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Optimization of ultrasonic-microwave assisted extraction of oligosaccharides from lotus (*Nelumbo nucifera* Gaertn.) seeds

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A B S T R A C T

The traditional technology replaced by ultrasonic or microwave assisted extraction has attracted considerable research interest. In order to maximize the yield of different degree polymerized oligosaccharides from lotus seeds, response surface methodology (RSM) was applied for optimization by ultrasound-microwave assisted extraction (UMAE) in this study. The results indicated that the optimal conditions for UMAE of lotus seed oligosaccharides were: extraction time 325.00 s, liquid-solid ratio 10.00 mL/g (L/S ratio), ultrasonic power 300.46 W, microwave power 250.00 W, and the yield of total oligosaccharides, trisaccharides, and tetrasaccharides was increased by 76.59%, 17.47%, and 27.21%, respectively. On the other hand, the extraction time was shortened by 12.18, 8.92, and 1.16 times, respectively, compared to the hot water extraction, ultrasonic-assisted extraction (UAE), and microwave-assisted extraction (MAE). These results confirmed that UMAE has a greater potential and efficiency for extraction of oligosaccharides than traditional hot water, ultrasonic or microwave assisted extraction alone.

1. Introduction

In the medicine industry, the extraction of bioactive substances is mainly based on the accurate solvent selection when heating or stirring. Microwaves are electromagnetic waves generated from the electric and magnetic field by the interaction of perpendicular oscillations, which interact with polar molecules to produce heat after penetrating the substance. Heating efficiency is dependent on the heating of raw materials, ion conduction, and dipolar rotation. Ion conduction is the process of kinetic energy conversion into thermal energy due to the dipolar molecule rotation to maintain similar electric field orientation when charge carriers of electrophoretic mobility have occurred under the influence of microwave (Mahesar et al., 2008). Microwave-assisted extraction (MAE) technology has been widely used in the study of medicinal plants to reduce the cost of consumption and optimal operational performance under normal pressure. The extraction process has a certain selective and targeted microwave extraction conditions which lead to enriching the carbohydrates at varied degrees (Coelho et al., 2014). Compared to MAE, the ultrasonic-assisted extraction (UAE) is more simplified and rapid. The mechanical effects generated by acoustic waves in UAE can enhance the ability of the solvent to penetrate into the porous surface of the cell to improve the mass transfer property. Additionally, micro-jet, high pressure, and high temperature caused by cavitation bubbles collapsed when biological cells ruptured to the plants matrix during the compression cycle. The physical forces include micro-jet force, shear force, shock waves, and turbulence (Chandrapala et al., 2012; Chemat and Khan, 2011). Consequently, it can release intracellular insoluble materials, reduce extraction time, and increase the yield under lower temperature (Awad et al., 2012). Contrastingly, UAE is not restricted by solvent, matrix or moisture content (Luque-Garcıa and Luque de Castro, 2003). Therefore, ultrasound-microwave assisted extraction (UMAE) not only appropriately combines the advantages of both technologies, but also compensates for each deficiency. High momentum and high energy to crush the plant cell are offered by UMAE resulting in solubilized active compounds into solvent that can effectively shorten the extraction time and reduce the loss of solvent. Currently, UMAE has been applied to extract various active compounds, such as lycopene (Zhang and Liu,

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2008), vegetable oil (Cravotto et al., 2008), and polysaccharides (Chen et al., 2010) from plants.

Functional oligosaccharides are important alternative regulators of the gastrointestinal tract seeking further consideration (Jovanovic-Malinovska et al., 2015). Lotus seeds are known as mature seeds of *Nelumbo nucifera* Gaertn., which have been extensively cultivated in Fujian, Jiangxi, Hubei, Jiangsu, and Taiwan, provinces of China, and classified as medicinal and edible by the health ministry of the nation. Oral ingestion of lotus seeds fermented milk in mice can accelerate the colon movement and reduce the intestinal fecal retention time, thereby indicating its association to several raffinose analogs (Wu et al., 2005). Lotus seed oligosaccharides (LOS) are mainly composed of disaccharides, trisaccharides, and tetrasaccharides, with glycosidic bonds comprising of Manp-(1→, Galp-(1→, α(1 → 6)-Glup and α(1 → 6)-Manp. LOS can promote the production of acetic acid, propionic acid, and butyric acid from *Bifidobacterium adolescentis* as well as digestive enzymes and also bile resistance (Lu et al., 2015; Zhang et al., 2015). Compared to the conventional extraction methods, UAE has been proved to be more efficient in the extraction of oligosaccharides from the fruits and vegetables and stimulate bifidobacteria proliferation by accelerating the hydrolysis of lactose in milk (Jovanovic-Malinovska et al., 2015; Nguyen et al., 2012). However, the effects of various UMAE process parameters for varying degrees of the yield of oligosaccharides have not been discussed. RSM analysis method has been applied in exploring the relationship between the target yield and independent variables in the extraction process, supporting multi-variable output results, in order to optimize the process; for example, guaranteed higher phenol contents, ABTS and DPPH radical scavenging potential under the selective optimal conditions (Xu et al., 2015). However, discernible optimal extraction conditions for the yield of each carbohydrate component has yet to be adopted for regular usage.

This study is aimed at applying UMAE to improve the yield of LOS, and combining RSM to investigate the effect of extraction time, ultrasonic power, microwave power, and liquid to solid ratio on the yield of total oligosaccharides, trisaccharides, tetrasaccharides, and interaction of each factor, as well as the range of optimum conditions with UMAE for the extraction yield of individual fractions. The results offer a theoretical reference for efficient extraction of oligosaccharides by UMAE.

2. Materials and methods

2.1. Plant material

The raw material for this experiment was quick-frozen fresh lotus seeds provided by a local company (Green Acres Food Co., Ltd, Fujian, China).

2.2. Ultrasound-microwave assisted extraction (UMAE) device

Operating principle of ultrasound-microwave assisted extraction device was shown in Fig. 1. The device was equipped with automated frequency search and lock, to maintain the maximum acoustical power while the property and viscosity of the reactants are altered. The precision of temperature probe was ≤ ± 0.2 °C, heterotype three-port glass flask for 250 mL was equipped for the tests with the ultrasonic generator probe directly dipped into the suspension (probe diameter: Φ18 mm) and the condensation pipe connected for backflow reaction to reduce the solvent loss. The device also harbored an open system, which enabled the microwave reaction to occur at atmospheric pressure. In comparison to the equipment used by Zhang and Liu (2008), this facility used the digital control low-frequency ultrasonic generator (25 KHz), which could produce lower frequency ultrasound, and the power could be changed during the extraction (0–1500 W). Moreover, the microwave power could also be set by the digital controller.

Absolute ultrasonic power *P* was measured when the device was switched to the single ultrasonic mode and proceed under relative heat-

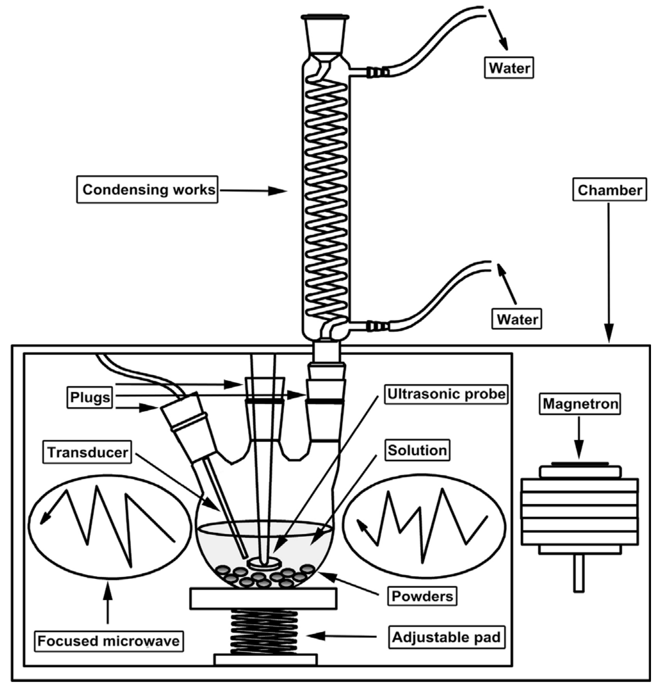

Fig. 1. Constructional drawing of ultrasonic-microwave assisted extraction device.

insulated conditions to calculate the practical ultrasonic power. *P* was adopted as an experimental parameter, and the output power of the equipment was adjusted to meet its requirements. Specific formula was shown in Eq. (12) (Sivakumar and Pandit, 2001),

$$P = mc_p(\frac{dT}{dt}) \quad (12)$$

m-the mass of extraction solvent (kg), c_p-thermal capacity of the solvent [J/(kg °C)], *T*-temperature of the extraction solvent (°C), *t*-time (s).

2.3. Selection of experimental factor

Independent variables and their scope were based on the results of the pretest. Because the smooth part in the curve of the single-factor tests would lead to reduced factor variability, the range of surface response tests at which every factor fluctuated significantly was determined to fit the response surface model. The device was simultaneously operated with ultrasonic and microwaves. Hence, both will generate thermal energy to heat sample thereby, positively correlating the power of ultrasonic and microwaves to time; temperature was not chosen as a factor for these tests. The temperature of water in the solvent could not remain constant before it reached 100 °C, following which it measurably evaporated and dissipated from the condensation pipe, thus changing the liquid-solid ratio. If the test temperature was constant before 100 °C, other design factors (microwave power) would change dynamically to maintain constancy. Therefore, it necessitated the maintenance of the liquid temperature at less than 100 °C. Ascertained level of the factors was shown in Table 2. Also, according to the results of the single-factor tests, the yield of lotus seed oligosaccharides was a non-linear trend for microwave causing a dramatic increase in the extraction solvent temperature when the liquid-solid ratio was too small. Thus, the liquid-solid ratio obligates at least 10 mL/g. The single-factor test (statistics are not given here) on ethanol concentration demonstrated that the yield of lotus seed oligosaccharides continually decreased when the ethanol concentration enhanced. This suggested that higher the concentration of ethanol, lower the solubility of the yield of lotus seed oligosaccharides. The result was consistent with those of the studies by Sen et al. (Sen et al., 2011). Although the solubility of oligosaccharides decreased, the purity in the solvent was