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Effects of ultrasound parameters on ultrasound-assisted ultrafiltration using cross-flow hollow fiber membrane for *Radix astragalus* extracts

Ming Cai [a,b,*], Wei Li [b], Hanhua Liang [b,**]

[a] Department of Food Science and Technology, Zhejiang University of Technology, Hangzhou, Zhejiang 310014, People's Republic of China
[b] Department of Applied Biology and Chemical Technology, The Hong Kong Polytechnic University, Hunghom, Kowloon, Hong Kong

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ABSTRACT

In this study, the effects of ultrasonic parameters on permeate flux and fouling resistances in the ultrasound-assisted ultrafiltration (UAUF) process of *Radix astragalus* extracts with hollow fiber membrane (HFM) was investigated. Ultrasonic parameters such as frequency, power and irradiation mode all can significantly affect the flux and fouling during the UAUF process, though the ultrasound power intensity was attenuated to be 10% only from generator. Concentration polarization and cake layer were decreased effectively by the ultrasound irradiation, resulting in a high flux performance. Meanwhile, HFM was susceptible to the irradiation at high power and low frequency compared with a flat sheet membrane. Ultrasound can not only enhance the permeate flux in UAUF but also cause breakage of HFM at an unsuited ultrasound field. It can be applied to the ultrafiltration process with HFM at an appropriate power and frequency.

1. Introduction

In recent decades, membrane techniques such as ultrafiltration (UF) and microfiltration (MF) have been used to clarify or separate the natural product mixtures in their manufacture processes [1–4]. However, membrane filtration is not widely applied to industries as expected due to its major drawback of membrane fouling, which reduces the membrane performance, especially in the permeate flux, thus adversely affect the attractiveness of the membrane filtration. Therefore, it would be desirable to apply alternative and effective methods to reduce the membrane fouling. Current membrane fouling control methods for UF or MF processes include vibration [5], gas sparging [6,7], backflushing [8,9], pulsatile flow [10] and electrical field [11]. All those techniques have some limitations. For example, gas sparging is time consuming due to the interruption of operation process for cleaning [12]; electrical techniques used for permeate flux enhancement may induce the electrolysis effect [13].

Ultrasound technique has been used in the studies to reduce membrane fouling in UF or MF process with flat sheet membrane [14]. Significant improvement in permeate flux in dead-end UF of dextran solution was obtained at a low frequency of ultrasound irradiation, and the enhancement was attributed to the hydrodynamic motions generated by ultrasound [15]. Ultrasound was also applied to a cross-flow flat sheet membrane filtration for water treatment by Kobayashi et al. [16,17], who found that all factors such as ultrasound frequency, power intensity and irradiation direction had significant effects on the filtration process. These effects of ultrasound on the membrane filtration process were mainly ascribed to its characteristics such as cavitations, acoustic streaming, micro streaming, etc. [18]. However, most of these studies focused on the dead-end or cross-flow flat sheet membrane filtration setups, and used only model solutions such as dextran and bovine serum albumin (BSA). In our previous studies, ultrasound was applied to a stirred dead-end UF process for a natural product extracts [19]. Our results showed that it was feasible to apply ultrasound to the UF process with more complicated systems such as natural plant extracts. Though cross-flow hollow fiber UF is wildly used in the industry, few studies have been carried out to introduce ultrasound technique into this model. Therefore, it is worthwhile to investigate the feasibility of applying ultrasound to the hollow fiber UF process for natural product separations.

Purpose of this work was to study the effects of various ultrasound parameters on the cross-flow UF process using hollow fiber membrane (HFM) for separation of desired components from *Radix astragalus* (*RA*) extracts. *RA* was selected as a target material

* Corresponding author at: Zhejiang University of Technology, Food Science and Technology, No.18 Chao Wang Road, Hangzhou, People's Republic of China. Tel.: +86 571 88320345; fax: +86 571 88320345.
** Corresponding author. Tel.: +852 34008663; fax: +852 23649932.
E-mail addresses: caiming@zjut.edu.cn (M. Cai), bchhliang@polyu.edu.hk (H. Liang).

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because it has a wide distribution of molecular weight and consists of polysaccharides, saponins and flavonoids, all of which have satisfactory medicinal activities [19]. Effects of ultrasound frequency, power and irradiation mode on the permeate flux and fouling in HFM system were investigated. Ultrasound power intensity inside the hollow fiber module was measured at different frequencies. The feasibility of introducing ultrasound to HFM and the enhancement of UF process was demonstrated.

2. Materials and methods

2.1. Materials

100 g pulverous *RA* was first extracted with 1 L DI water at 95–100 °C for 1 h, and the first extracts were collected. 0.8 L fresh DI water was added for another 1 h extraction. The combined extracts were cooled to room temperature and then filtered by a filter paper (Whatman, UK) as a stock solution.

2.2. Ultrasound-assisted UF process

The experimental setup is shown in Fig. 1, which was also described by Cai et al. [20]. The hollow fiber module has a bundle of polysulfone (PS) membranes with 10 kDa molecular weight cut-off (MWCO) and total effective area of 0.015 m^2. The ultrasound plate can operate at frequency 45 kHz and variable output power up to 300 W with a 30 W interval or at 100 kHz and variable power up to 600 W with a 60 W interval, respectively. *RA* extracts were transmitted by a peristaltic pump (Masterflex, Cole-Parmer Ins., Co., USA) at a fixed flow rate of 40 mL/min. The system transmembrane pressure (TMP) was adjusted at 0.8 bar. The feed container was put on a magnetic/heat plate (Thermolyne Cimarec 1, Dubuque, USA) with a stirring bar inside. Permeate solution was collected and its weight was measured by an electronic balance (Ohaus, USA). All experiments were operated at a constant pressure and total recycle mode.

Flux reduction and flux enhancement are used as indexes to indicate the effects of ultrasound on the UF process. These two indexes are defined as follows:

$$\text{Fluxreduction} = \left(1 - \frac{\text{flux}_{i-\text{end}}}{\text{flux}_{\text{initial}}}\right) \times 100\% \quad (1)$$

$$\text{Fluxenhancement} = \left(\frac{\text{flux}_{i-\text{end}}}{\text{flux}_{\text{without}}} - 1\right) \times 100\% \quad (2)$$

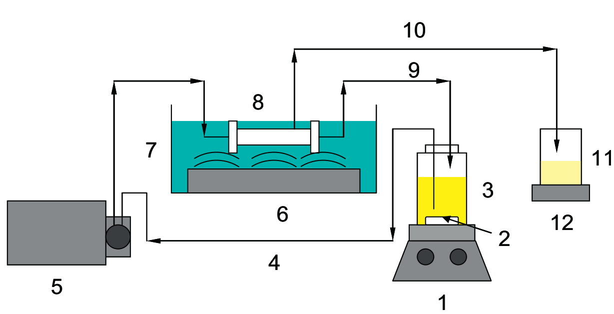

Fig. 1. Experimental set-up of ultrasound assisted hollow fiber ultrafiltration. (1) Magnetic stirring and heat plate; (2) stirring bar; (3) feed container; (4) feed; (5) peristaltic pump; (6) ultrasonic transducer plate; (7) water bath; (8) hollow fiber module; (9) retentate; (10) permeate; (11) permeate container; and (12) balance [20].

where $\text{flux}_{\text{initial}}$ is the flux at the initial 2 min, $\text{flux}_{i-\text{end}}$ is the flux at the end of the process under different ultrasound power irradiation that lasted for 1 h and $\text{flux}_{\text{without}}$ is the flux of a process without ultrasound irradiation.

2.3. Determination of fouling resistances

The permeate flux can be described according to Darcy's law [15]:

$$J = \frac{\Delta P}{\mu R_{\text{tot}}} \quad (3)$$

where J is the permeate flux (L/m^2 h), ΔP the TMP (bar), μ the viscosity of solution (bar h), and R_{tot} the total resistance (1/m).

The total resistance, R_{tot}, can be defined as follows:

$$R_{\text{tot}} = R_{\text{m}} + R_{\text{cp}} + R_{\text{cg}} + R_{\text{frev}} + R_{\text{firr}} \quad (4)$$

where R_{m} is the intrinsic membrane resistance (1/m), R_{cp} the concentration polarization (1/m), R_{cg} the cake/gel layer resistance (1/m), R_{frev} the reversible fouling that can be removed by chemical cleaning (1/m), R_{firr} the irreversible fouling that cannot be removed by chemical cleaning (1/m). Each resistance can be determined following the experimental procedures described by Jiraratananon and Chanachai [21] and Cassano et al. [22] with some modifications.

2.4. Determination of ultrasound intensity

Ultrasonic power intensity could be measured by a calorimetric method that monitors the temperature changes against time. Besides the calorimetric method, a sonic probe connected to a pulse receiver was used to estimate the ultrasound power intensity in the ultrasound-assisted filtration system [17]. It was found that the ultrasound power intensity inside the membrane unit would be attenuated sharply because the sound wave has to pass through the case which has very different impedance with the surrounding medium. In this study, a needle hydrophone and oscilloscope were used to measure the power intensity both inside and outside the HFM module which was put in a fixed position of a water bath. The needle hydrophone was positioned vertically into the module and its tip was immersed in water near the membrane. The waveform amplitude displayed on the oscilloscope was recorded. It was converted into the acoustic intensity, I, by the following equation:

$$I = \frac{P^2}{\rho c} \quad (5)$$

where P is the acoustic pressure (Pa), ρ the density of the propagating medium (kg/m^3), and c the velocity of sound in the propagating medium (m/s).

2.5. Scanning electron microscope (SEM) analysis

Hollow fiber UF membranes used at different experimental conditions were freeze-dried at −60 °C for 24 h, sputter-coated with gold, and then imaged using a SEM (JSM-6490, JEOL, USA).

3. Results and discussion

3.1. Ultrasonic power intensity

Fig. 2 shows the ultrasound waves at 45 kHz detected by the needle hydrophone in the ultrasound bath and inside the UF