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Ultrasound assisted adsorption and desorption of blueberry anthocyanins using macroporous resins

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ABSTRACT

In this study, the impact of ultrasound on the adsorption and desorption features of blueberry anthocyanins on macroporous resins were studied. Sonication was performed at 106–279 W/L and 20–30 °C in a pulsed mode, respectively. Generally, ultrasound treatments within the selected experimental range enhanced the adsorption/desorption process of anthocyanins on macroporous resins. The recovery of blueberry anthocyanins after ultrasound-assisted adsorption/desorption process at 279 W/L and 20 °C was 82.12%, which was 52.84% higher than that obtained after adsorption/desorption with shaking at 100 rpm. Meanwhile, higher acoustic energy density (AED) levels and lower temperatures benefited the adsorption process through enhancing the adsorption capacity and shortening the equilibrium time, whereas higher temperatures promoted the desorption process. Furthermore, malvidin-3-galactoside had the highest adsorption and desorption capacities among all the studied monomeric anthocyanins. No organic acids and sugars were detected after adsorption/desorption processes, indicating the successful improvement of anthocyanin purification. Sonication mainly enhanced the adsorption process by means of strengthening the formation of hydrogen bond on resins surface and increasing their surface roughness. Overall, ultrasound can be an effective tool to improve the purification of anthocyanins using macroporous resins.

1. Introduction

Blueberry (*Vaccinium* L.) is a rich source of dietary bioactivities compounds, such as organic acids and polyphenols, especially anthocyanins. According to the regulation of USDA [1], it has a series of health-related benefits, such as anti-oxidant, anti-cancer, anti-atherosclerosis, and anti-cholesterol activities [2–4]. It has been listed as one of the five healthy fruits by United Nations Food and Agriculture Organization [5]. However, many researches indicated that a harsh stickiness problem appeared during blueberry dehydration processes because of the high contents of sugars and acids in berries, resulting in poor quality properties, low yield and high energy consumption [6,7]. Besides, it has been confirmed that phytochemicals extracted with low sugar and acid contents from berries were more effective and healthier than whole fruits for obese patients and diabetics [8]. Therefore, it is necessary to produce anthocyanin concentrates with few or no sugars and acids as a dietary supplement or health care product.

In previous studies, the concentration of sugar-free anthocyanins from berries has been investigated by high-speed counter-current chromatography [9], preparative high-performance liquid chromatography [10] and others. However, some insufficiencies still exist in these methods, such as time-consuming, expensive and unable to meet the need of industrial-scale production. Macroporous resins have attracted a lot of attention in concentration of anthocyanins with no sugars and acids from berry-related fruits due to their outstanding adsorption properties, such as high mechanical feature, low solvent consumption, good selectivity and low cost [11,12]. Generally, macroporous resins usually adsorb adsorbates through noncovalent bonding in aqueous solution. Next, adsorbates can be desorbed from the adsorbents using some organic solvents [13]. There are different types of macroporous resins that can be used for the adsorption of phenolics, including polar, non-polar and weak-polar ones. Among them, XAD-7HP resins had a satisfactory performance on adsorption and desorption of anthocyanins as a result of its low polarity and large surface area [11,14]. On the other hand, it is time-consuming for both adsorption and desorption processes to reach the equilibrium when it comes to use

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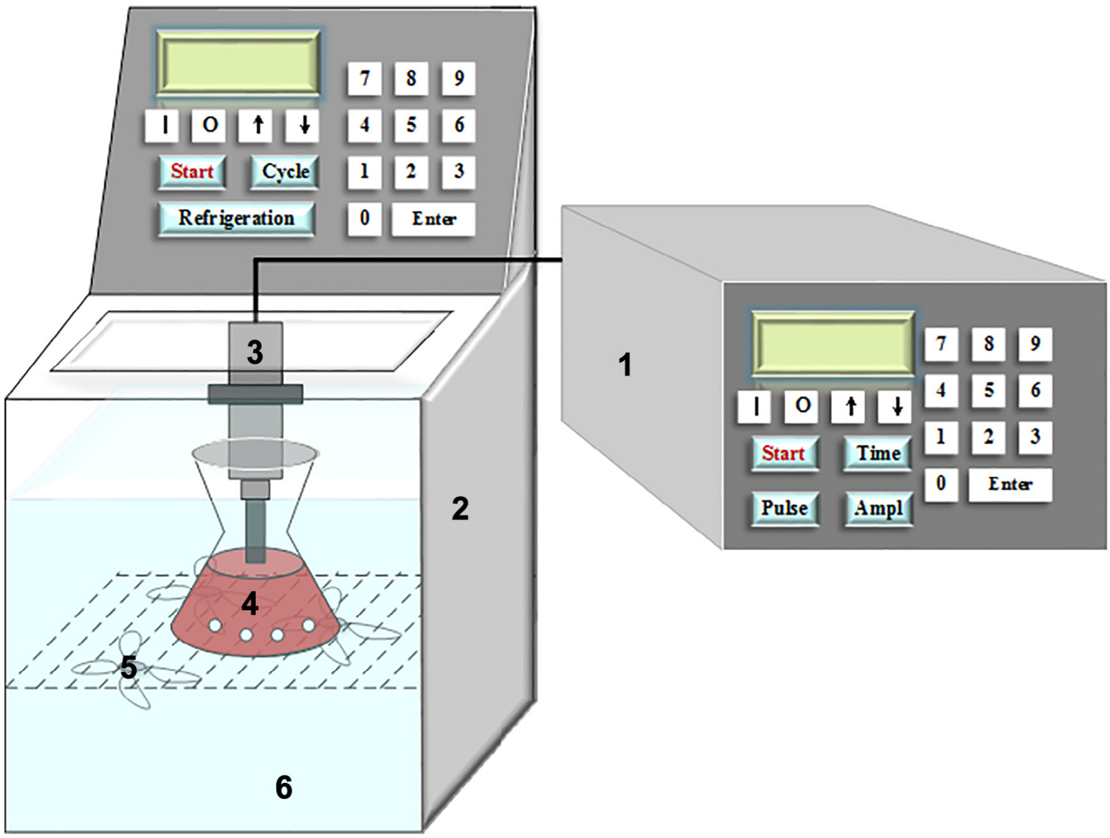

Fig. 1. Diagram of the experimental set-up for ultrasound-assisted adsorption/desorption of blueberry anthocyanins using macroporous resins. 1: Generator; 2: Electro-thermostatic Water Cabinet; 3: Ultrasonic probe; 4: Conical flask wrapped up in tinfoil containing blueberry extracts and resins; 5: Propeller; 6: Water.

marcoporous resins for anthocyanin purification [11,13]. Thus, it is of significance to develop novel technologies to enhance the anthocyanins adsorption/desorption processes. In the last decade, a series of studies have reported that ultrasound is an effective tool to assist the adsorption and desorption of target compounds using various materials as adsorbents [15–17].

The principles of ultrasound for the enhancement of adsorption/desorption process are its physical and cavitational properties. Ultrasound can enhance the mass transfer process from solutions to adsorbents and break the affinity between adsorbents and adsorbates by ultrasonic cavitation [18]. Hamdaoui et al. [19] reported that ultrasound improved the adsorption rate in the adsorption of *p*-chlorophenol with granular activated carbon. Jing et al. [17] found that ultrasound could both raise adsorption rate and shorten equilibrium time of chromium using polymeric resins, but it had no influence on desorption. Korkut et al. [20] reported that ultrasound promoted the desorption of Cu (II), but had an adverse impact on Pb (II) desorption. Karimi et al. [21] demonstrated that the impact of ultrasound on nitrate desorption was positive at low temperature. However, most studies of ultrasound-assisted adsorption/desorption were focused on the adsorption/desorption of metal ions and organic pollutants, which have not yet included adsorption/desorption of anthocyanins. Therefore, it is meaningful to study how ultrasound affects adsorption and desorption of anthocyanins using macroporous resins for the pigment purification.

This study aimed at studying the effects of different acoustic energy densities (AED, 106, 199 and 279 W/L) and temperatures (20 and 30 °C) on adsorption and desorption of anthocyanins using macroporous resins. The kinetic analysis of adsorption/desorption processes, component changes and resins characterization were investgated. This work can provide guidance to food industry for the separation and purification of anthocyanins pigments more efficiently.

2. Materials and methods

2.1. Materials

Fresh blueberries (*Vacciniumashei*) were provided by a local plantation in Lishui, Nanjing, China. After reaching the laboratory, blueberry samples were immediately stored at −18 °C, prior to use. XAD-7HP resins and standards of anthocyanins were purchased from Solarbio Technology Co., Ltd. (Shanghai, China). All other chemicals used were of analytical or chromatographic grade.

2.2. Preparation of macroporous resins

Before application, the XAD-7HP resins were pretreated following the method of Buran et al. [13]. Specifically, resins were filled in a glass column (I.D. × L: 30 × 150 mm) and then washed with 200 mL of 95% ethanol, followed by deionized water until eluate was clear. Next, 200 mL of 4% HCl was used for resin rinsing. After that, deionized water was utilized again to resins until neutral. 200 mL of 5% NaOH was further used for resins rinsing. Lastly, resins were rinsed by deionized water until the pH value of eluate reached 7.0.

The aforementioned resins were vacuum-dried at 60 °C and 0.1 MPa until constant weight was achieved.

2.3. Extraction of blueberry anthocyanins

Crude anthocyanin extracts were recovered from blueberries following the method of Buran et al. [13] with slight modifications. To be exacted, frozen blueberries were thawed at room temperature for 12 h. Next, thawed blueberries were crushed by a beater and mixed with distilled water containing 0.02% HCl at a ratio of 1:10 (g:mL). After that, the mixture was sonicated in an ultrasonic cell grinder (XO-