Comparative study of essential oils extracted from young and ripe pomelo (*Citrus maxima* (Burm.) Merr.) peels by steam distillation

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Abstract

This study investigated the essential oil yield and chemical composition of young and ripe pomelo (*Citrus maxima*) peels prepared in different physical forms. Using steam distillation and GC–MS analysis, the research found that ripe peels produced higher oil yields, while young peels showed consistently high purity of D-limonene. The chemical profiles of ripe peels were more diverse, containing additional monoterpenes and sesquiterpenes. These results demonstrate the potential of pomelo peel by-products for value-added applications in natural product industries.

The results showed that ripe pomelo peels consistently produced higher essential oil yields than young peels across all preparation methods. The highest yield (24 mL/300 g) was obtained from ripe dried and finely ground peels, while the lowest (3 mL/300 g) was recorded in young large fresh pieces. GC–MS analysis revealed that D-limonene was the predominant volatile compound in all samples, with relative abundances ranging from 62.07% to 100%. Young peels exhibited a simpler chemical profile characterized by high limonene purity, whereas ripe peels demonstrated greater chemical diversity, including β-myrcene, α-pinene, α-phellandrene, linalool, and other oxygenated monoterpenes and sesquiterpenes. Furthermore, storing ground peels for one day, even under refrigeration, resulted in a noticeable reduction in essential oil yield due to volatilization and oxidation.

These findings indicate that fruit maturity and physical preparation significantly influence essential oil characteristics. Young pomelo peels are suitable for producing high-limonene oils, whereas ripe peels provide more complex aromatic profiles. Overall, the study supports the value-added utilization of pomelo by-products and provides essential information for optimizing extraction processes in cosmetic, aromatherapy, and natural product applications.

Keywords: Pomelo peel; Essential oils; Steam distillation; D-limonene; GC–MS)

1. Introduction

Samut Songkhram Province is well known for cultivating *Citrus maxima* (Burm.) Merr. 'Khao Yai Ampawa,' a pomelo variety with a unique sweet flavor and high quality that has been granted the Geographical Indication (GI) certification. However, during cultivation and harvesting, large quantities of immature fruits and discarded peels from fresh pomelo markets become agricultural waste, creating environmental and economic concerns for local farmers. Utilizing these by-products for value-added purposes could serve as an effective approach to waste reduction and community income generation. The pomelo peel, particularly the green outer layer, is a rich source of essential oils containing bioactive compounds such as D-limonene, known for its antimicrobial, anti-inflammatory, and antioxidant properties. Extracting essential oils from pomelo peels, therefore, holds potential for developing various health and cosmetic products. Nevertheless, the characteristics of raw materials, including fruit maturity and physical form (e.g., large pieces, small pieces, finely ground, or dried powder), play a crucial role in determining both the yield and chemical composition of the extracted oils. Despite this importance, comparative studies investigating these factors remain limited.

Hence, this study aims to compare the essential oils extracted from young and ripe pomelo peels with different physical characteristics using steam distillation, and to analyze their chemical compositions using Gas Chromatography–Mass Spectrometry (GC–MS). The results will provide fundamental data to identify the most effective extraction conditions, supporting the development of high-quality essential oil products and the value-added utilization of pomelo by-products.

1.1 Research Objective

1) To investigate and compare the yield of essential oils extracted from young and mature pomelo (*Citrus maxima* (Burm.) Merr.) peels exhibiting different physical characteristics through the steam distillation technique.

2) To analyze and compare the chemical profiles of essential oils obtained from young and mature pomelo peels by employing Gas Chromatography–Mass Spectrometry (GC–MS).

2. Literature Review

Botanical Characteristics and Significance of Pomelo (*Citrus maxima* (Burm.) Merr.)

Pomelo (*Citrus maxima* (Burm.) Merr.) is a perennial fruit tree belonging to the family Rutaceae and is native to Southeast Asia. Botanically, it is a medium to large-sized tree, reaching a height of 5–15 meters, with thorny branches. The leaves are simple, alternately arranged, ovate to obovate, dark green, and glossy with a rounded or slightly pointed apex. The white flowers are fragrant and occur in clusters at the ends of branches. The fruit is large with a thick peel consisting of two main layers: the outer flavedo, light green to yellow in color, which contains numerous essential oil glands, and the inner albedo, a spongy white layer rich in dietary fiber. The pulp is divided into segments and has a sweet-to-sour taste depending on the cultivar and growing conditions.

In Thailand, pomelo is considered an economically important fruit crop. The "Khao Yai Amphawa" cultivar from Samut Songkhram Province is particularly renowned for its sweetness, pleasant aroma, and superior quality, earning a Geographical Indication (GI) certification from the Department of Intellectual Property. The cultivation area is in fertile alluvial plains at the river estuary, where slightly brackish water enhances the characteristic sweetness and flavor of the fruit. However, during the production process, a large quantity of immature fruits and discarded peels from fresh pomelo sales become agricultural waste. Utilizing these by-products for essential oil extraction represents a potential strategy for value addition, waste reduction, and community income generation (Ban Thip Suan Thong, 2016; Department of Agricultural Extension, 2021).

Essential oils are volatile organic compounds naturally synthesized and stored in specialized oil glands or tissues of plants, such as flowers, leaves, bark, and fruit peels. Chemically, they consist mainly of terpenes and terpenoids, which exhibit diverse biological activities including antibacterial, antifungal, anti-inflammatory, and antioxidant properties (Muyumba et al., 2021). Among citrus species, pomelo peel oil is particularly rich in D-limonene, which typically accounts for 65–95% of the total oil content, along with other key compounds such as β-myrcene, α-pinene, linalool, neral, and geranial, contributing to its characteristic aroma and pharmacological activities (Apirak, 2006).

Several studies have reported variations in the yield and chemical composition of pomelo essential oils among cultivars and fruit maturity stages. Che-Nurwati et al. (2004) extracted essential oils from the peels of Thong Dee and Khao Nam Phueng pomelo cultivars and found that the Thong Dee cultivar produced a higher oil yield and exhibited mosquito-repellent activity for up to 120 minutes, suggesting that varietal differences significantly influence oil quality. Similarly, Das et al. (2022) investigated essential oils from white and red pomelo varieties in Bangladesh using hydrodistillation and GC–MS analysis and reported that the white variety produced a higher yield (1.09%) with D-limonene as the major component (74%). These findings indicate that cultivar type and fruit maturity significantly affect both the chemical profile and quality of essential oils.

Therefore, the study of essential oils derived from pomelo peels is scientifically and economically important. It not only promotes the utilization of agricultural by-products but also provides fundamental information for optimizing extraction conditions and identifying quality determinants in natural product development. Comparing the yield and chemical composition of essential oils from young and mature pomelo peels can provide valuable insights for selecting suitable raw materials and improving extraction processes for future applications in cosmetics, pharmaceuticals, and health-related industries.

3. Methodology

3.1 Essential Oil Extraction from Young and Ripe Pomelo Peels

The extraction of essential oils from young and ripe pomelo (*Citrus maxima* (Burm.) Merr.) peels was performed using steam distillation in accordance with the Thai Herbal Pharmacopoeia (THP, 2023). Three different forms of peel samples, large fresh pieces, fresh ground peels (finely blended), and dried and finely ground peels, were prepared. For each peel type, 300 g of sample was accurately weighed and placed into a round-bottom flask connected to a Clevenger-type steam distillation apparatus.

Steam generated from boiling water was passed through the plant material for 6 hours, allowing the volatile constituents to be vaporized, condensed, and collected in the graduated receiver. The separated essential oil layer was removed carefully, dried over anhydrous sodium sulfate, and the final oil volume was measured.

All distillations were performed in triplicate (n = 3) for both young and ripe pomelo peel samples. The essential oil yield was calculated and expressed as milliliters per 100 grams of sample (mL/100 g) according to the THP standard. The oils obtained were stored in amber vials at 4 °C until further analysis.

3.2 Analysis of Chemical Composition of Essential Oils from Young and Ripe Pomelo Peels Using Gas Chromatography–Mass Spectrometry (GC–MS)

3.2.1 Sample Preparation

The test samples were prepared at a concentration of 10 mg/mL. Essential oil samples weighing 50 mg were accurately transferred into a test tube and dissolved in 3 mL of methanol and 2 mL of chloroform. The solution was mixed thoroughly until completely dissolved.

A 1 mL aliquot of the prepared solution was filtered through a syringe fitted with a membrane filter and then transferred into a Shimadzu LabTotal™ autosampler vial for GC–MS analysis (Palmieri et al., 2021).

3.2.2 Gas Chromatography–Mass Spectrometry (GC–MS) Analysis (Corrected Version)

GC–MS analysis was performed using a Shimadzu GC-MS-QP2020 system equipped with an HP-5MS capillary column (30 m × 0.25 mm i.d., 0.25 µm film thickness; 5% phenyl-methylpolysiloxane stationary phase). The operating conditions were as follows:

1) Carrier gas: Helium (purity ≥ 99.999%) was used as the carrier gas at a constant flow rate of 1.0 mL/min.

2) Injection conditions:
- Injection volume: 1 µL
- Injection mode: Split injection, split ratio 1:20
- Injector temperature: 250 °C

3) Oven temperature program:
- Start at 70 °C, hold for 2 min
- Increase at 5 °C/min to 200 °C, hold for 10 min
- Increase at 5 °C/min to 230 °C, hold for 10 min
- Increase at 5 °C/min to 250 °C, hold for 5 min
- Increase at 5 °C/min to 320 °C, hold for 20 min

4) Mass spectrometer conditions:
- Ionization mode: Electron Impact (EI), 70 eV
- Ion source temperature: 250 °C
- Interface (transfer line) temperature: 280 °C
- Mass scan range: m/z 35–500
- Acquisition mode: Scan

5) Data acquisition and identification:

Chromatograms were recorded as Total Ion Chromatograms (TICs). Volatile compounds were identified by comparing the obtained mass spectra with the NIST17 mass spectral library and confirmed by matching retention indices where available (Palmieri et al., 2021; Chokchaisiri et al., 2025; and Khophai et al., 2025).

3.3 Statistical Analysis

All experiments were conducted in triplicate (n = 3). The results were expressed as mean ($\bar{x}$) ± standard deviation (SD). Differences between groups were analyzed using one-way analysis of variance (ANOVA). A p-value < 0.05 was considered statistically significant. In addition, pairwise comparisons were performed using the independent t-test with a significance level of $p < 0.05$.

4. Results

The essential oil yields from young and ripe pomelo (*Citrus maxima*) peels prepared in different physical forms were determined using the steam distillation method. The results are presented in Tables 1 and 2.

The essential oil yields obtained from young pomelo peels prepared in three physical forms, large fresh pieces, fresh ground (finely blended), and dried and finely ground, are presented in Table 1. The dried and finely ground sample yielded the highest essential oil volume of 13 mL, indicating efficient oil release following moisture removal and particle size reduction. The fresh ground peel yielded 10 mL, showing moderate extraction efficiency due to increased surface area and ruptured oil glands. The large fresh pieces yielded the lowest volume at 3 mL, likely due to the intact peel structure and limited exposed oil glands. Table 2 shows the essential oil yields obtained from ripe pomelo peels prepared in large fresh pieces, fresh ground (finely blended), and dried and finely ground forms. The highest yield was obtained from the dried and finely ground sample, which produced 24 mL of essential oil. The fresh ground peel yielded 10 mL, while the large fresh pieces resulted in 5 mL of essential oil. Ripe peels demonstrated higher yields compared with young peels in all corresponding physical forms.

Table 1 Volume of Essential Oils Obtained from Steam Distillation of Young Pomelo Peels

No.	Physical Characteristics of Pomelo Peel		Weight (g)	Distillation Time (hrs.)	Essential Oil Volume (mL)	Remarks
01		Large fresh pieces	300	6	3	Contains intact oil glands; large particle size; single peel layer
02		Fresh ground (finely blended)	300	6	10	Increased surface area; more ruptured oil glands
03		Dried and finely ground	300	6	13	Moisture removed; highest oil release efficiency

Table 2 Volume of Essential Oils Obtained from Steam Distillation of Ripe Pomelo Peels

No.	Physical Characteristics of Pomelo Peel		Weight (g)	Distillation Time (hrs.)	Essential Oil Volume (mL)	Remarks
04		Large fresh pieces	300	6	5	Thick peel; intact oil glands; limited surface area
05		Fresh ground (finely blended)	300	6	10	Higher surface area promotes better oil release
06		Dried and finely ground	300	6	24	Most efficient form: moisture removed; maximum rupture of oil glands

4.1 Comparison of Young and Ripe Pomelo Peels

Based on the extraction results, ripe pomelo peels consistently produced higher essential oil volumes than young pomelo peels for all three preparation methods. The highest overall yield was obtained from ripe dried and finely ground peel (24 mL), whereas the lowest yield was observed in young large fresh pieces (3 mL). These findings suggest that both ripeness and physical preparation of the peel greatly influence extraction efficiency, with dried and finely ground peels offering the most effective recovery of essential oils.

4.2 Chemical Composition of Pomelo Essential Oils (GC–MS)

The chemical composition of essential oils extracted from both young and ripe pomelo (*Citrus maxima*) peels was analyzed using Gas Chromatography–Mass Spectrometry (GC–MS). The results revealed that D-Limonene was the predominant volatile compound in all samples, regardless of peel maturity or physical preparation. The percentage peak area of D-Limonene ranged from 62.07% to 100%, indicating that it is the major monoterpene responsible for the characteristic aroma profile of pomelo essential oils.

For young pomelo peels (01–04), D-Limonene content was consistently high and stable, ranging between 89.03% and 91.56% (Tab. 3). Minor constituents detected in young peels included β-Myrcene, α-Phellandrene, α-Pinene, 2-Furanmethanol derivatives, Caryophyllene, and Germacrene D, each present at low levels (<5%). Young peel samples generally exhibited less chemical diversity and a simpler volatile profile compared to ripe peels. In contrast, ripe pomelo peels (AN005–AN008) showed a wider variation in chemical composition. D-

Limonene levels varied markedly from 62.07% to 96.86%, with the highest purity observed in sample AN002, which contained only D-Limonene. Ripe samples also demonstrated greater diversity of secondary volatile compounds, including β-Myrcene, α-Pinene, α-Phellandrene, Linalool, Limonene oxide, Carveol, Carvone, and Germacrene D. The dried and finely ground ripe sample (04) exhibited the greatest chemical complexity, containing multiple oxygenated monoterpenes and sesquiterpenes.

Overall, the GC–MS analysis confirmed that D-Limonene is the dominant component in all pomelo peel essential oils, while the proportion and diversity of minor constituents depend on both peel maturity and physical form before extraction. Young peels produce essential oils with high limonene purity and low variability, whereas ripe peels yield a more chemically diverse oil profile due to advanced metabolic development and oxidative transformation of terpenes.

Table 3 Major Compounds Identified in Essential Oils from Young Pomelo Peels and Ripe *Pomelo Peels*

No.	Physical Form of Peel	Major Compound	%Area	Retention Time (min)
	Young Pomelo Peels			
01	Large fresh pieces	D-Limonene	91.56%	6.743
02	Fresh ground (finely blended)	D-Limonene	89.03%	6.728
03	Dried and finely ground	D-Limonene	90.30%	6.748
	Ripe Pomelo Peels			
04	Large fresh pieces	D-Limonene	84.74%	6.740
05	Fresh ground (finely blended)	D-Limonene	96.86%	6.704
06	Dried and finely ground	D-Limonene	62.07%	6.710

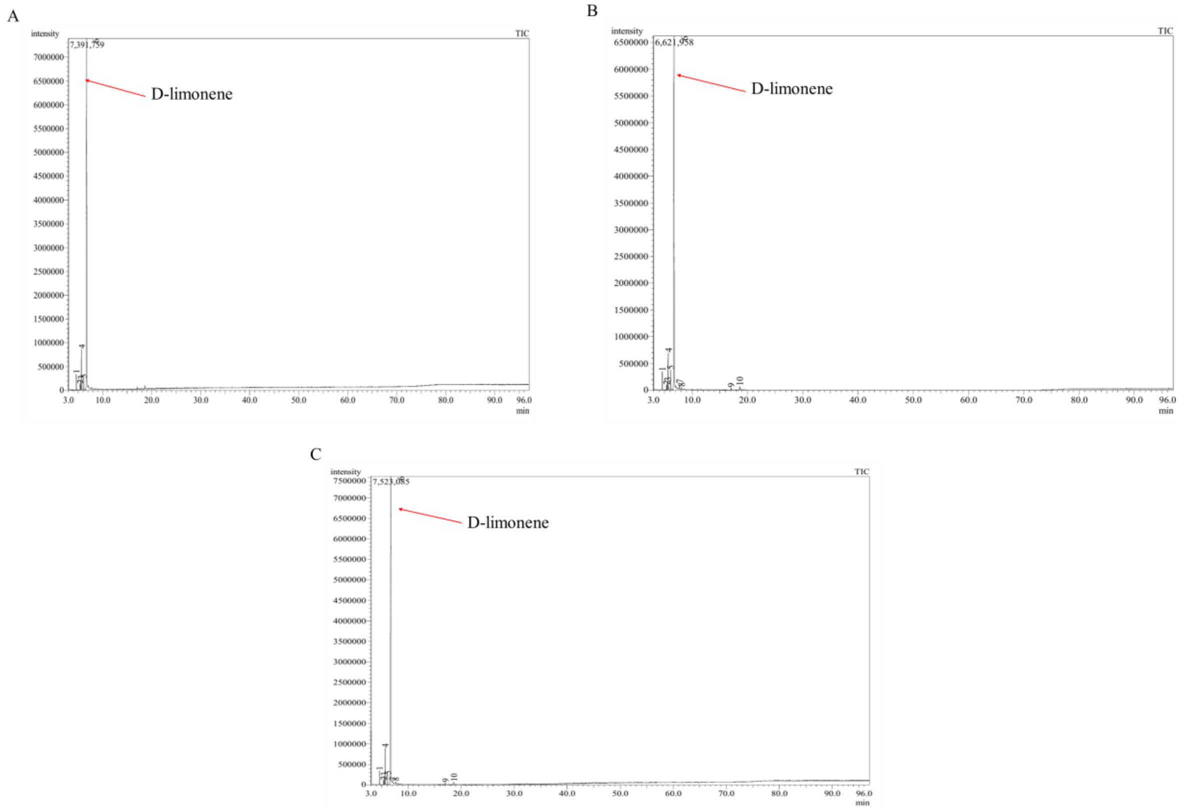

Figure 1. GC–MS chromatograms of essential oils obtained from young pomelo peels analyzed using gas chromatography–mass spectrometry (GC–MS).

The samples were prepared in three physical forms: (A) large fresh pieces, (B) fresh ground (finely blended), and (C) dried and finely ground.

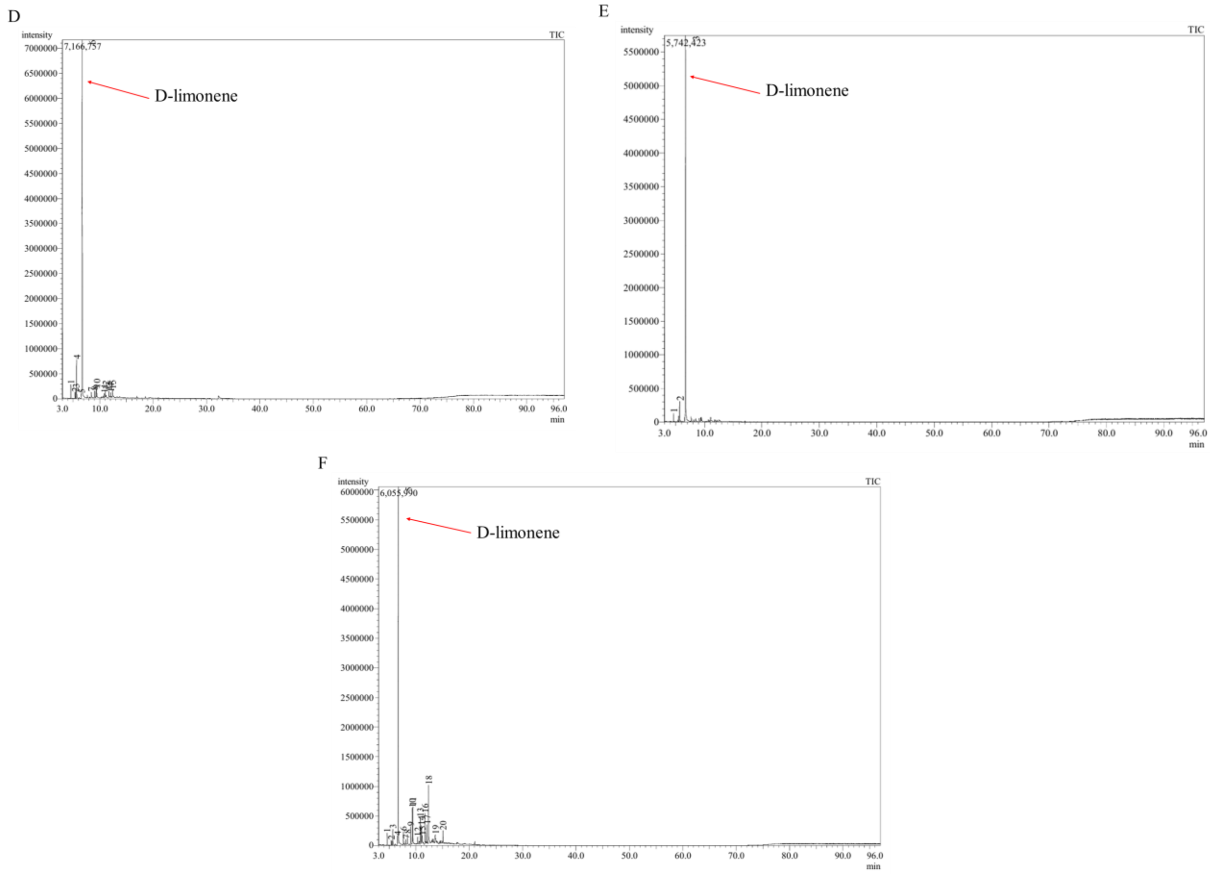

Figure 2. GC–MS chromatograms of essential oils obtained from Ripe Pomelo Peels analyzed using gas chromatography–mass spectrometry (GC–MS).

The samples were prepared in three physical forms: (D) large fresh pieces, (E) fresh ground (finely blended), and (F) dried and finely ground.

5. Conclusion

This study demonstrated that the essential oil characteristics of pomelo peels are strongly influenced by fruit maturity and the physical form of the raw material. Ripe pomelo peels produced a greater oil yield and exhibited more chemically diverse volatile profiles, whereas young pomelo peels yielded oils with consistently high purity of D- limonene. Regardless of maturity stage, D-limonene was the predominant compound in all essential oil samples.

Drying and fine grinding significantly enhanced oil extraction efficiency, indicating that removal of moisture and reduction of particle size are critical steps for maximizing oil recovery in steam distillation. In addition, the findings revealed that grinding the pomelo peel and leaving it for as little as one day, even when stored under refrigeration, resulted in a noticeable decline in essential oil yield. This reduction is likely due to rapid volatilization of monoterpenes, oxidation of exposed oil glands, and loss of aroma components once the peel tissue is ruptured. These results suggest that ground peel material is highly unstable, and delaying distillation regardless of cold storage causes essential oil loss and reduced extraction efficiency.

The chemical diversity observed in ripe samples reflects advanced metabolic development and oxidative transformation of terpenes during fruit maturation. These findings suggest that young pomelo peel is well-suited for industrial applications requiring high-limonene essential oils, while ripe pomelo peel provides a more complex volatile profile that may be beneficial for fragrance and therapeutic applications. The study also highlights the potential for utilizing agricultural by-products, specifically young, discarded fruits and peel residues, to create value-added essential oil products for cosmetic, aromatherapy, and natural product industries. Furthermore, the results emphasize the importance of minimizing the time between grinding and distillation to prevent terpene loss and preserve the chemical integrity of the essential oil.

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