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Original research article

Extraction of gingerols and shogaols from ginger (*Zingiber officinale* Roscoe) through microwave technique using ionic liquids

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A R T I C L E I N F O

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A B S T R A C T

An ionic liquid-based microwave-assisted extraction (ILMAE) method was developed to extract 6-, 8-, 10-gingerols and 6-, 8-, and 10-shogaols from ginger (*Zingiber officinale* Roscoe). A series of water-miscible ionic liquids (ILs) with different kinds of anions and cations were investigated. Results indicated that among the investigated ILs, the carbon chains of alkyl cations remarkably affected the extraction efficiency of the six compounds and 1-decyl-3-methylimidazolium bromide ([C_{10}MIM]Br) was the optimal IL. The selected conditions for ILMAE were as follows: [C_{10}MIM]Br concentration, 0.80 M; extraction temperature, 75 °C; irradiation time, 30 min; and irradiation power, 400 W, in the case of solid–liquid ratio of 0.1:10 g/mL. This study showed that compared with the total extraction yield of 0.595 ± 0.031% by methanol marinated extraction and 0.673 ± 0.043% by methanol-based microwave-assisted extraction, ILMAE attained higher yield of 0.716 ± 0.051%; and ILMAE obtained a shorter time of 30 min for extraction of gingerols and shogaols from ginger. This study provides a basis for separating and purifying bioactive components from ginger. Moreover, the use of small amounts of [C_{10}MIM]Br as extraction solution in the method was considered environmentally friendly.

1. Introduction

Ginger, the dried rhizome of the plant *Zingiber officinale* Roscoe, is a widely used spice worldwide (Wen et al., 2014). Ginger mainly contains 6-, 8-, and 10-gingerols and 6-, 8-, and 10-shogaols (Varakumar et al., 2017). Fig. 1 shows the chemical structures of gingerols and shogaols. Many studies focus on determination of 6-, 8-, and 10-gingerols and 6-shogaol in ginger (Salmon et al., 2012; Eren and Betul, 2016). 8- and 10-shogaols, the corresponding dehydration products of 8- and 10-gingerols, possess higher bioactivities than those of their corresponding gingerols (Guo et al., 2014; Ho and Su, 2016). Therefore, determining all six compounds in ginger is necessary.

Ionic liquids (ILs) are novel solvents with excellent properties, such as environmental friendliness, excellent thermal stability, non-ionizing (e.g., non-polar) property, high viscosity, low combustibility, wide liquid region, and favorable solvating properties for a range of non-polar and polar chemical compounds; examples of ILs include some organic salts in the liquid state that consist of organic cations paired with organic or inorganic anions (Lemos and Oliveira, 2015; Yang and Dionysiou, 2004; Zhao et al., 2016). IL-based technologies can be used to enrich and separate a series of inorganic and organic compounds. These technologies are widely used in plant evaluation, food and biological analyses, environment monitoring, and other areas (Zhang et al., 2010; Liu et al., 2017). Scholars have reported the microwave-assisted extraction (MAE) of organic compounds directly from solid substrates (Kusuma and Mahfud, 2016; Liu et al., 2016; Zhao et al., 2016). Compared with traditional techniques, MAE is a more efficient and alternative approach for extraction of compounds. Conventional marinated extraction is widely used to extract gingerols and shogaols from dried ginger. However, this method is time consuming, laborious, and needs abundant, hazardous, and volatile organic solvents (Guo et al., 2014). To date, no research has focused on IL-based technologies for extraction of gingerols and shogaols from ginger.

In this study, an effective and rapid ionic liquids-based microwave-assisted extraction (ILMAE) approach was developed for extracting 6-, 8-, and 10-gingerols and 6-, 8-, and 10-shogaols from ginger. Five water-miscible ILs, including 1-hexyl-3-methylimidazolium bromide ([C_6MIM]Br), 1-octyl-3-methylimidazolium bromide ([C_8MIM]Br), 1-decyl-3-methylimidazolium bromide ([C_{10}MIM]Br), 1-decyl-3-methylimidazolium chloride ([C_{10}MIM]Cl), and 1-decyl-3-methylimidazolium toluenesulfonate ([C_{10}MIM]ToS), were selected as the extraction solvents for two reasons: 3-methylimidazolium ILs aqueous solution is

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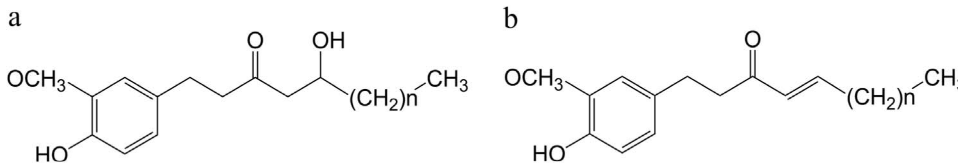

Fig. 1. Basic chemical structures of selected (a) gingerols (n = 2, 4, 6, 8, 10; 4-, 6-, 8-, 10-, and 12-gingerol) and (b) shogaols (n = 4, 6, 8; 6-, 8-, and 10-shogaol).

proven to be an attractive alternative to organic solvent for extracting bioactive components from plants; and ILs containing anions (e.g. ToS^-, Cl^-, and Br^-) are miscible with water (Liu et al., 2017; Zhang et al., 2014). The proposed method does not use traditional chemical reagents to extract gingerols and shogaols. Moreover, the use of small amounts of IL as extraction solution in the method was considered environmentally friendly.

2. Materials and methods

2.1. Reagents and apparatus

$[C_6MIM]Br$, $[C_8MIM]Br$, $[C_{10}MIM]Br$, $[C_{10}MIM]Cl$, and $[C_{10}MIM]ToS$ were obtained from Cheng Jie Chemical Co., Ltd. (Shanghai, China) and used as extraction solvents. Standards (purity > 99%) of 6-, 8-, and 10-gingerols and 6-, 8-, and 10-shogaols were purchased from Chromadex Inc. (Irvine, CA). All the chemicals used were of analytical or HPLC grade. Distilled deionized water or ultrapure water was used throughout the study. Samples were weighed using a XS-204 Mettler Toledo analytical electrical balance (Greifensee, Switzerland). Extraction was performed using an MDS-6 temperature self-controlled microwave decomposition system (Shanghai, China).

2.2. Sample preparation

Fresh ginger (*Zingiber officinale* Roscoe) samples were purchased from Linfen, Shanxi, China, rinsed clean, and cut into pieces. The pieces were powdered after air drying at 55 °C for 24 h in an oven. The dried ginger powder was stored in a moisture-controlled cabinet.

2.3. ILMAE

Fig. 2 shows a diagram of the microwave equipment used in this work. Accurately weighed ginger samples (0.1 ± 0.0001 g) and IL aqueous solution (10 mL) were placed in 50 mL polytetrafluoroethylene (PTFE) tubes. The vessel was sealed with a lid and placed into a polyetheretherketone (PEEK) bomb jacket, which was tightly fixed by hand on a bracket with a stainless-steel screw cap. The vessel was then placed in a temperature self-controlled microwave decomposition system. Pressure changes during microwave heating were detected using a pressure transducer. The obtained extracts were cooled to room temperature, quantitatively transferred to a 10-mL volumetric flask, and filtered through a 0.22-μm micronylon filter before HPLC analysis.

2.3.1. Selection of different ILs

Five water-miscible ILs with three kinds of anions and cations, i.e., $[C_6MIM]Br$, $[C_8MIM]Br$, $[C_{10}MIM]Br$, $[C_{10}MIM]Cl$, and $[C_{10}MIM]ToS$, were studied to investigate the effects of ILs with different anions and cations on extraction efficiency. The concentration of each IL aqueous solutions was set to 0.80 M. The microwave temperature, irradiation time, and power were set to 70 °C, 30 min, and 400 W, respectively.

2.3.2. Single factor extractions

Different $[C_{10}MIM]Br$ concentrations, ranging from 0.50 M to 1.00 M, were studied to determine the optimum $[C_{10}MIM]Br$ concentration for ILMAE of the six compounds. The temperature, irradiation time, and power were set to 70 °C, 30 min, and 400 W, respectively, with solid–liquid ratio of 0.1:10 g/mL.

Different extraction temperatures of 50 °C, 60 °C, 70 °C, 80 °C, and 90 °C were applied to determine the effect of temperature on the extraction yield of the analytes. The other extraction parameters were 0.80 M $[C_{10}MIM]Br$ aqueous solution, 30 min extraction time, and 400 W microwave power with a solid–liquid ratio of 0.1:10 g/mL.

Radiation durations of 15, 20, 25, 30, and 35 min were also investigated. The other extraction parameters were set to 0.80 M $[C_{10}MIM]Br$ aqueous solution, 70 °C extraction temperature, and 400 W microwave power, with a solid–liquid ratio of 0.1:10 g/mL.

The radiation power was tested at the following levels: 400, 600, 800, and 1000 W. The other extraction parameters were 0.80 M $[C_{10}MIM]Br$ aqueous solution, 70 °C extraction temperature, and 30 min radiation time with solid–liquid ratio of 0.1:10 g/mL.

2.3.3. Optimization of ILMAE conditions

Response surface methodology (RSM) is a tool for optimization in analytical chemistry (Bezerra et al., 2008). Based on a series of single-factor experimental results, three major factors and their appropriate ranges were finally determined for a three-level three-factor Box-Behnken design (BBD). In order to facilitate RSM analysis, the response value was expressed in the total extraction yield of the six major bioactive components of ginger, including 6-, 8-, and 10-gingerols and 6-, 8-, and 10-shogaols. The software Design Expert version 8.06 (Minneapolis MN) including built-in analysis of variance (ANOVA)

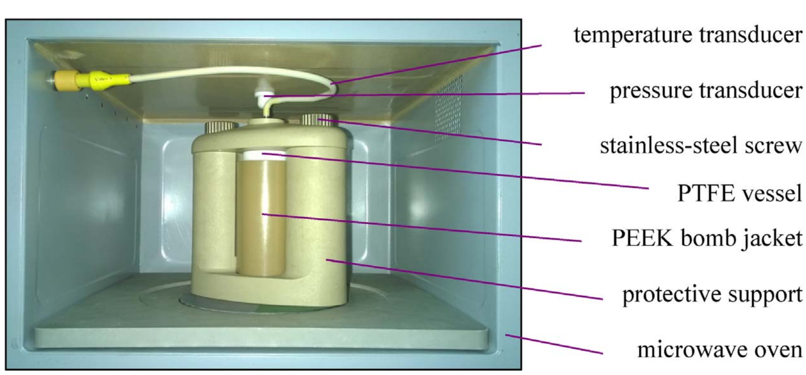

Fig. 2. Diagram of microwave equipment.Abbreviations: PTFE—polytetrafluoroethylene; PEEK—polyetheretherketone.