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New direction in research on extraction of *Citrus aurantifolia* (Lemon fruit) essential oil grown in Mekong Delta - Vietnam via microwave-assisted hydrodistillation

Thien Hien Tran[1,2], Van Tien Nguyen[2], Tan Phat Dao[1,2], Tri Duc Lam[1], Tran Quoc Toan[3], Trinh Duy Nguyen[1], Dai-Viet N. Vo[4], Tran Anh Vy[5], Le Minh Bui[1]

[1]NTT Hi-Tech Institute, Nguyen Tat Thanh University, Ho Chi Minh City, Vietnam
[2]Faculty of Chemical Engineering and Food Technology, Nguyen Tat Thanh University, Ho Chi Minh City, Vietnam
[3]Institute of Natural Products Chemistry, Vietnam Academy of Science and Technology, Ha Noi, Vietnam
[4]Faculty of Chemical and Natural Resources Engineering, Universiti Malaysia Pahang, Malaysia
[5]Department of Chemical and Biochemical Engineering, Gachon University, South Korea
*Corresponding author: labams2013@gmail.com

Abstract. A new research approach has been applied to the extraction of lemon essential oils to increase the extraction efficiency and improve the quality of isolated essential oils. In this study, the combination of microwave extraction method and response surface method is employed to investigate factors influencing lemon peel essential oil extraction process; including size of the raw material particles, raw material to water ratio, extraction time, and microwave power. The results showed that lemon peel of sizes 1 to 2 mm, material: solvent ratio 1:3 g/mL, extraction time of 60 min and microwave power of 450 W gave the highest yield of essential oil (2.4 %). The GC-MS results showed the domination of D-Limonene, the main ingredient of citrus essential oils, in higher concentrations than those reported by previous studies.

1. Introduction

The importance of the genus Citrus has been long recognized both commercially and scientifically. Among a wide variety of species belonging to the genus Citrus, *Citrus aurantifolia* (lemon fruit) offers a widespread coverage on many aspects including medicine, cultivation, industrial use and chemistry [1-4]. In addition to juice production, lemons could be used for the extraction of essential oils from its peel. The oil is a crucial and widely-used flavorant in medicines, cosmetics, beverages, household products, fragrances, and food. The composition of lemon essential oil is complex and its components are classified into three groups: oxygenates, terpenes and sesquiterpenes. The main compounds in oils are limonene, γ-terpinene, citral, linalool and β-caryophyllene.

The conventional methods of essential oil extraction from plant tissues include hydrodistillation, expression, simultaneous distillation extraction and solvent extraction [5-7]. However, these traditional techniques have several disadvantages regarding the extraction time, quantity of solvent, and oil recovery, rendering them particularly inefficient. The microwave processing technology has been applied in many areas of food processing such as pasteurization, cooking, reheating, drying, baking and thawing. This technique has gained great interest as a tool for synthesis and process development [8-11]. For isolation of target ingredients, particularly extraction of essential oil constituents, microwave heating has been widely used. In comparison to traditional extraction methods, microwave assisted extraction has many benefits such as higher yields of the isolated products, improved extract quality, shorter extraction time and reduced solvent consumption [11-16]. The aim of the present study was to investigate the applicability of microwave-assisted hydrodistillation for the extraction of essential oil from Vietnam *Citrus aurantifolia* (lemon fruit). Optimum values for the process variables, including the size of lemon peels, raw material - water ratio, extraction time, and microwave power were investigated to maximize essential oil yield.

2. Materials and Methods

2.1 Plant sample preparation

Citrus aurantifolia (lemon fruit) was collected from Tien Giang Province, Vietnam. The fruits were washed several times with water to remove impurities and dried. After that, the peels were separated from the sarcocarp. Prior to extraction, raw materials were kept in a non-hygroscopic bag and stored in a cooler (LC-1416B, Alaska, Vietnam) at temperatures below 10°C.

2.2 Extraction method

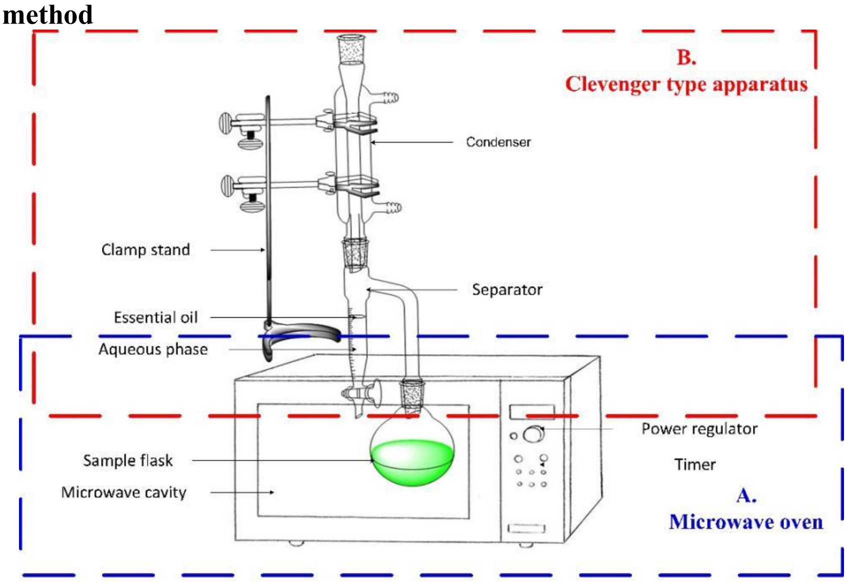

Figure 1. Schematics of microwave-assisted extraction of lemon essential oil

This method was conducted to individually investigate the factors affecting the yield obtained by the extraction process of lemon oils. These factors include the size of lemon peels (1-2 mm, 2-4 mm, and larger than 4 mm), raw material: water ratio (1:1, 1:2, 1:3 and 1:4 g/mL), extraction time (30, 45, 60, 74 and 90 min), microwave power (150, 300, 450 and 600 W). The yield of orange oil obtained (%) is calculated by quotient of the volume of attained oil (V, mL) and the amount of orange peel originally used for the experiment (m, g) were presented as in equation:

$$Y = \frac{V}{m} \times 100$$

Gas Chromatography-Mass Spectrometry (GC-MS) (GC Agilent 6890 N, MS 5973 inert) was used to analyze the components of essential oils. 25 µL sample of essential oil in 1.0 mL *n*-hexane and dehydrated with Na_2SO_4. . The used column was HP5-MS column with head column pressure of 9.3 psi. GC-MS were obtained under the following conditions: carrier gas He; flow rate 1.0 mL/min; split 1:100; injection

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volume 1.0 µL; injection temperature 250°C; oven temperature progress included an initial hold at 50°C for 2 min, a rise to 80°C at 2°C/min, a rise to 150°C at 5°C/min, a rise to 200°C at 10°C/min and a rise to 300°C at 20°C/min for 5 min.

3. Results and Discussion

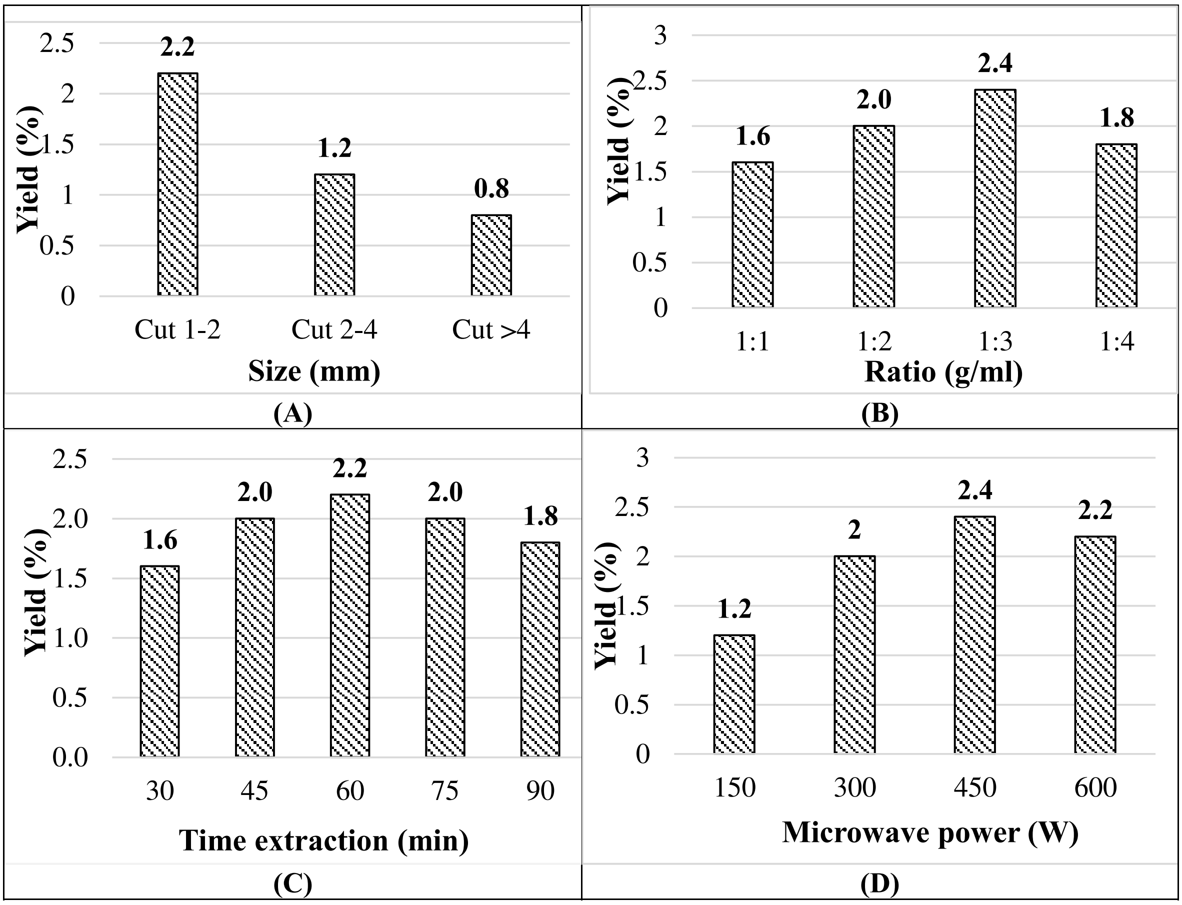

Figure 2. Factors affecting the yield of lemon oil extraction process by Single factor investigation method. The effect of the raw material particle size (A), raw material to water ratio (B), extraction time (C), the applied microwave power (D) on the yield of essential oils.

The factors that affect the yield of lemon oils in the extraction process are shown in Figure 2. Fig 1A illustrated that the yield of lemon essential oil gradually increased as the material size decreases. When the lemon peel size changes from particles larger than 4 mm to those of 1-2 mm, the essential oil yield increases from 0.8% to 2.2%. The reason for this is that by material cutting, the contact surface between water and material is extended, facilitating water diffusion into the essential oil bags, quickly pushing the oil outside under the effect of microwave. However, if the material is cut into very small pieces, more essential oil will be lost by the cutting process, finally resulting in reduced oil yield. Therefore, the best size of lemon peels was 1-2 mm.

In the second survey, the factor of raw material to water ratio was investigated. According to Figure 1B, by using more water, the diffusion of oil into the water is enhanced. The increase of yield of the extracted oil from 1.6% to 2.4% when increasing the raw material to water ratio from 1:1 to 1:3 was obtained. It is because the water could easily be absorbed by the material, promoting the extraction of soluble components which in turn causes higher oil yield. However, at ratio of 1:4, the essential oil content decreased to 1.8% since excessive water will dissolve or emulsify the essential oil. So, the ratio of raw material to water of 1:3 g/mL gave the best results (yield of 2.4%).

Similarly, Figure 2C and 2D demonstrated the effects of extraction time and microwave power on the yield of extracted lemon oils. When the extraction time prolonged, the efficiency of the extracted oil gradually increases from 1.6% at 30 min to 2.4% at 60 min. However, yield decreased to 1.8% at 90 min because of denaturation of certain substances due to prolonged exposure to high temperature. Figure 2D demonstrated that the higher microwave power leads to better yield of the oil but only to a certain limit of power level. It is because higher temperatures, caused by increased microwave power, incite the movement of the constituents, subsequently raising the velocity of chaotic movement of components in the mixture. Therefore, the extraction time and the microwave power in the survey were chosen at 60 min and 450 W, respectively.

From Figure 3 it can be seen that D-Limonene was detected as the major constituent of the essential oil (71.898%), as expected. This substance is important as it possess numerous biological activities. D-Limonene content in present study is significantly higher than it is reported in previous studies [17,18]. Other identified components were *o*-Cymene, L-beta- pinene, and beta-bisabolene with corresponding levels of 13.765%, 5.221%, and 1.617%, respectively.

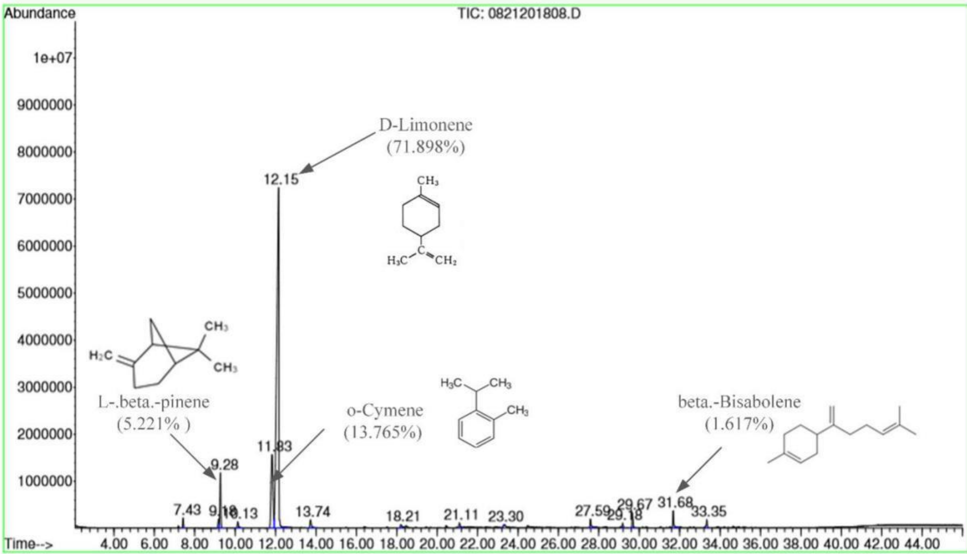

Figure 3. Chromatograph of lemon essential oil obtained by GC-MS with marked peaks of the main components and their chemical structures

4. Conclusion

To conclude, the parameters that affect the extraction of the lemon peel essential oil using microwaves during extraction were optimized. The obtained yield of lemon oil of 2.4% was obtained by use of lemon peel with particle sizes from 1 to 2 mm, water-to-material ratio 3:1 mL/g, at the extraction time of 60 min and microwave power of 450 W. The results of GC-MS analyses also revealed that the content of D-Limonene was the highest in samples produced using described parameters. The results showed that D-Limonene, a main compound of lemon peels essential oil can be effectively isolated by microwave technology what could be of great importance for food industry as this procedure is based on environmental green approach, allows time saving, and complete valorization of waste.

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