



Limonene determination in *Citrus* peel extracts obtained by green supercritical fluid extraction

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ARTICLE INFO

HISTORY

Received: 30 July 2025

Revised: 13 November 2025

Accepted: 14 November 2025

Online Published: 30 December 2025

KEYWORDS

Citrus essential oils

Citrus peel

Freeze drying

Limonene

Phenolic compounds

Supercritical fluid extraction

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ABSTRACT

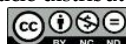
This study aimed to determine the chemical composition of essential oils extracted from the peels of four citrus fruit species—mandarin (*Citrus reticulata* L.), grapefruit (*Citrus paradisi* L.), lemon (*Citrus limon* L.), and orange (*Citrus sinensis* L.)—using supercritical fluid extraction (SFE). The extracted oils were analyzed and identified by gas chromatography–mass spectrometry (GC–MS). The main components and yields of the essential oils were as follows: mandarin oil contained limonene (85.8%), β -myrcene (6.2%), and γ -terpinene (5.1%) with a yield of 6.88%; lemon oil included limonene (79.8%), γ -terpinene (9.5%), and β -pinene (15.1%) with a yield of 5.56%; orange oil consisted predominantly of limonene (98.5%), with minor amounts of γ -terpinene (1.4%) and β -myrcene (1.7%) yielding 7.5%; and grapefruit oil contained limonene (97.9%), linalool (1.5%), and myrcene (1.8%) with a yield of 6.95%.

Limonene was identified as the predominant compound in all four *Citrus* essential oils, accompanied by relatively high extraction yields. The variation in limonene content among species may be attributed to genetic factors, geographical origin, and seasonal differences, as well as environmental parameters such as soil type and climate. Additionally, postharvest handling, drying conditions, and the plant tissues selected for extraction are likely to influence essential oil composition.

Citation: Burgaz, O., Yildirim, I., Sana, F., Baycan, A., Sahin Nadeem, H., & Polat, I., (2025). Limonene determination in *Citrus* peel extracts obtained by green supercritical fluid extraction. *Turkish Journal of Food and Agriculture Sciences*, 7(2), 206-215.

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1. Introduction

The *Citrus* genus (Rutaceae) comprises 28 taxonomically accepted species, encompassing both naturally occurring and cultivated hybrids collectively known as *Citrus* fruits, such as oranges (*C. sinensis*), lemons (*C. limon*), grapefruits (*C. paradisi*), limes (*C. aurantiifolia*), mandarines (*C. reticulata*), and citrons (*C. medica*) (Jing et al., 2015; Mora et al., 2025). These fruits hold substantial nutritional and therapeutic importance, as they are rich sources of vitamins B6 and C, amino acids, fibers, and bioactive phytochemicals, including essential oils, coumarins, and flavonoids (Ben Hsouna et al., 2023; Yüce et al., 2025). The extraction of these essential oils is commonly performed through the cold-pressing method; however, this conventional approach often yields oils of lower purity and reduced efficiency compared to techniques such as steam distillation or SFE, which provide improved temperature control and solvent selectivity. *Citrus* essential oils are characterized by a complex mixture of over 200 constituents, primarily composed (85–99%) of mono- and sesquiterpene hydrocarbons and their oxygenated derivatives, including limonene, myrcene, β -pinene, α -pinene, γ -terpinene, and sabinene. The remaining 1–15% corresponds to the non-volatile fraction, which is particularly rich in coumarins and polymethoxylated flavones that contribute significantly to the biological activity of the extracts (Russo et al., 2021; Yang et al., 2023; Modica et al., 2024; Mora et al., 2025).

Among these components, limonene ($C_{10}H_{16}$) stands out as the predominant monocyclic monoterpene found in *Citrus*-derived essential oils and their byproducts. It occurs as two enantiomers—R-(+)-limonene (D-limonene) and S-(–)-limonene (L-limonene)—which exhibit similar physicochemical characteristics yet differ slightly in aroma and biological activity (Figure 1) (Ciriminna et al., 2014; Yu et al., 2017; Siddiqui et al., 2022). R-limonene is the major naturally occurring isomer, especially in *C. sinensis*, *C. limon*, *C. reticulata*, and *C. paradisi*. Classified as Generally Regarded as Safe (GRAS) and characterized by low oral toxicity ($LD_{50} = 5\text{--}6$ g/kg), limonene has wide industrial applications as a flavoring and fragrance agent in the food, cosmetic, and pharmaceutical industries, and is also valued as an eco-friendly solvent and insect repellent (Ciriminna et al., 2014; Sun, 2007). Beyond its industrial relevance, limonene exhibits a range of pharmacological activities, including anticancer, antioxidant, anti-inflammatory, antimicrobial, and cholesterol-dissolving effects, making it a versatile bioactive compound with potential nutraceutical and therapeutic applications (Lu et al., 2004; Hirota et al., 2010; Chaudhary et al., 2012; Kim et al., 2013; Eddin et al., 2021; Asikin et al., 2022).

Preserving the bioactivity of such compounds during processing is crucial, and advanced techniques such as freeze-drying (lyophilization) play a key role in maintaining the structural and biochemical integrity of temperature-sensitive materials. This process involves three main stages: freezing, primary drying (sublimation), and secondary drying (desorption) (Figure 2) (Igual et al., 2019; Pudziuvelyte et al., 2020; Álvarez-Castillo et al., 2021; Stabrauskiene et al., 2024). Through the controlled removal of moisture under low pressure and temperature, freeze-drying ensures the production of stable, long-lasting, and chemically preserved materials suitable for further analysis or formulation.

Citrus fruits are not only valuable for their sensory appeal but also for their contribution to human health, owing to their high-water content, low caloric value, and abundance of bioactive compounds such as phenolics and carotenoids. Among them, *C. sinensis*, *C. limon*, *C. reticulata*, and *C. paradisi* are particularly notable for their nutritional richness and health-promoting properties. However, their seasonality and limited shelf life constrain their availability, which underscores the need for developing preservation and extraction methods that retain their beneficial compounds year-round (Igual et al., 2019).

To investigate these valuable compounds, a variety of analytical techniques are employed for characterizing *Citrus* essential oils (CEOs). Gas chromatography–mass spectrometry (GC–MS) serves as a primary tool for the identification and quantification of volatile compounds, while high-performance liquid chromatography (HPLC) is used to analyze non-volatile components such as flavonoids and phenolics (Ternelli et al., 2020; Trovato et al., 2021; García-Fajardo et al., 2023; Güzel et al., 2023). Complementary methods such as Fourier-transform infrared spectroscopy (FTIR) provide insights into the structural and molecular features of essential oils, whereas physicochemical properties—including density, refractive index, and optical rotation—help determine their potential industrial applications. Furthermore, *in vitro* and *in vivo* studies have demonstrated the antioxidant, antibacterial, and anti-inflammatory activities of CEOs, reinforcing their importance in functional and therapeutic product development.

In this context, extraction techniques play a pivotal role in defining oil quality. SFE, in particular, offers several advantages over traditional methods such as steam distillation and hydrodistillation, notably in maintaining the integrity of thermolabile compounds while providing superior efficiency and selectivity (Boughendjioua et al., 2020; Felicia et al., 2024). For instance, Yüce et al. (2025) reported that SFE performed on the peels of *C. limonia*, *C. deliciosa*, *C. latifolia*, and *C. sinensis* yielded extracts rich in key flavonoids—nobiletin, sinensetin, and tangeretin—and coumarins such as citropten, as revealed by LC–MS and GC–MS analyses (Yüce et al., 2025; Güzel et al., 2023).

Building upon this background, the present study addresses a notable gap in the literature by systematically evaluating the physicochemical characteristics of *Citrus* essential oils extracted from fruit peels using supercritical carbon dioxide (SCO₂) as a green solvent within the framework of SFE. This environmentally sustainable technique is distinguished by its high extraction efficiency, superior selectivity, and ability to preserve the natural composition of thermolabile compounds while yielding high-purity extracts with minimal solvent residues and reduced energy consumption. Although conventional methods such as cold pressing and hydrodistillation are frequently applied for *Citrus* oil extraction, research employing SFE remains relatively scarce. Therefore, the present work focuses on achieving high extraction yields and improved oil quality profiles among different *Citrus* species. The main objectives are to optimize the extraction parameters of *Citrus* peel essential oils, quantify their limonene content, and evaluate their potential as functional bioactive constituents for food-related applications. The novelty of this study lies in integrating green extraction technology with comprehensive chemical characterization to demonstrate the capability of SFE to enhance extraction efficiency while maintaining the compositional integrity and purity of *Citrus* essential oils. The *Citrus* fruits analyzed in this research were sourced from Seyhan, Adana Province, Türkiye (36.994049° N, 35.326356° E)—a region renowned for its favorable climate and fertile soils supporting extensive *Citrus* cultivation. Overall, this study contributes to the sustainable valorization of *Citrus* byproducts and provides scientific evidence supporting the implementation of eco-efficient extraction technologies within the food and cosmetic industries.

2. Material and methods

2.1. Raw material and sample preparation

These four *Citrus* fruit peels were obtained in a moist state from Özler Agriculture Company Ltd. These *Citrus* peels were grown in the Adana region. *Citrus* fruit peels were peeled in fruit peeling machines. Each *Citrus* peel (10 kg) was freeze-dried (Liyolife, FD5CT, 900W, Türkiye), ground (Emir, EMR-Ö-01, 1.5 kg, Türkiye), and sieved to obtain particles smaller than 0.30 mm. The prepared samples were stored at +4 °C until analysis.

2.2. Supercritical CO₂ extraction of *Citrus* peels

The SFE system (Polat Extraction Technology, Türkiye) consisted of a CO₂ cylinder, recirculating chiller, CO₂ and co-solvent pumps, an extraction vessel, heat exchanger, separation vessel, automated back-pressure regulator, and a programmable logic controller (PLC) for real-time process monitoring and control (Figure 3). Prior to extraction, the peels of *C. reticulata*, *C. paradisi*, *C. limon*, and *C. sinensis* were freeze-dried and stored at 25 °C until use.

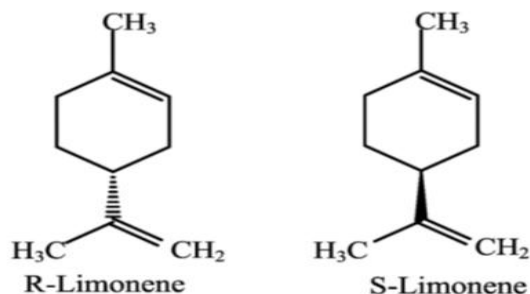


Figure 1. The chemical structure of limonene enantiomers (Güzel et al., 2024).



Figure 2. An Outline of the three key steps in the lyophilization process (Stabrauskiene et al., 2024).

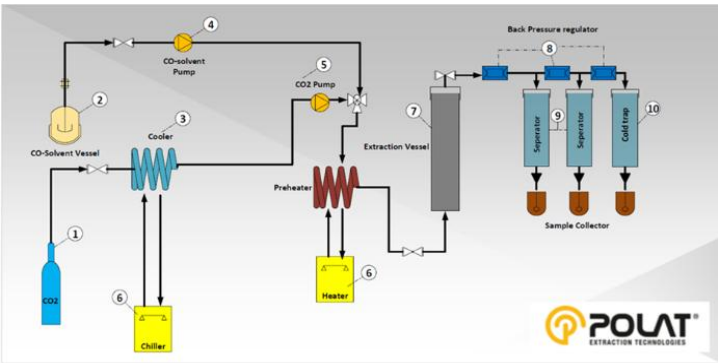


Figure 3. Diagrammatic representation of the SFE apparatus developed by Polat Extraction Technology, showcasing the main operational units and flow pathways (Burgaz et al., 2024).

During extraction, carbon dioxide was compressed and heated above its critical point (31 °C, 73 atm), achieving a supercritical state with both liquid-like solvent power and gas-like diffusivity. This dual property enabled CO₂ to effectively penetrate the *Citrus* peel matrix and dissolve the essential oil components with high efficiency. For each species, approximately 1085 g (*C. reticulata*), 1115 g (*C. paradisi*), 1100 g (*C. limon*), and 1090 g (*C. sinensis*) of peel material were placed in the extraction vessel. The main process parameters—temperature (°C), pressure (bar), and CO₂ flow rate (g/min)—were systematically varied to optimize extraction performance (Figure 3).

Following extraction, the obtained CEOs were collected in the separation vessel and stored at 4 °C until subsequent analyses. The SFE process provided an environmentally sustainable and efficient approach to obtain high-purity essential oils from *Citrus* peels while minimizing solvent residue and thermal degradation.

2.3. Design of experiments (DoE) and process optimization

DoE enables the optimization of key parameters in SCO₂ extraction to improve performance and preserve compound integrity (Burgaz et al., 2024).

The experimental work utilized the PEX1002C supercritical fluid extraction system, a laboratory-scale unit produced by Polat Extraction Technologies. Each *Citrus* peel sample underwent a single extraction cycle using carbon dioxide as the extraction solvent. A schematic representation of the extraction process is shown below. For the extraction process of *C. reticulata*, *C. paradisi*, *C. limon*, and *C. sinensis* peels dried in a freeze dryer, 1085 g, 1115 g, 1100 g and 1090 g were placed in the extractor, respectively. These four processes were carried out at 40 °C temperature, 120 Bar pressure, CO₂ flow rate 100 g/min and extraction time of 30 minutes. The resulting *Citrus* fruit peel extracts were stored at +4 °C for analysis. High yields and limonene were determined in these four runs (Table 1).

Table 1. Optimum operating conditions to obtain higher global yields of *C. reticulata*, *C. paradisi*, *C. limon* and *C. sinensis* extracts by supercritical CO₂ extraction

Sample	Conditions				
	P (Bar)	T (°C)	Time (min)	Sample Quantity (g)	qCO ₂ (g/min)
<i>C. reticulata</i>	120	40	30	1085	100
<i>C. paradisi</i>	120	40	30	1115	100
<i>C. limon</i>	120	40	30	1100	100
<i>C. sinensis</i>	120	40	30	1090	100

2.4. Analysis of limonene

Gas chromatography (GC) is a versatile analytical technique widely used for the effective separation of target compounds in complex samples. In this study, key GC parameters were systematically optimized to enhance mass spectrometry (MS) detection and overall analytical performance. The developed GC-MS method underwent comprehensive validation, assessing critical performance metrics such as linearity, sensitivity (including limits of detection and quantification), repeatability, and reproducibility. All validation procedures adhered strictly to the guidelines established by EURACHEM and the Standards for Standard Method Performance Requirements (Güzel et al., 2024).

For this purpose, 0.2 mL of the sample extract was added to 5 mL of saturated salt solution in 20 mL vials. Vials containing the samples were sealed and incubated at 40°C for 30 min before exposing a 65 µm PDMS/DVB fiber (Supelco) to the headspace under the same temperature for volatile adsorption. The SPME fiber was then desorbed in the GC-MS injector (Restek Rxi-5ms column) at 250°C for 2 min in splitless mode. The GC oven temperature program included an initial hold at 50°C (1 min), ramping to 200°C at 3°C/min, followed by an increase to 250°C at 8°C/min with a 5-min hold. MS detection scanned 35–450 m/z at 70 eV ionization energy. Identification was based on comparison with Wiley9N11 and NIST11 libraries. Essential oils containing limonene (Figure 1) are expressed as a percentage.

2.5. Higher global yields from *C. reticulata*, *C. paradisi*, *C. limon* and *C. sinensis* extracts

The essential oil yields of extracted *C. reticulata*, *C. paradisi*, *C. limon* and *C. sinensis* bark were calculated on a dry matter basis using the following equation (Felicia et al., 2024).

Yield of *C. reticulata*, *C. paradisi*, *C. limon* and *C. sinensis* (%) =

$$\left(\frac{\text{Weight of collected essential oil (g)}}{\text{Total weight of the sample (g)}} \right) \times 100$$

This method is employed to quantify the yield of CEOs extracted from plant materials. At the end of the extraction process, the amount of essential oil obtained (in grams) is weighed using a precision balance and is then divided by the total weight of the sample (in grams). The resulting ratio is multiplied by 100 to calculate the yield as a percentage. This calculation provides a scientifically grounded approach for evaluating the efficiency of essential oil extraction and enables comparisons across different plant species.

3. Results and discussion

3.1. Higher essential oil yield from *Citrus* peels

The essential oil content and composition of *Citrus* fruit peels can vary depending on factors such as *Citrus* variety, climate, geographical origin, maturity, size, pollination and extraction methods. Considering the significant oil yield and limonene molecules observed in *C. reticulata*, *C. paradisi*, *C. limon* and *C. sinensis* peel samples, these *Citrus* fruit peels, considered as waste, can be considered as a promising source of oil and potentially marketed as a value-added product. Since cold pressing and hydrodistillation are methods based on mechanical pressing and steam distillation rather than true extraction techniques, not all components may be fully recovered. The maximum global extraction yields were found to be 3.93% for *C. limon*, 4.18% for *C. deliciosa*, 3.48% for *C. latifolia*, and 5.32% for *C. sinensis* (Mora et al., 2025).

In a recent study, supercritical fluid extraction was employed as a sustainable method for isolating essential oils from orange peel. Chemical composition analysis revealed that the primary constituents included limonene (43.96%), α-pinene (15.60%), β-myrcene (10.21%), γ-terpinene (5.00%), and α-terpineol (4.15%), all present in substantial proportions (Felicia et al., 2024).

In another study conducted in Türkiye, R-limonene content in orange oil ranged between 56.39% and 72.85%, while S-limonene content ranged between 2.53% and 5.71%. In lemon oils, R-limonene content ranged between 54.73% and 73.99%, while S-limonene content ranged between 3.78% and 4.79%. In mandarin oils, R-limonene content was 58.02% and 65.05%, while S-limonene content was 3.05% and 4.87%. Analysis of grapefruit oil revealed that R-limonene constituted 60.69%, with S-limonene present at a significantly lower level of 3.12% (Güzel et al., 2024).

In lemon oil, thirty-one compounds have been reported across various analyses, representing about 91% of the overall oil profile. Among these, limonene dominates with a concentration of 69.9%, accompanied by β -pinene (11.2%) and γ -terpinene (8.21%). The remaining compounds were detected at trace levels (area <0.06%) and include camphene, octanal, α -phellandrene, cis-limonene oxide, trans-limonene oxide, terpinen-4-ol, decyl aldehyde, and citronellal (Viuda-Martos et al., 2008).

Analysis of mandarin (*C. reticulata*) essential oil revealed a total of twenty-three chemical constituents, collectively representing 88% of the total oil composition. The oil was found to be particularly rich in limonene (74.7%), which dominated the volatile profile, followed by notable concentrations of γ -terpinene (15.7%), p-cymene and α -pinene (each 2.0%), and myrcene (1.4%). In addition to these major components, several other constituents were detected at trace concentrations (approximately 0.06%), including octyl aldehyde, α -phellandrene, linalyl acetate, nonanal, trans-limonene oxide, α -farnesene, decyl aldehyde, β -caryophyllene, and α -bergamotene. These minor compounds, although present in low concentrations, may still contribute significantly to the oil’s aromatic profile (Viuda-Martos et al., 2008).

The essential oil extracted from grapefruit (*C. paradisi*) was found to contain 25 identifiable components, collectively accounting for 96% of the total oil composition. Among these, limonene was overwhelmingly dominant at 96.2%, followed by myrcene at 1.5%; both compounds contribute significantly to the characteristic aroma and potential bioactivity of the oil. Several minor compounds were also present at trace levels (approximately 0.06%), including α -thujene, β -pinene, β -ocimene, nonanal, isogeraniol, trans-limonene oxide, α -terpineol, trans-carveol, carvone, citral, geranyl acetate, germacrene D, and linalool. While these components are present in minimal quantities, they may contribute to the overall functional properties and complexity of grapefruit essential oil through synergistic or additive effects (Viuda-Martos et al., 2008).

Studies on the yield of CEOs from the peels of *C. reticulata*, *C. paradisi*, *C. limon*, and *C. sinensis* using supercritical fluid extraction have been reported, typically achieving relatively low yields of 3.48–5.32% (Mora et al., 2025). In the present study, freeze-dried peels of *C. reticulata*, *C. paradisi*, *C. limon*, and *C. sinensis* were extracted using the PEX1002C system designed and produced by Polat Makina under the following conditions: 120 bar pressure, 40 °C temperature, 30 min extraction time, and a CO₂ flow rate of 100 g/min. These conditions resulted in essential oil yields of 6.88%, 6.95%, 5.56%, and 7.50%, respectively (Table 2). Compared with reported values in the literature, the essential oil yields obtained in this study were higher, demonstrating the effectiveness of the applied extraction conditions.

Table 2. Yield of total essential oils obtained by supercritical fluid extraction from the peels of *C. reticulata*, *C. paradisi*, *C. limon*, and *C. sinensis*

Sample (<i>Citrus</i> species)	Mean yield (%) $\pm$ SD
<i>C. limon</i> (Lemon)	5.56 $\pm$ 0.15
<i>C. sinensis</i> (Orange)	7.50 $\pm$ 0.12
<i>C. paradisi</i> (Grapefruit)	6.95 $\pm$ 0.10
<i>C. reticulata</i> (Mandarin)	6.88 $\pm$ 0.18

Values are expressed as mean $\pm$ standard deviation (n = 3). Statistical analysis was performed using one-way ANOVA followed by Tukey’s post hoc test (p < 0.05)

3.2. Limonene characterization

In this study, essential oils rich in limonene were extracted from the peels of four *citrus* species using the PEX1002C SCO₂ extractor (Polat Makina, Türkiye) and subsequently analyzed by GC–MS. The limonene content of the oils ranged from 79.8% in *C. limon* to 98.5% in *C. sinensis*, confirming that limonene was the predominant component across all species (Table 3). The limonene content of the freeze-dried peels was found to be high. Compared with literature values, the limonene concentration in this study was among the highest reported (Viuda-Martos et al., 2008). As shown in Figure 4, the highest relative percentage of limonene was detected in *C. sinensis* essential oil.

Table 3. Analysis of essential compounds in supercritical CO₂ extracts of *C. reticulata*, *C. paradisi*, *C. limon* and *C. sinensis* peels

Compounds	Kovats Index (KI)	<i>C. limon</i> (% ± SD)	<i>C. sinensis</i> (% ± SD)	<i>C. paradisi</i> (% ± SD)	<i>C. reticulata</i> (% ± SD)
α-Pinene	939	1.5 ± 0.1	0.9 ± 0.1	0.5 ± 0.1	1.8 ± 0.2
Sabinene	975	1.8 ± 0.1	0.4 ± 0.1	0.4 ± 0.1	2.3 ± 0.2
β-Pinene	979	15.1 ± 0.3	0.5 ± 0.1	0.7 ± 0.1	0.4 ± 0.1
β-Myrcene	991	1.3 ± 0.1	1.7 ± 0.1	0.5 ± 0.1	6.2 ± 0.4
Myrcene	990	nd	nd	1.8 ± 0.2	nd
Limonene	1024	79.8 ± 0.5	98.5 ± 0.3	97.9 ± 0.3	85.8 ± 0.4
γ-Terpinene	1059	9.5 ± 0.2	1.4 ± 0.1	0.3 ± 0.1	5.1 ± 0.2
Linalool	1095	1.4 ± 0.1	0.6 ± 0.1	1.5 ± 0.1	0.9 ± 0.1
Citral (Neral + Geranial)	1232	1.6 ± 0.1	nd	nd	1.2 ± 0.1
Terpineol-4-ol	1177	0.5 ± 0.1	0.2 ± 0.1	0.3 ± 0.1	0.6 ± 0.1
α-Terpineol	1189	0.6 ± 0.1	0.3 ± 0.1	0.4 ± 0.1	0.4 ± 0.1
Octanal	1002	nd	0.28 ± 0.05	0.06 ± 0.01	0.19 ± 0.02
Decanal	1159	nd	0.28 ± 0.05	nd	nd

Values represent mean ± standard deviation (SD) from triplicate GC–MS analyses. “nd” indicates not detected

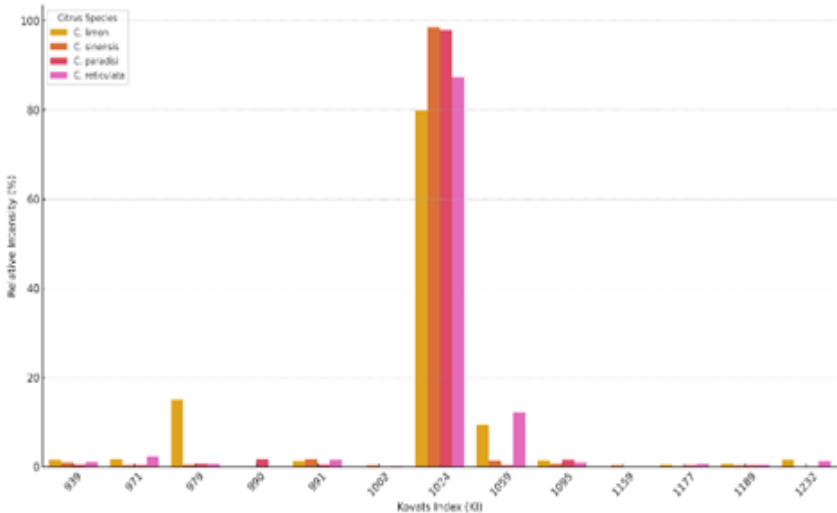


Figure 4. Essential compounds obtained by SFE extraction of freeze-dried *C. reticulata*, *C. paradisi*, *C. limon* and *C. sinensis* peels

4. Conclusion

This study demonstrates that the peel essential oils of *C. reticulata*, *C. paradisi*, *C. limon*, and *C. sinensis* represent promising bioresources when extracted using SFE. Compared with conventional extraction techniques such as cold pressing, hydrodistillation, and solvent extraction, SFE yielded higher recovery rates and superior chemical purity. Despite compositional variations among species, the results confirmed the feasibility of producing high-quality essential oils from *citrus* peels, with limonene identified as the dominant constituent in all samples.

The highest limonene concentrations and overall yields were obtained from freeze-dried peels, emphasizing the significance of pretreatment conditions in optimizing extraction efficiency. Across all species, limonene accounted for 79.8–98.5% of the total volatile fraction, while extraction yields ranged from 5.6% to 7.5%. These outcomes highlight the synergistic effect of combining freeze-drying and SFE in improving both recovery efficiency and the preservation of thermolabile compounds.

Considering that *citrus* peels are typically regarded as underutilized agro-industrial by-products, their valorization through SFE offers an environmentally sustainable and economically viable approach to obtaining high-value natural compounds. Future research may focus on fine-tuning process parameters to enhance the recovery of limonene and other bioactives with antioxidant potential. Moreover, further investigation into the functional and industrial applications of *citrus* peel essential oils in the cosmetic, pharmaceutical, and food sectors could expand their commercial and environmental significance.

Compliance with Ethical Standards

Conflict of Interest

The authors claim that they have got no conflict of interest.

Authors' Contributions

Osman BURGAZ: Conceptualization, Validation, Formal analysis, Investigation, Data curation, Writing manuscript, Supervision, Visualization, Extraction, Project administration, Funding acquisition. **Ilker YILDIRIM:** Validation, Investigation, Data curation, Review and editing, Supervision. **Ferhat SANA:** Formal analysis, Investigation. **Alper BAYCAN:** Formal analysis, Investigation. **Hilal SAHIN NADEEM:** Data analysis, Review. **Ibrahim POLAT:** Conceptualization, Validation, Funding, Funding acquisition.

Ethical approval

This manuscript has not been submitted elsewhere, and the findings presented here have not been utilized in any studies involving animals or human subjects.

Funding

The author has not disclosed any funding.

Data availability

Not applicable

Consent for publication

The author of this manuscript consent to the publication of the study's findings in this journal.

Acknowledgement

We also appreciate the valuable contribution of lab boys, field staff and juniors during the experimental period. We sincerely thank want to express our sincere gratitude to Mr. Volkan Polat and Polat Makina Industry and Trade Inc. Polat Makina Tic. A.S. for their invaluable support and contribution to this work-study. Their guidance and the resources they provided were instrumental in the successful completing this work-study.

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