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Yield and Composition Analysis of Vietnamese Lemon (*Citrus aurantifolia*) Essential Oils Obtained from Hydrodistillation and Microwave-assisted Hydrodistillation

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Abstract. In this study, the essential oils from lemon (*Citrus aurantifolia*) peels were extracted using hydrodistillation and microwave-assisted hydrodistillation and their volatile compositions were compared via gas chromatography-mass spectrometry (GC-MS) analysis. Microwave-assisted hydrodistillation (MHD) achieved the oil yield of 2.48% at microwave power of 470W and extraction time of 60 min. The yield obtained using conventional hydrodistillation (HD) was 2.2% at 90 min at 120 °C. GC-MS analysis revealed a total of 17 and 8 components existing in the oil samples produced by MHD and HD methods respectively. The main components shared by both samples were limonene, α -pinene, β -pinene, β -cymene, ocimene, and sabinene. Overall, these results suggest that MHD could produce lemon (*Citrus aurantifolia*) peels essential oil at a higher yield within a shortened period of time as compared to the traditional HD, thus it could be used as an alternative for HD to reduce costs of the extraction process.

1. Introduction

Essential oils are natural products that are typically derived from flowers, herbs and other plant materials and are often composed of diverse and complex volatile mixtures of chemical compounds. It is estimated that the global number of essential oil-bearing plants amounts to about 300,000 and around ten percent of which is exploitable for commercial production of essential oils [1]. Among potential plant families applicable for essential oil production, the Citrus family of Rutaceae is promising but its potential has not been fully exploited and utilized. There have been many studies on citrus essential oils [2-7].

Citrus aurantifolia, or typically known as lemon, is a citrus plant that belongs to family Rutaceae and is widely grown in tropical and subtropical countries, especially in Vietnam where its geographical distribution mainly covers the plains and central regions. Lemon is primarily used as a food or food additive and is a common medicinal materials in folk medicine for treatment of fever, sore throat, cough, colds and indigestion [8]. The



lemon trees have small stems, thorny trunks, staggered leaves, serrated edges, and special aromas. Small flowers are white, growing solitarily among the leaves. The sphere is slightly flattened and has many seeds with the smooth or grainy skin. As the fruit ripens, its color turns from green to bright yellow. Despite that, green, unripe lemon is usually used. The lemon essential oil has been applied in many products, such as cosmetics, medical formulas, beverages, and foods, as flavoring agents as well as for flavoring. They are also used for bactericidal, antioxidant and anticancer properties [9-11]. It was found that lemon essential oil composition is dominated with terpene, aldehyde derivatives, alcohols, and ketones accumulating in different structures of the material [12-13]. To be specific, notable compounds of lemon essential oil consisted of Limonene, α -terpineol, terpinen-4-ol, 1,4-cineole, 1,8-cineole, α -pinene, p -cymene, and citral (geranial and neral) [14-16].

To obtain essential oils from *Citrus aurantifolia* peel, various methods such as HD, Ultrasound-Assisted Extraction, MHD, CO₂ supercritical extraction, steam distillation, maceration or expression could be adopted, depending on the plant materials [17-19]. The influence of time and temperature during the extraction of essential oils affects the yield of essential oils [20,21]. While older techniques such as expression or maceration may exhibit inherent limitations including longer extraction times, high solvent consumption and degradation of thermally sensitive compounds, the modern methods such as microwave assisted extraction have been shown to be economical and productive as well as being capable of improving product quality [22-24]. For this reason, this study selected MHD to compare with the conventional HD in terms of oil yield, as well as chemical composition, thereby determining an effective method for extraction of *Citrus aurantifolia* essential oil.

2. Materials and Methods

2.1. Plant materials

Fresh lemons are selected and collected from Tien Giang province (10 ° 25'13.04 " N, 106 ° 17'48.64 " E), Vietnam from March to April 2019. The lemon fruits were treated and cleaned, had peels removed. Raw materials were stored in a refrigerator under 10°C prior to extraction. The peels were then pureed and subjected to hydrodistillation or microwave assisted hydrodistillation.

2.2. Hydrodistillation (HD) of Lemon essential oil

HD extraction of lemon essential oil was performed using a Clevenger type instrument as described previously by Kosar et al. (2007) [25]. First, 100g of pureed lemon peels was placed into a 1000ml flask at lemon/water ratio of 1: 3. The flask was heated for 90 minutes and maintained at 120 °C. The extraction lasted until no essential oils was collected. The obtained mixture of water and essential oils was then dehydrated with sodium sulphate and stored in dark bottles. The hydrodistillation extraction process was demonstrated in Figure 1.

2.3. Microwave assisted hydrodistillation (MHD) of Lemon essential oil

MHD of lemon essential oil was based on the method previously described by Tran et al. (2007) [3]. The microwave oven used for extraction was supplied by Samsung (MW71E) and operated at 2450 MHz. The maximum capacity of the oven is specified at 800 W. The extraction system consisted of a microwave oven connected to Clevenger type apparatus where the flask was located in the oven cavity. The extraction process was carried out at a material/solvent ratio of 1: 3. A condensing device is placed on top, outside the oven, was used to collect the extracted essential oils. The microwave was heated for 60 min and operated at a fixed power of 470W. Obtained essential oil was collected, dried on anhydrous sodium sulfate and stored until use. The hydrodistillation extraction process was also demonstrated in Figure 1.

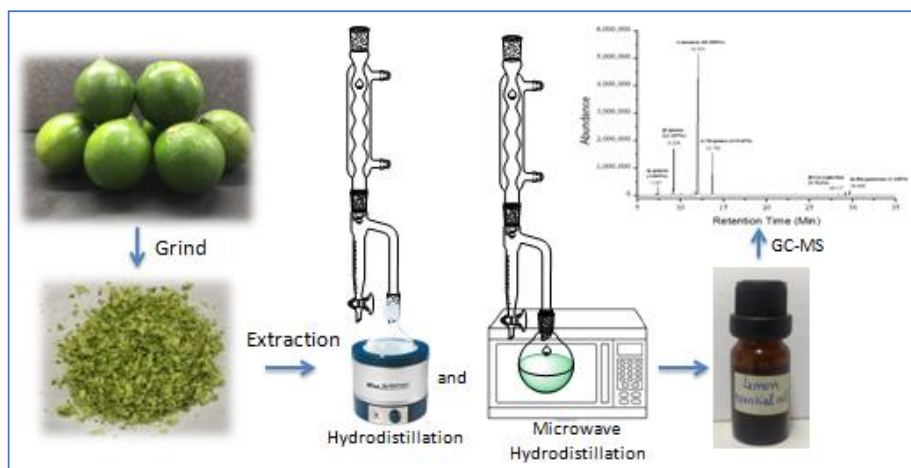


Figure 1. The process of extracting lemon essential oil

2.4. Gas chromatography-mass spectrometry analysis

Chemical composition of the lemon fruit peel essential oil was determined by GC-MS analysis using GC Agilent 6890 N instrument attached to HP5-MS column and MS 5973 inert [26]. Samples were added with n-hexane and dehydrated with Na_2SO_4 . The pressure of the head column was 9.3 psi. The flow rate was constant at 1 mL/min. The rate of division is 30 and the injector temperature is 250 °C. Thermal program is as follows. Commencing at 50 °C, this temperature was maintained for 2 min and then increased by 2 °C/min to 80 °C. Afterwards the temperature was elevated to 150 °C, to 200 °C and to 300 °C at the rate of 5 °C/min, 10 °C/min and 20 °C/min respectively. The last temperature (300 °C) was maintained for 5 min. The compounds were determined by comparing retention indices with the Wiley library or with published mass spectra (Web NIST, GMD).

3. Result and Discussion

Distillation to attract essential oils with steam is a common method used to separate mixtures such as those composed with water and essential oils. From the data of Table 1, it was found that the yield of lemon essential oil extracted by hydrodistillation was 2.2%. This yield was obtained under extraction temperature of 120 °C and the period of 90 min. However, using microwave-assisted distillation, the whole extraction process took only about 60 min at microwave power of 470 W to achieve the optimum performance of 2.48%. The shorter distillation time and improved extraction efficiency of the microwave-assisted distillation when compared with traditional hydrodistillation could be due to the effect of the microwave, which rapidly heats water in plant cells, in turn rises the internal pressure and ruptures the tissue containing essential oils [27]. The essential oil is therefore easier to be carried by steam. The microwave assisted extraction method is recommended for extraction of essential oils that are strongly characterized by a polarized composition. In general, time, temperature and power greatly affect the extraction process [28]. When the distillation time is short, essential oil may not be fully extracted. On the contrary, excessively long distillation time may dry the material, causing thermal damage to the oil and gives off unfavorable smell. In addition, prolonged microwave irradiation at high power could greatly accelerate the evaporation velocity, facilitating only partial vapor release and resulting in reduced extraction performance [29].

Table 1. The optimum parameters of extraction process of lemon essential oil by HD and MHD

Extraction methods	Peel/Water (g/mL)	Time (min)	Temperature (°C)	Power (W)
HD	1:3	90	120	-
MHD	1:3	60	-	470

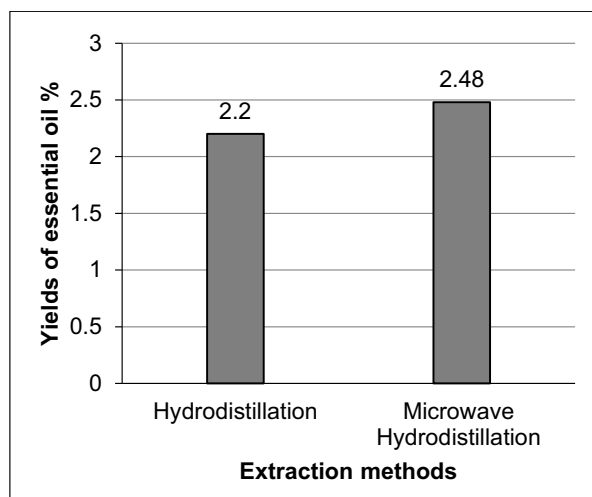


Figure 2. The performance of lemon essential oil extracted from HD and MHD

The total chromatography of lemon essential oil in HD and MHD is shown in Figure 2 and Table 1. Compositions of essential oils obtained from both methods are found to be qualitatively similar, albeit with some differences in quantity. Conventional HD of *C. aurantifolia* gave the essential oil sample that was constituted by about 17 major compounds, accounting for 99.999% of the total number of components. In the oil sample produced by microwave-assisted distillation, there were 8 compounds accounting for 97.247% of total compounds. From the results of the chemical composition of Table 2, it is shown that the essential oil obtained by HD has a higher hydrocarbon content than the essential oil sample obtained by MHD method. This is mostly due to the polar nature of hydrocarbons, causing them to be less affected by microwaves.

Table 2. Concentrations of the Lemon essential oil compounds obtained by different methods

Compound	Hydrodistillation (%)	Microwave Hydrodistillation (%)
α -Thujene	0.422	-
α -Pinene	1.855	0.807
Sabinene	1.448	1.617
β -pinene	11.287	5.221
β -Myrcene	0.778	-
α -Terpinen	0.258	-
β -Cymene	1.503	13.765
Limonene	65.988	71.898
γ -Terpinen	12.943	-
Ocimene	-	1.039
α -Terpieolen	0.369	-
α -Terpineol	0.375	-
β -Citral	0.696	-
β -Caryophyllen	0.711	-
α -Bergamotene	1.168	1.283
β -Elemen	0.198	-
α -Bisabolene	-	1.617
Total	99.999	97.247

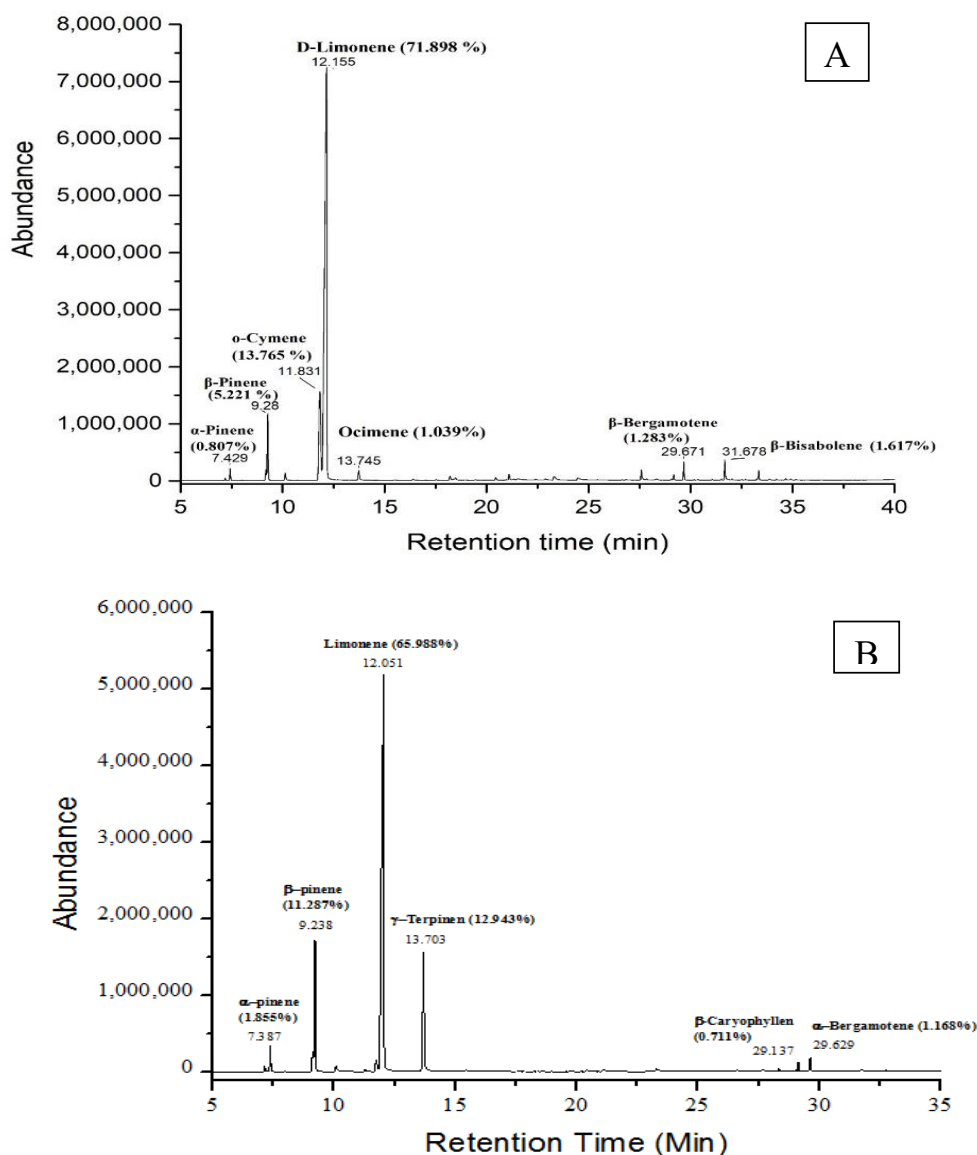


Figure 3. The result of chromatography of lemon essential oil (A) MHD and (B) HD

The compounds identified in lemon essential oil obtained in the both methods are mainly hydrocarbon monoterpene, accounting for most of the compound content in lemon essential oil. An oxygenated compound (α -Terpineol 0.375%) was detected in the essential oil sample extracted using HD. However, limonene content of the essential oil sample obtained from HD is slightly lower than that of microwave irradiation, possibly because of the oxidization of limonene during storage and distillation. Both methods produced essential oils with some major, shared compounds including limonene (65.988% and 71.989%), α -pinene (1.855% and 0.807%), β -pinene (11.287% and 5.221%), β -cymene (1.503% and 13.765%), α -bergamotene (1.168% and 1.283%) and sabinene (1.448% and 1.617%). The current composition shows stark quantitative difference from results of Asnaashari et al. (2010) where the main ingredients were limonene (28.27%), α -terpineol (19.61%), p-cymene (8.6%) and β -pinene (5.7%) [20]. However, comparing with other published results of *C. aurantifolia* essential oils, our

compositions are qualitatively similar [13], suggesting that the quantitative differences could originate from variations in growing conditions [30]. On the other hand, Jantan et al. (1996) conducted an analysis on a sample of lemon essential oil from Malaysia and obtained the composition that is different from this study with Limonene (16.41%), neral (11.43%), nerol (9.54%), and geranial (19.42%) as main components [31]. This result supports the explanation in which growing habitat could play an important role in determining essential oil composition of the plant.

4. Conclusion

Yields and volatile compositions of lemon essential oils obtained by two different extraction methods were compared in this study. As compared with hydrodistillation, the microwave-assisted technique seemed to give higher oil yield (2.48%) in a shorter extraction time (60 min). GC-MS results show that the contents of important compounds, particularly limonene, present in lemon essential oils obtained from MHD were generally higher than HD. Therefore, MHD is a good substitute for extracting essential oils from lemon zest as it provides essential oils of similar quality to conventional HD while reducing the time and cost of the extraction process.

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