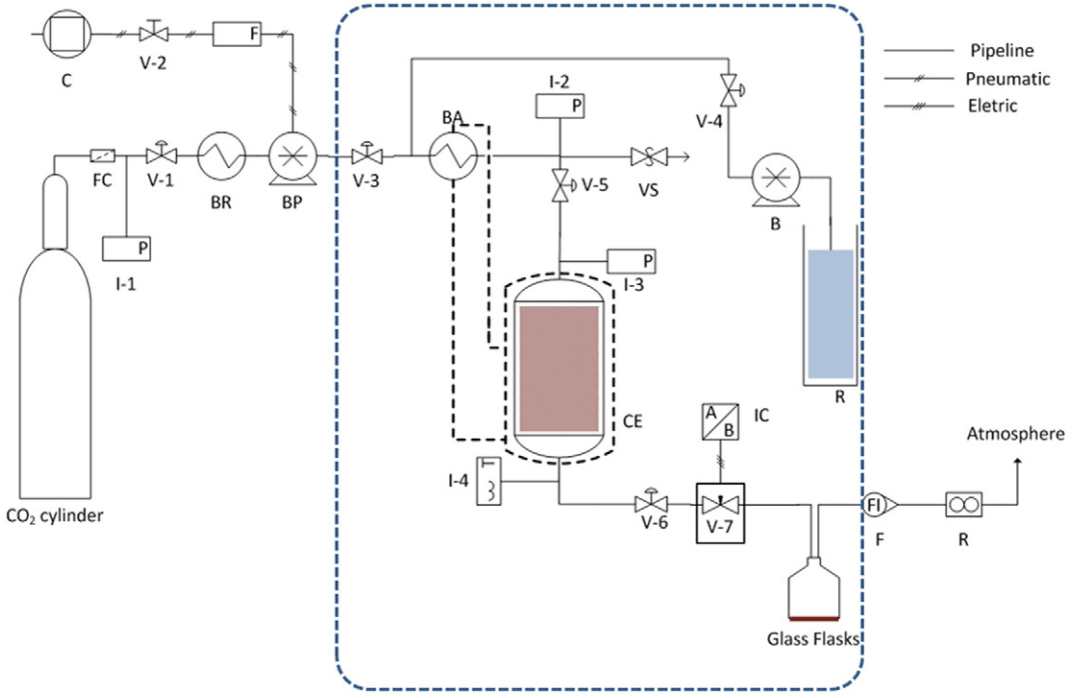

Fig. 1. Flowchart of the SFE/PLE extraction unit (Viganó et al., 2016); V-1 to V-6: block valves; V-7: micrometer valve; VS: safety valve; C: compressor; F: air filter; FC: CO_2 filter; BR: refrigeration bath; BP: CO_2 pump; BA: heating bath; CE: jacketed extraction column; R: solvent reservoir; B: HPLC pump; I-1, I-2 e I-3: pressure indicators; I-4: temperature indicators; IC: temperature controller; F: flow meter; R: flow totalizer.