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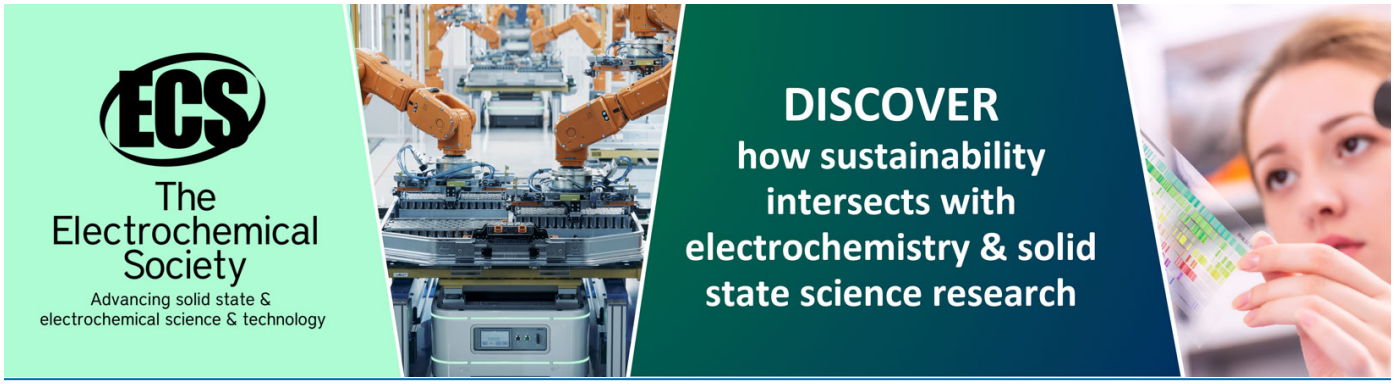

Pomelo (*Citrus maxima.* Merr) essential oil extraction by cold pressing and hydrodistillation methods and comparison of chemical composition

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Abstract. Essential oils extraction from the shell bring many applications in life, as well as contribute to the development of the food flavoring and cosmetic industry. Pomelo (*Citrus maxima* Merr) essential oil (EO) was extracted by cold pressing and hydrodistillation methods. The EO content obtained was 0.067% and 0.33%, respectively. The chemical composition was determined by the GC-MS analysis method, with 4 main components being D-Limonene, β-Myrcene, α-Pinene, and α-Phellandrene which exhibited high antibacterial and antioxidant capacity). The EO obtained from pomelo peels by cold pressing conditions was limited and less effective with more natural aroma, as compared to the hydrodistillation methods. Therefore, further research on EO pressing equipment and methods that can be used for effective oil extraction and recovery are necessary.

1. Introduction

Currently, fruit trees are widely employed as important materials for the transformation of crop structure, increase economic efficiency and improve the ecological environment [1–3]. Pomelo (*Citrus maxima* Merr) is one of the citrus fruit trees that are grown widely in our country as well as other Asian countries such as China, India, Thailand, Malaysia, and Philippines. Pomelo tree has brought high economic values to farmers. Pomelo EO has a characteristic aroma, high limonene content, and is widely used in food and cosmetics. Various parts of pomelo have been used, such as flowers, leaves, peels, and even seeds. Specifically, the outer rind is rich in naringin, which gives rise to the bitter taste. The essential oil (EO), which is present in pomelo peel at a level of 0.8 - 0.84%, contains d-limonene, α-pinene, linalool, geraniol, and citral [4–7]. The characteristic aroma constituents belong to the aldehyde and alcohol functional groups, which exhibit strong antibacterial ability against *Staphylococcus aureus*, *Escherichia coli*, *Salmonella typhimurium*, and *Bacillus cereus*, and also antioxidant, anti-inflammatory, anti-cancer and prevents cardiovascular diseases [8–10].

One of the common methods of EO separation is steam distillation. If the substances in the EO are decomposed by steam distillation, extraction with organic solvents (eg, kerosene ether, benzene, etc.) is

used. New and modern extraction methods are increasingly being developed to improve the extraction efficiency and quality of essential oils including supercritical extraction (CO_2 and H_2O), ultrasonic combined extraction, and microwave combined extraction [11–14]. However, the cold pressing method is widely used to extract EO from citrus peels. The resulted cold-pressed EOs usually have a fresh aroma and a visually clear color. Furthermore, as the pomelo peels are often discarded after the peels collection, utilization of pomelo peels for EO extraction and applications can be considered as a potential research direction to diversify the products with high nutritional values and minimize agricultural wastage and environmental problems. Results from the present study are the essential knowledge for developing effective extraction process and the chemical composition of pomelo EO for potential applications in the future. Therefore, the present study aimed to conduct preliminary assessment of chemical components in pomelo EO obtained from cold-pressing and hydrodistillation extraction method by using gas chromatographic-mass spectrometry (GC-MS).

2. Materials and methods

2.1. Plant materials

Healthy and mature pomelo (20 kg) was harvested in a local orchard in in Mekong Delta, Vietnam. The harvested pomelo was washed several times with tap water to remove the impurities, then was peeled and removed the inner pulp.

2.2. Cold-pressing method

A total of 4.6 kg of raw green-skinned pomelo peel was obtained, then placed into the screw press with a speed of 35 hertz (Hz) and resulted in 1.785 L of juice containing EO. The obtained juice was allowed to settle overnight at a temperature below 10 °C and separated into two different parts. To recover the EO, the extract was centrifuged at 6000 rpm for 5 minutes. The EO was dehydrated by using anhydrous Na_2SO_4, then stored in a freezer at 4 °C for further analysis as show that Figure 1.

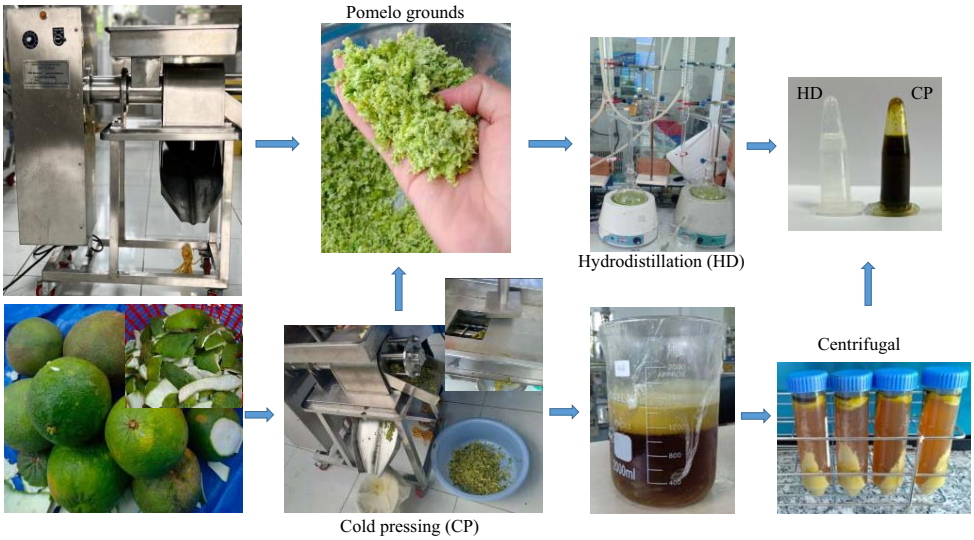

Figure 1. Pomelo essential oil extraction process by cold pressing and hydrodistillation

Pomelo grounds after pressing continues to be distilled by hydrodistillation, weighing each bottle 150g ± 0.02g residue. Distillation for 2 hours. Continuous distillation for 2 hours. Then, the obtained essential oil was purified by water removal Na_2SO_4.

2.3. GC-MS Analysis

GC-MS method by GC Agilent 6890N, Inert MS 5973 (USA) was considered to be the most effective in analyze the components of the pomelo EO. First, 25 µL of EO was added to 1.0 mL of n-hexane (Sigma - Aldrich), followed by dehydration by using Na_2SO_4. The head pressure is 9.3 psi. GC-MS was performed such as He carrier gas; injection temperature 250 °C; flow rate 1.0 mL/min; injection volume 1.0 µL; division 1:100; oven temperature progression 50 °C for 2 minutes, increased to 80 °C at 2 °C/min, then to 150 °C at 5 °C/min, to 200 °C at 10 ° C/min and finally maintained at 300 °C at 20 °C/min for 5 min.

3. Results and discussion

3.1. Chemical composition of EO obtained from cold-pressing

The EO obtained from cold-pressing method displayed a more natural greenish-black color and aroma than other methods, since the extraction process occurs at room temperature. The yield of EO obtained from 4.6 kg of raw pomelo peel was 0.067% with a density of 0.8520 (g/mL). Ferhat et al. (2016) [15] there are also studies comparing different extraction methods of essential oils from citrus fruits: cold pressing, hydrogen distillation and microwave distillation. Obtaining microwave results offers important advantages over traditional alternatives, shorter extraction times (30 minutes compared to 3 hours for hydrogen distillation and 1 hour for cold pressing); better performance (0.24% vs 0.21% for HD and 0.05% for CP). Furthermore, the content and stability of EO chemical constituents is dependent on various factors, such as variety, genetics, parts of the plant, soil, fertilizer, weather, light, and time of harvesting. Therefore, the determine of the time is an important factor to collection for the maximum oil content and the quality of EO [16].

The chemical composition of the extracted pomelo EO obtained from pressing method was determined by the GC-MS method. Results in Table 1 and Figure 2 have demonstrated 4 main components of the pomelo EO, including D-limonene (96.631%), β-myrcene (1.911%), α-pinene (0.763%), and α-phellandrene (0.694%). D-limonene is an important component of citrus EO that exhibits antifungal and insecticidal activities, thus it can be applied for agricultural pest control. In addition, D-limonene is also used in food and some medicines, such as flavoring to mask the bitterness of alkaloids, and as a fragrance in perfumes, aftershave lotions, shower gels and other personal care products [17].

In the investigation to determined the components by other research about EO from the rind and flowers of *C. maxima* Merr by cold pressing, hydrodistillation and supercritical CO_2 extraction (CO_2-SFC) techniques. The antimicrobial activity of EOs against different strains of microorganisms. The results have shown that the EOs extracted from the above method contained limonene, myrcene, pinene, sabinene, and linalool [18]. In addition, there are more than 200 volatile compounds present in pomelo EO obtained from cold pressing method, of which 22 substances contributed mainly to the oil aroma, such as 1,8-cineole, dodecanal, octanal, and nootkatone [19]. EOs contained in plants have a composition that is not fixed but always changes according to the plant growth at the same time as the influence of other factors. Each type of EO plant, in each part containing essential oil, has a certain composition under certain conditions, different varieties have different composition of essential oils. During the fruit growth, the composition of the EO undergoes numerous transformation based on which the time of maximum EO accummulation for harvesting and processing can be determined. This variation tends to be the accumulation of more and more oxygen-containing compounds (alcohols, aldehydes, ketones, esters) in

EOs, depending on the different essential oils, the formation and accumulation of chemical compounds will be different.

Table 1. Chemical composition of pomelo peel essential oil by cold pressing method

No.	R.T (min)	Compounds	(%)
1	8.080	α-Pinene	0.763
2	10.907	β-Myrcene	1.911
3	11.505	α-Phellandrene	0.694
4	13.009	D-Limonene	96.631

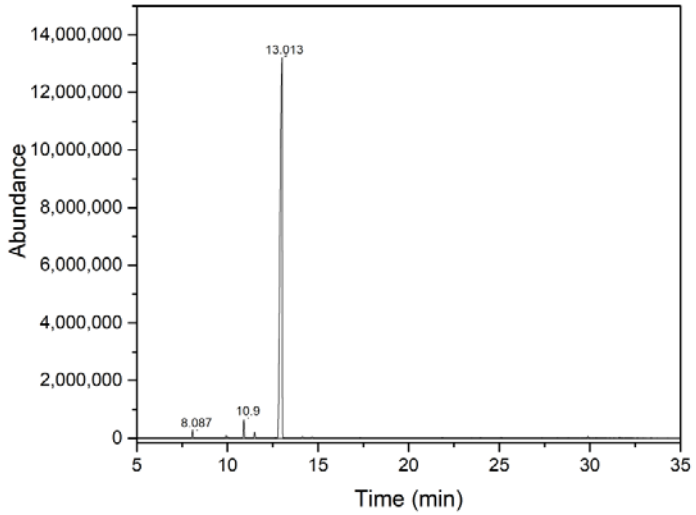

Figure 2. Chromatography of EO from pomelo peel extraction by cold pressing method

3.2. Hydrodistillation process of pomelo grounds from the cold pressing process

After 2 hours of extraction, the yield of EO obtained by hydrodistillation method of directly pressed grounds with water is 0.33%. The chemical composition of pomelo grounds EO, after being directly distilled with water, also has the same components as the essential oil obtained by mechanical pressing, including α-pinene (0.709%), β-myrcene (1.972%), α-phellandrene (0.716%), and d-limonene (96.603%) as Table 2 and Figure 3. The analytical results have shown that the volatile components in both cold-pressed and hydrodistilled EOs. However, as compared to the cold-pressed EO, the distilled EO has an unfavorable scent and transparent white color. When [20] compared the limonene content of EO from supercritical carbon dioxide extraction, vacuum steam distillation and cold-pressing. Results have shown that limonene was the major component in the EO from all methods (93.4–95.4%).

In addition, the *C. maxima* distributed in India was used to extract EOs by hydrodistillation [21], resulting in an extraction efficiency of 7.3 mL/kg. The main chemical constituents in the EO analyzed by GC-MS were specifically Z-citral (13.38%), 1-hexene-4-methyl (15.22%), E-citral (17.75%), and D-limonene (31.83%). In addition, EO obtained by steam distillation is purer than EO extracted with volatile solvents, since other impurities will dissolve into the water or compounds will be mixed with the color and

aroma of the essential oil, or cold pressing method will not be possible with awood and flowers EOs, is not suitable for unsafe raw materials because all chemicals are soluble in essential oils.[22–25].

Table 2. Chemical components of pomelo peel EOs by hydrodistillation method

No.	R.T (min)	Compounds	(%)
1	8.075	α-Pinene	0.709
2	10.903	β-Myrcene	1.972
3	11.502	α-Phellandrene	0.716
4	13.018	D-Limonene	96.603

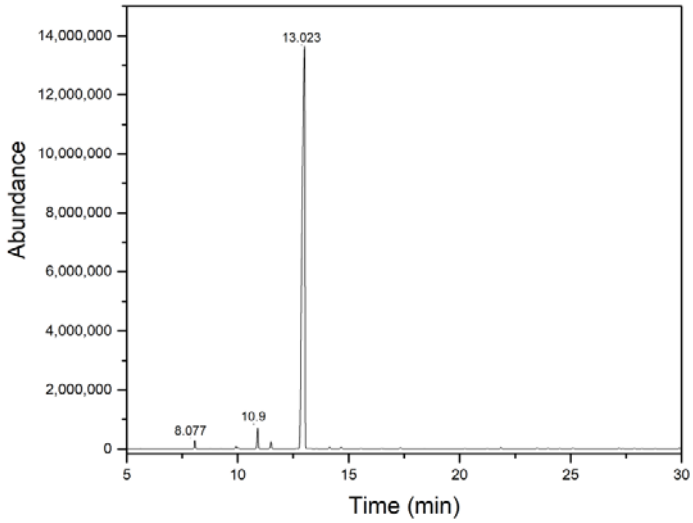

Figure 3. Chromatography of essential oil from pomelo grounds by hydrodistillation method

4. Conclusion

The present study investigated the extraction of volatile EO of green-skinned pomelo by cold-pressing and hydrodistillation and confirm the chemical compositions by GC-MS analysis. Results have shown that the chemical composition of the obtained EO was not significantly different, with four components being D-limonene, β-myrcene, α-pinene, and α-phellandrene. The content of EO after cold pressing and hydrodistillation extraction was 0.067%, and 0.33%, respectively , indicating that the EO was still remained after pressing. The results have shown that different extraction techniques can affect the scent and volatile components in pomelo peel EO.

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Acknowledgments. This research is funded by Nguyen Tat Thanh University, Ho Chi Minh City, Vietnam; Science and Technology Department of Ben Tre Province, Vietnam; Ministry of Science and Technology of Vietnam (Project No. 50/2020.NTMN.TW, 05/08/2020).