



Sequential ultrasound-microwave assisted acid extraction (UMAE) of pectin from pomelo peels



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ABSTRACT

This study aims to optimize sequential ultrasound-microwave assisted extraction (UMAE) on pomelo peel using citric acid. The effects of pH, sonication time, microwave power and irradiation time on the yield and the degree of esterification (DE) of pectin were investigated. Under optimized conditions of pH 1.80, 27.52 min sonication followed by 6.40 min microwave irradiation at 643.44 W, the yield and the DE value of pectin obtained was respectively at 38.00% and 56.88%. Based upon optimized UMAE condition, the pectin from microwave-ultrasound assisted extraction (MUAE), ultrasound assisted extraction (UAE) and microwave assisted extraction (MAE) were studied. The yield of pectin adopting the UMAE was higher than all other techniques in the order of UMAE > MUAE > MAE > UAE. The pectin's galacturonic acid content obtained from combined extraction technique is higher than that obtained from sole extraction technique and the pectin gel produced from various techniques exhibited a pseudoplastic behaviour. The morphological structures of pectin extracted from MUAE and MAE closely resemble each other. The extracted pectin from UMAE with smaller and more regular surface differs greatly from that of UAE. This has substantiated the highest pectin yield of 36.33% from UMAE and further signified their compatibility and potentiality in pectin extraction.

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1. Introduction

Citrus peels are the most common commercial sources for pectin production [1]; attributing to the high pectin content of 25–30% in the dried peel mass [2]. Pomelo (*Citrus grandis* (L.) Osbeck) is a member of citrus family, and its peels have been reported to have natural chemical components, such as cellulose, flavonoids, essential oil, pectin and etc.; making it a good source for valuable extracts. The conventional acid extraction used for extracting pectin is time consuming, energy intensive, and thermally unsafe. The method also consumes high quantity of solvent and gives low efficiencies. Alternative non-conventional techniques like ultrasonic assisted and microwave assisted extraction have thus emerged to overcome the above mentioned limitations.

Ultrasound assisted extraction (UAE) is a simple, non-thermal, efficient and inexpensive technique that has been investigated by

many in the extraction of plant components. Extraction of pectin using ultrasound assisted extraction has been studied in different plant materials, such as tomato waste [3], grapefruit peel [4,5], sisal waste [6], pomegranate peel [7] and etc. The mechanism of UAE is based on sonication via induced micro streaming effect to propagate sound waves that create cavitations in the liquid solution (Fig. 1A). Following the subsequent collapse of the cavitations bubbles near the plant material surface, it results in an increase in pressure and temperature. These above phenomena destroy cell walls of the plant matrix and release extractive compounds into the solution. The full mechanism describing how the mechanical effects are created from ultrasonic activity can be found in articles [8,9].

Microwave assisted extraction (MAE) is another non-conventional heating method, which can directly extract plant constituents from different solid matrixes. It is very effective compared to conventional techniques and usually the extraction time varies only from a few seconds to less than an hour. Unlike X-rays and gamma rays, microwaves are non-ionizing radiations and they do not break chemical bonds or cause molecular changes in a compound by removal of electrons. The mechanism of microwave

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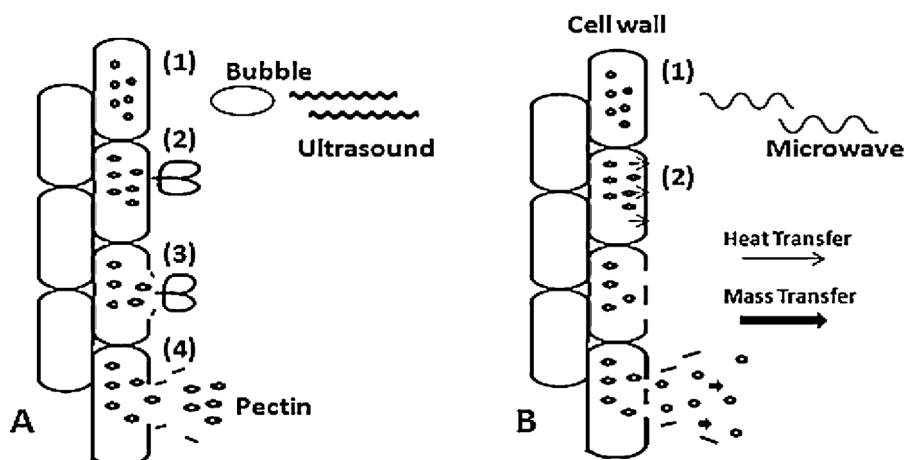


Fig. 1. Modified schematic diagram of basic (A) ultrasound and (B) microwave extraction mechanisms of natural products.

activity and a comprehensive study between MAE method and other non-conventional technologies are reported in reference [8]. During extraction, microwave energy causes the moisture inside cells to heat and evaporate which lead to a tremendous increase in pressure on the cell walls. As a result, it ruptures plant tissue and releases the targeted compounds into the surrounding solvent (Fig. 1B) [10]. This extraction technique has also been employed by many to extract bioactive compounds. In recent years, MAE has been successfully applied to pectin extraction from a wide variety of natural resources such as lime, orange peel and pomelo peel [1,11,12].

Ultrasound extractions are operated under non-thermal conditions while microwave radiation extractions involve short extraction time. Employing a sequential UMAE could potentially minimize or prevent the degradation of extract. It has been reported that the combined techniques resulted in higher yields and were found to be suitable for the extraction of thermally labile active compounds. The findings reported by Bagherian et al. [2] on grapefruit pectin extraction using ultrasound as a pre-treatment step for microwave extraction showed better results than employing only MAE. In the extraction of anthraquinones from *Heterophyllaeapustulata* Hook f. (Rubiaceae) by Barrera Vázquez et al. [13], the combination of ultrasound with microwave was proven to be an attractive extraction technique; exhibiting highest efficiency when compared with soxhlet and UAE. In terms of energy consumption (for the extraction of leave), the researchers reported that UMAE consumed about 1.45×10^7 kJ/gAQs of energy which is lesser than that of soxhlet (2.88×10^7 kJ/gAQs) while exhibiting similar consumption with UAE [13]. Despite recent investigations utilizing ultrasound with microwave assisted extraction technique on natural products, there is a lack of a comprehensive study on UMAE using citric acid in the extraction of pectin. The use of *Citrus grandis* (L.) Osbeck peel as the source of extraction has also not been reported.

In view of their respective mechanisms, combining UAE and MAE with suitable operating conditions might accelerate the extraction processes and would probably yield better quality pectin while avoiding possible thermal degradation. In the present study, UMAE was employed to extract pectin from *Citrus grandis* (L.) Osbeck with optimization conducted using a Box–Behnken design (BBD). Subsequently, pomelo pectin extraction performance was evaluated for sole UAE, sole MAE and MUAE using UMAE optimized conditions. The pomelo pectin extraction performance was ascertained via compositional properties, morphological structure and rheological analyses.

2. Materials and methods

2.1. Pectin extraction by sequential UMAE method and optimization study

2.1.1. Materials

Pomelo (*Citrus grandis* (L.) Osbeck) fruit of same batch and ripeness was obtained from Perak, Malaysia. The peels were cut and dried in a hot air oven (Memmert 600, Schwabach, Germany) at 60°C until a constant weight is attained. The dried peel was grinded and sieved into $250\text{ }\mu\text{m}$ – $355\text{ }\mu\text{m}$. The peel powder was stored in an air tight container and kept in dry condition prior to use. All solvents and chemicals used in this study were of analytical grade.

2.1.2. Sequential UMAE experimental method

Ten (10) g dried pomelo powder was weighed and mixed with 290 mL distilled water. The pH of the mixture solution was adjusted by using citric acid. The mixture solution was placed in an ultrasonic bath (Branson 3800, Danbury, USA) at 40 kHz for different sonication times. The ultrasound pre-treated mixture solution was then transferred to a microwave oven (ME711 K, Suwon, South Korea) and heated under different powers and irradiation times. The extraction was performed in triplicates.

2.1.3. Optimization study

Four factors, three levels (−1, 0, 1) Box–Behnken response surface design was employed to investigate and optimize the effect of process variables on the pectin yield and DE from pomelo peel, shown in Table 1. The variables considered were: pH (X_1), sonication time (X_2), microwave power (X_3) and irradiation time (X_4). The pH ranged from 1.7 to 2.3 with sonication time between 12 and 28 min, while microwave power was varied between 350 and 650 W and irradiation time between 4 and 12 min.

A total of 29 experiments including 5 centre points were designed ($N = 2K(K-1) + C_0$, where N is the total number of experiments, K is the number of independent variables and C_0 is the centre point). The statistical package Design Expert 6.0.6 (State-Ease Inc., Minneapolis, USA) was used to construct the experimental design and analysis of experimental data. The experimental data were fitted to a second-order polynomial equation to establish the relationship between independent variables and responses. The generalized form of the equation is:

$$Y = \beta_0 + \sum_{j=1}^k \beta_j X_j + \sum_{j=1}^k \beta_{jj} X_j^2 + \sum_i \sum_{j=2}^k \beta_{ij} X_i X_j \quad (1)$$

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