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Study of two-stage microwave extraction of essential oil and pectin from pomelo peels

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ABSTRACT

Pomelo peels were first processed by a solvent-free microwave extraction (SFME) for essential oils, then by a hot-solvent microwave extraction (HSME) for pectin. SFME was superior to the conventional hydrodistillation (HD) method for essential oil extraction and HSME was better than acidic solution method for pectin extraction in terms of extraction efficiency and yield of targeted component. Chemical composition analysis by GC–MS showed that SFME did not affect the quality of essential oils. By using the response surface methodology, the optimal conditions of HSME for pectin was found at microwave power of 520 W, solvent pH value of 1.5 and extraction time of 5.6 min. Surface view by optical microscope (OM) and cross sectional view by scanning electron microscope (SEM) of the peels suggested that microwave can enhance the extraction process by two distinct mechanisms: one attributes to the diffusion across the intact oil gland while the other involves the convection through the broken oil gland.

1. Introduction

The pomelo (*Citrus grandis*), which commonly grows in southern China, is the largest and juiciest fruit of the citrus family. It contains large amounts of carotenoids, vitamin E, flavonoids, limonoids, phenolic compounds, polysaccharides, lignin, fiber, pectin, essential oils, and is very popular in Southeast Asia. Essential oils in pomelo peels are responsible for the typical citrus-like aroma of the fruit (Saikaew, Kaewsarn, & Saikaew, 2009). As abundant sources of terpenoids, essential oils are made up of a mixture of many volatile compounds (Baser & Buchbauer, 2010). They are obtained as by-products of citrus processing and have widespread applications around the world as aroma flavor in many food products and in the pharmaceutical industry (Başer & Demirci, 2007). Pomelo peels are also rich in pectin. As a group of complex polysaccharides, pectin is localized in the middle lamella, intercellular crevices, and primary cell walls, and is known for the possession of pharmacological, hypoglycemic, and cholesterol-lowering effects (Francis, 2000). Pectin isolated from pomelo peels, based on their methoxyl content and degree of esterification, can be classified as low methoxyl pectin and has been applied in the manufacture of low-sugar products such as low-sugar jam and jelly (Norziah, Fang, & Abd Karim, 2000).

A traditional method of essential oil extraction is cold-press of the citrus peel (Bousbia, Vian, Ferhat, Meklati, & Chemat, 2009). Methods such as hydrodistillation (HD) and steam distillation are also popular for the extraction of volatile oils from plant materials (Lucchesi, Chemat, & Smadja, 2004). Conventionally, pectin is extracted in acidic solution at about 80–82 °C for 1 h with continuous stirring (Kratchanova, M., et al., 2004). However, these methods have disadvantages such as losses of some volatile compounds, low yield and degradation of target compounds (Lucchesi et al., 2004). Microwave extraction, a relatively novel technique that combines microwave and traditional solvent extraction, has received increasing attention over the years due to its shorter extraction time, higher yields of a target compound and less solvent consumption (Vongsangnak, Gua, Chauvatcharin, & Zhong, 2004). Solvent-free microwave extraction (SFME), one of the microwave extraction methods, combines microwave heating and dry distillation and performs in an atmospheric condition without any addition of solvent or water required (Bayramoglu, Sahin, & Sumnu, 2008). On the other hand, HSME has been successfully applied for

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the extraction of pectin from apple pomace (Wang et al., 2007), orange peel (Liu, Wei, Guo, & Kennedy, 2006) and lime (Fishman, Chau, Hoagland, & Hotchkiss, 2006).

The objective of this work is to establish a two-stage microwave extraction process for pomelo peels and to study the effects of microwave power, temperature, extraction time and solvent pH on the yield of essential oil and pectin. The extraction efficiency of SFME for essential oil and HSME for pectin will be compared with that of the conventional extraction methods. The response surface methodology was applied to optimize the condition of HSME for pectin extraction. Structural changes of pomelo peels were observed by optical microscope (OM) and scanning electron microscope (SEM) after the processes of SFME and HSME. The mechanisms of mass transfer in microwave extraction were also discussed.

2. Materials and methods

2.1. Plant materials and reagents

Fresh Shatin pomelos (Teaka) were purchased from a local supermarket, then washed and peeled manually. The peel obtained was about 40 g/100 g whole fruit weight. The peel materials were cut into pieces of approximately 5 × 5 × 10 mm for immediate used in the subsequent experiments. The initial moisture content of the peels was 80.7 g/100 g total weight, as determined by a freeze-drying method (Chåfer, Gonzålez-Martînez, Chiralt, & Fito, 2003) at −60 °C in a freeze-dryer (Alpha 1-4 LD2 freeze dryer, Germany). All reagents were of analytical grade and purchased from a local chemical provider (Advanced Technology & Industrial Co. LTD, Hong Kong).

2.2. Methods for the extraction of essential oils

According to the Chinese Pharmacopoeia (Tu, 1988), conventional HD was conducted at 100 °C for 180 min in a Clevenger apparatus equipped with a heating jacket. The ratio of peel material to water was 1:6. Essential oils were collected in amber-colored vials, then dried with anhydrous sodium sulfate, and finally stored at 4 °C for further analysis. The mean yield of essential oils was expressed as ml per 100 g of fresh peels ±S.D. (Standard Deviation). All experiments at each condition were conducted in triplicates.

SFME was performed using a focus microwave system, Model 961 (Microwave Power Consultants, VIC Australia). The power of the microwave reactor can be continuously varied up to 1000 W, and the system is equipped with a fiber-optical sensor for temperature control and measurement during extraction. Microwave is delivered from the generator to a cavity through a wave guide. The power modulation unit is equipped with the wave guide to modulate the output power levels. The power level reported in this study is the output power from the system. The same Clevenger apparatus used in HD was deployed here. 120 g fresh peels were treated during each trial without adding any solvent. Three microwave power levels, 150, 300 and 450 W, were selected to evaluate the effect of microwave energy on the extraction of essential oils. Extraction durations of 30 and 90 min were also used for studying the effect of extraction time on the yield of essential oils. The collection, treatment and storage of the obtained essential oils were the same as aforementioned HD process.

2.3. Methods for the extraction of pectin

The conventional hot solvent extraction for pectin was performed in a 500 mL flask by adding 10 g oil-free pomelo peels into 180 mL HCl aqueous solution at pH 2.0 and temperature 90 °C for 90 min. After extraction, the samples were hot-filtered and precipitated with 180 mL ethanol solution (95 ml/L) for 3 h. The coagulated pectin was separated and rinsed with ethanol solution (75 ml/L) and anhydrous ethanol. The treated samples were dried at 60 °C till constant weight. The pectin yield (%) was expressed in terms of gram of collected pectin per 100 g of oil-free pomelo peel.

HSME was conducted in a similar manner but performed in the same microwave system used for SFME excluding the Clevenger apparatus. In order to find the optimum condition of HSME, the response surface methodology (RSM) with the Box-Behnken experiment design was applied. Based on our previous works, only three major factors, microwave power, extraction time and solvent pH, were selected in this study. The low, middle and high levels were set as: 390, 520 and 650 W for microwave power; 3, 5 and 7 min for extraction time, and 1, 2, and 3 for solvent pH value, respectively. A total of 15 experimental trials, including 12 factorial points and three replicates at the center point, were conducted (Table 3). The pure error sum of squares was estimated by Design-Expert (Version 7.0, Stat-Ease, Inc., USA).

2.4. Gas chromatography-mass spectrometry identification

The composition of essential oils from the pomelo peels was analyzed by GC–MS (7890A GC system – 5975 C VLMSD with triple-axis detector, Agilent Technology, USA) equipped with a fused-silica capillary column HP-5MS (30 m × 0.25 mm × 0.25 μm). The GC conditions were: helium carrier (gas flow rate at 1.0 ml/min; split at 0:1; injection volume of 1.0 μl; injection temperature of 260 °C); oven temperature (programmed from 50 °C to 150 °C for 15 min, holding for 4 min, and 150 °C to 260 °C for 10 min, holding for 4 min); ionization mode (as electronic impact at 70 eV; ionization temperature of 230 °C); MSE quadruple temperature of 150 °C; transfer line temperature of 260 °C; and solvent delay for 3 min. The components of the extracted essential oils were identified by comparing their mass spectral fragmentation patterns with those stored in the MS database (National Institute of Standards and Technology 05 libraries).

2.5. Stability of essential oil

A stability study was performed in order to find out the effects of microwave irradiation on essential oil. The experiments were performed by treating 1.0 ml of essential oil extracted with HD method under various processing conditions as shown in Table 1. The amounts of essential oil before and after the treatment were measured and compared.

2.6. Morphological analysis

After processed by HD and SFME, the microstructure of the treated pomelo peel tissues was examined using optical microscope (OM) and scanning electron microscope (SEM).

For a surface view of the tissues, samples were sliced into extremely thin layers and observed directly by an optical microscope (Olympus IX17, Japan) with a magnification of 40. For a cross-sectional view, the specimens were freeze-dried in a freeze-dryer (Alpha 1-4 LD2 freeze dryer, Germany) for 2 days, then mounted on a specimen holder with tape and coated with Au using a sputtering coater (Gatan, model 861, Japan), and examined by a field-emission SEM (Model JEOL-JSM6335F, Japan).

2.7. Statistical analysis

Yields of essential oil and pectin in different extraction