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Simultaneous and fast separation of three chlorogenic acids and two flavonoids from bamboo leaves extracts using zirconia

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ABSTRACT

Phenolic acids and flavonoids in bamboo leaves are of great importance for their functional attributes, but they can hardly be separated simultaneously. In this study, zirconia was prepared and applied as a potential absorbent for simultaneous separation of these phenolic compounds. Three phenolic acids (neochlorogenic acid, chlorogenic acid and cryptochlorogenic acid) and two flavonoids (isoorientin and orientin) were isolated at the same time. The influence of bamboo leaves extraction conditions, zirconia calcination temperatures, desorption conditions and absorption/desorption dynamics on the separation were further investigated. When zirconia-400 (calcined at 400 °C) was treated with 70% ethanol extract of bamboo leaves for 40 min followed by desorption with 70% acetic acid solution for 60 min, the recovery of three chlorogenic acids and two flavonoids was about 65%. To conclude, the concise method developed here may provide a new way for simultaneous separation of phenolic acids and flavonoids from various plants.

1. Introduction

Bamboo (*Phyllostachys heterocycla* (Carr.) Mitford cv. *Pubescens* Mazel ex H.de leh.) is widely distributed in the mountainous areas of China, and their leaves are always used as an important herb in Chinese traditional medicine (e.g. the water extract of the leaves was used to treat fever of children) (Lu et al., 2005, 2006). Bamboo products hold an essential position in China and many parts of Asian countries like India as a healthy and nutritious food (Thomas et al., 2016; Wang et al., 2017). In the past decade, many functional foods have been developed from their shoots and leaves (Nirmala et al., 2014). The main functional constituents in bamboo leaves are phenolic acids and flavonoids (Guo et al., 2013; Liu et al., 2016; Shang et al., 2014), which have been proved to show various healthy functions as free radical scavenging, anti-oxidation, treatment of inflammatory bowel disease and diabetes, anticancer and antibacterial activities (Hu et al., 2000; Seki and Maeda, 2010; Sun et al., 2016; Yang et al., 2014). Purified bamboo leaves extracts rich in phenolic acids and flavonoids hence are considered as potential additives for functional foods (Lu et al., 2005; Zhang et al., 2007). However, the existing methods can be seldom applied to obtain this kind of extracts. Macroporous resin absorption is a common method to separate phenolics as flavonoids from bamboo leaves extracts (Yang et al., 2014; Zhang et al., 2005), but with this method, phenolic acids (e.g. chlorogenic acids) were lost in the step of water washing while removing water-soluble impurities, due to the weak interaction of phenolic acids with the resin (Zhang et al., 2008). Membrane filtration was also used to separate phenolics from plant extracts, but large amount of phenolic acids or flavonoid may be absorbed by the filter membrane (Ulbricht et al., 2009). Moreover, it is time-consuming to evaluate different membranes to remove various non-phenolic compounds. Therefore, method for simultaneous and fast separation of phenolic acids and flavonoids from bamboo leaves extracts is required.

Zirconia is a common inorganic material of amphoteric properties (Kassaye et al., 2016). Due to the specific surface properties, and improved chemical and thermal stabilities, it is widely applied in ion exchange, especially in sample preparation (Cacciola et al., 2007; Xu et al., 2016). Recent studies have revealed that zirconia displayed specific absorption toward carboxylates as humic acid and plasmids (nucleic acids) (Madiraju et al., 2000; Thuyavan et al., 2014). Phenolic acids and flavonoids in bamboo leaves extracts are rich in groups as carboxyl- and phenolic hydroxyl-, and therefore, they might be simultaneously isolated by zirconia.

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The aim of this study was to explore the potential of using zirconia as a novel absorbent to simultaneously separate phenolic acids and flavonoids from bamboo leaves extracts. Considering that the absorbent-based separation was affected by various factors (Ma et al., 2018), the effects of plant extraction solvents, zirconia calcination temperatures and desorption conditions, as well as absorption and desorption dynamics were also examined.

2. Materials and methods

2.1. Materials

Bamboo leaves collected from the local bamboo forest (Xuancheng, China) and dried under daylight for 2 days. Standard compounds as neochlorogenic acid, chlorogenic acid, cryptochlorogenic acid, isoorientin, orientin, vitexin and isoitexin were purchased from Nanjing Spring & Autumn Biological Engineering Co., Ltd. (Nanjing, China). Syringe filters (0.45 μm, 13 mm) were supplied by Pall (Beijing, China), and deionized water was obtained from a water purifier system (Millipore, Germany). All chemicals and solvents were of analytical or HPLC grade.

2.2. Synthesis and characterization of zirconia

Zirconia was prepared through a precipitation method using ammonia as precipitation agent. In a typical synthesis, to a clear solution of zirconium oxychloride octahydrate (0.4 mol/L), ammonia was added dropwise until pH = 10, and the mixture was stirred for another 1 h at room temperature. The precipitate was washed with deionized water and dried in an oven (110 °C), then calcined at 400 °C for 2 h to obtain a white powder, which was named zirconia-400. Zirconia dried or calcined under various temperatures was denoted as ZrP-n (n: temperature). The structures of these materials were then characterized by fourier-transform infrared spectroscopy (FT-IR, Agilent, Cary 630) or field emission scanning electron microscope (FE-SEM, Hitachi, SU8020). The FT-IR spectrum was recorded in the range 400–4000 cm^{-1} using a potassium bromide (KBr) method. Further, the scanning electron microscopy (SEM) (JSM-6490LV, Japan) images were recorded at an acceleration voltage of 5 kV.

2.3. Preparation of bamboo leaves extracts

Dried bamboo leaves were grounded into 40 meshes and extracted with water-ethanol solutions of different ratios for 1 h by an ultrasonic cleaner (45 kHz, 240 W). In a typical extraction, bamboo leaves (1 g) were mixed with 70% ethanol (v/v, 10 mL) and sonicated for 1 h, and the resulting extract was separated by a 0.45 μm filter and further analyzed with high performance liquid chromatography (HPLC) or Liquid chromatography–mass spectrometry (LC-MS).

2.4. Separation of chlorogenic acids and flavonoids

The general procedures for the isolation of chlorogenic acids and flavonoids from bamboo leaves extracts were as follows: the mixtures of zirconia and bamboo leaves extracts were shaken for several hours on a shaker (150 rpm, 37 °C). After removing the supernatants by centrifugation, zirconia was washed with deionized water, and subsequently mixed with acetic acid solution. The resulting mixtures were then shaken for several hours; finally, zirconia was removed by centrifugation, and desorption solutions were obtained. In a typical separation, the ratio of zirconia and bamboo leaves extract was 1:10 (0.1 g: 1 mL), and the shaking time for each section was 24 h. The zirconia was finally desorbed by 70% acetic acid (in water, v/v, 1 mL). To study the adsorption and desorption dynamics of chlorogenic acids and flavonoids on zirconia, the supernatants from each section were extracted at different time ranges. For the recovery study of chlorogenic acids and flavonoids, the final supernatants after adsorption or desorption were collected. Each experiment was repeated three times, and the corresponding samples were analyzed by HPLC.

2.5. HPLC and HPLC/MS analysis

The bamboo leaves extracts, adsorption supernatants and desorption solutions were analyzed on a reverse-phase high-performance liquid chromatographic (RP-HPLC) system (Agilent, 1220), which was equipped with HC-C_{18} reverse-phase column (250 × 4.6 mm, 5 μm, Agilent) and EZChrom Elite software (Agilent). The mobile phase consisted of a phosphoric acid solution (0.4% in water, v/v, solvent A) and acetonitrile (solvent B). The samples were eluted as follows: the solvent B linearly increased from 5% to 15% at 0–10 min, it was maintained at 15% at 10–20 min and finally it was increased from 15% to 40% at 20–30 min which was followed by decreased from 40% to 5% within 5 min. The flow rate was 1 mL/min, and UV detection was performed at 330 nm. LC-MS analysis was performed with Agilent 1260/6460 LC/MSD system; the mobile phase consisted of aqueous formic acid (0.8% in water, solvent A) and acetonitrile (solvent B), and the mass spectra were obtained using electro spray ionization in the negative ionization modes in the range of m/z 100–1000. The remaining conditions were identical to HPLC.

2.6. Data analysis

2.6.1. Relative content

To determine the content change of the main polyphenols in the bamboo leaves extracts (including neochlorogenic acid, chlorogenic acid, cryptochlorogenic acid, isoorientin, orientin, vitexin and isovitexint) and the main polyphenols in desorption solutions (including neochlorogenic acid, chlorogenic acid, cryptochlorogenic acid, isoorientin and orientin), the relative content was considered.

$$\text{Relative content of the main polyphenols (\%)} = S_T/S_M \times 100 \quad (1)$$

where S_T was the total HPLC peak area of the main polyphenols in bamboo leaves extracts or desorption solutions, and S_M was the maximum of that in the corresponding group.

2.6.2. Relative recovery

To estimate the separation efficiency of the main polyphenols under different conditions, the recovery of the main polyphenols was determined.

$$\text{Relative recovery (\%)} = S_D/(S_O\text{-}S_S) \times 100 \quad (2)$$

where S_D was the total HPLC peak area of the main polyphenols in the desorption solution, S_O was the corresponding total area in the original bamboo leaves extract, and S_S was the corresponding total area in the supernatant of bamboo leaves extract after adsorption.

2.6.3. Relative adsorption or desorption efficiency

To describe the general adsorption and desorption dynamics of the main polyphenols toward zirconia, the following expressions were used.

For adsorption:

$$\text{Relative adsorption efficiency (\%)} = S_A/S_O \times 100 \quad (3)$$

where S_A was the total HPLC peak area of the main polyphenols in the bamboo leaves extracts, which was extracted at different time points during the adsorption process, and S_O was the corresponding total peak area in the original bamboo leaves extract.

For desorption:

$$\text{Relative desorption efficiency (\%)} = S_B/S_D \times 100 \quad (4)$$

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