



Extraction and Comparison of Limonene Contents of Wastes in *Citrus* Species for Reuse

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ABSTRACT

Identification and extraction of limonene in fruits of the genus *Citrus* L. which has several medicinal, cosmetic, and industrial properties, is crucial for reusing fruit wastes after they have been processed into fruit juice. The aims of the current study are therefore (i) to compare limonene contents in different fractions such as peels, pulps and juices of citrus species including orange (*C. cinensis* L.), lemon [*C. limon* (L.) Osbeck], grapefruit [*C. paradisi* (L.) Macfad.] and tangerine (*C. tangerina* Tanaka)), (ii) to extract limonene from the most abundant fraction of selected citrus species and (iii) to optimize the extraction parameters of supercritical fluid carbon dioxide (SFC) extraction system. The limonene contents were determined using high performance liquid chromatography (HPLC) and extraction conditions of SFC were optimized by Response Surface Methodology (RSM). The highest limonene content was determined in the peel fraction of the lemon. The limonene was found to be <LOQ in all tested fruit juices. In the subsequent step, limonene was extracted from lemon peels using the SFC extraction system, and the optimal parameters for the system were determined to be 52°C and 147 Mpa. In conclusion, peels and pulp fractions of citrus fruits can be potential limonene extract due to the highest content of limonene after being processed into fruit juice. The results of the current study elucidated that by extracting limonene from citrus peel and pulp waste, not only environmental pollution was prevented, but also the reuse of the waste was transformed into a sustainable production model.

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Introduction

The genus *Citrus* L., which belongs to the subfamily *Aurantioideae* and the family *Rutaceae*, consists of important domesticated species such as orange (*Citrus cinensis* L.), lemon [*C. limon* (L.) Osbeck], grapefruit [*C. paradisi* (L.) Macfad.] and tangerine (*C. tangerina* Tanaka). As one of the fruits with the highest consumption rates worldwide, citrus fruits are popular for both their healthy components and health supplements[1]. According to the Food and Agriculture Organization (FAO) statistical database in 2022, the most produced citrus fruit was orange with 76.4 million tons followed by tangerine or mandarin, or clementine with a total 44.1 million tons, and lemon or lime with 21.5 million tons, while the least produced citrus was grapefruit or pomelo with 9.7 million tons in the world [2]. Despite the strong demand for citrus fruit, there is still a significant proportion has been wasted, for instance, almost 50% of lemons are utilized to make fruit juice and marmalade, and a considerable proportion of lemons, between 50% and 60% of the processed fruit, is wasted [3]. Global annual production of 25 million tons is estimated just for orange peel wastes, it is therefore omitted a significant peel waste and a chance to recover them [4]. In terms of worldwide production, citrus waste equals to around 110–120 million tons per year. The focus must be centered on valuable components made of organic acids derived from citrus wastes and biodegradable polymers in the food processing, pharmaceutical, and chemical industries [5]. Commercially damaged fruit and peeled or pressed wastes for fruit juice factories are the three primary forms of citrus waste. Since these materials are waste, they cannot be linked to the food chain. For every 1000 tons of fruit processed, commercial citrus juicing produces 450–550 tons of waste solids including peel and rag [6]. Citrus waste has a low pH (3–4), a high water content (80–90%), and a high organic matter level (95% of total solids). According to European regulations, these traits

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make citrus waste inappropriate for landfill disposal (Council Directive 2008/98/EC of 10th November 2008 on waste) [7]. On the other hand, these waste products have biological active contents that are essential for industrial properties, such as phenolics, flavonoids, and limonene. Citrus peels, which includes approximately 4% (w/w) of limonene, are appropriate product for the recovery of valuable molecules [8].

Limonene ($C_{10}H_{16}$) is a cyclic monoterpene [9] that a citrus-like odor and a colorless liquid at room temperature. In addition, it can be soluble in water 7.57 mg/L at 25°C [10]. This compound can be used as a scent in soaps and fragrances as well as a sweetener in some foods. Limonene is also extensively used in the food industry to flavor foods such fruit juices, sweets, chewing gum, soft drinks, and ice creams, such as lemon aroma in chemical industry [11]. The antibacterial activity of limonene against three bacteria and three yeasts, constituting the spoilage microflora of fruit juices, was observed [12]. Furthermore, limonene has antifungal properties were discovered as well [13]. Thus, limonene is considered as a safe food preservative. Moreover, in agricultural area, limonene is a safe and natural pesticide by directly or in combination with other compounds [14]. The anticancer effects of limonene have a positive impact on some animal cells. Regardless of the precise mechanism of limonene is unknown, it may be a promising new inquiry for breast cancer [15]. Hence the mentioned reasons, limonene is commonly preferred ingredient in numerous industrial applications. Extraction systems SFC appears to be a promising and different process because it operates at a low temperature, has a good mass transfer rate, and leaves no solvent residue in the finished product [16]. By employing SFC, it was possible to determine the separation of limonene from a certain plant fraction by selecting the proper temperature-pressure ratio during the extraction.

In the current study, citrus fractions compared to produce limonene, which is used in the food and agricultural industries in addition to the cosmetic business. Limonene content varies depending on environmental conditions and is affected by species of citrus and genotypes in the species. According to available literature, there is a gap on comparison of limonene content of citrus species. The current study is distinctive in that it compares the amounts of limonene found in various citrus fruits and their various components. The objectives of the current study are to (i) quantify the amount of limonene in a few *Citrus* species and (ii) identify the most precise SFC parameters.

Material and Methods

Plant materials

Citrus fruits used in the current study, orange (*Citrus cinensis* L.), lemon [*C. × limon* (L.) Osbeck], grapefruit [*C. × paradisi* (L.) Macfad.] and tangerine (*C. tangerina* Tanaka) were purchased from markets located in Antalya, Türkiye in December 2021. Collected materials were peeled and remaining parts were squeezed. In this case, three different fractions as peel, juice and pulp (remaining part after squeezing) were obtained. The juice fractions were analyzed directly, whereas peel and pulp fractions were analyzed after a drying process at the conditions of 40°C for 72 hours in an incubator. The dried materials were ground by a mechanical grinder and sifted by sieves. All the materials in the current study were stored at -18°C prior to extraction to protect the loss of active ingredients.

Chemicals

Limonene analytical standards were obtained from Sigma-Aldrich Co. (St. Louis, MO, USA). All chemicals used in the current study except analytical standards were of liquid chromatographic grade. Methanol (MeOH), ethanol (EtOH), and acetonitrile were obtained from Isolab (Wertheim, Germany), while acetic acid was purchased from Merck (Darmstadt, Germany). The analytical standards at various concentrations were prepared in MeOH for HPLC analysis. The water for preparing the HPLC mobile phase was produced by an ultrapure water purification system of MP Mini pure (Ankara, Türkiye). CO₂ gas for SFC extraction was supplied by HABAS (Antalya, Türkiye).

Extraction of limonene by SFC extraction system

According to the HPLC analysis results, the fraction with the highest level of limonene concentration were extracted by a SFC extraction system (SuperEx F-500, Pard Engineering, Konya, Türkiye). For this aim, the fraction was prepared in a larger amount and weighed 50 grams. The material was placed to the reactor part of SFC extraction system. The system was held for 15 min in the static phase and the total extraction time was set at 75 min, under selected temperature and pressure. The experimental conditions of SFC extraction system for optimization of parameters are shown in Table 1.

Table 1. Trial design to find optimum SFC conditions

Experiments	Temperature (°C)	Pressure (bar)	Material (g)	Extract (g)
1	33	120	50.2	0.53
2	47	120	50.0	0.48
3	47	180	50.8	0.56
4	33	180	50.7	0.46
5	30	150	50.2	0.69
6	40	108	50.4	0.33
7	40	200	50.5	0.32
8	40	150	50.6	0.27
9	40	150	50.9	0.28
10	40	150	50.2	0.29
11	40	150	50.5	0.56
12	40	150	50.6	0.47

Determination of limonene concentration

Prepared fractions of citrus fruits were analyzed according to following procedures using by a HPLC method. Briefly, 0.5 g of prepared sample were transferred to a glass bottle and 10 ml of EtOH was added. The mixture was applied to ultrasonic assisted extraction system (ISOLAB, Eschau, Germany) for 10 min. The system configuration was a frequency of 23 kHz, power of 750 watts and temperature of 50°C. After the homogenization the solution was filtered via paper filter and 0.22 µm PVDF membrane filter. Clarified solution was diluted with MeOH and injected to the HPLC system (Agilent 1200 Technologies, Böblingen, Germany) with Diode Array Detector (DAD). Chromatographic separation was performed via C18 column (250 mm × 4.6 mm × 5 µm). The mobile phase composition was applied to be acetonitrile (78%) and ultra-pure water (22%). The injection volume and column temperature were 20 µL and 40°C, respectively. The total chromatographic run was 30 min with a flow rate of 2 mL/min. All peaks in the chromatogram were detected at a wavelength of 210 nm.

Statistical analyses

Response-surface method was used to optimisation of the extraction parameters. In the optimization process, the specified parameters' first and second-order effects on the concentrations of the extracted limonene and their possible interactions with each other were statistically investigated. As a result of the analysis, 3-D response surfaces graphics that show these parameters' effects were created using the models in which the process parameters were effective. Student's t-test was used to evaluate the data. The data were evaluated at a 95% confidence interval and $p < 0.05$ significance level. MINITAB 18 and Microsoft Excell software were performed for descriptive images of the features.

Results

Method validation

According to the results of method validation study, the calibration curve was constructed by different concentration of limonene. It was concluded that the method was linear ($r^2=1.000$) in the specified concentration ranges (1-64 ppm). In addition, the concentrations of the analytical limonene standard at the same concentration with the LOQ level were injected six times, and the obtained peak area sizes and retention time shifts were evaluated. The results showed that the shift in the retention time of the limonene was found to be insignificant. The LOD (limit of detection) and LOQ (limit of quantification) values of the limonene were determined to be 0.07 and 0.24 ppm, respectively. In the repeatability study, the %RSD (relative standard deviation) value was determined to be 3.30%. An overview of the calibration chart is shown in Figure 1.

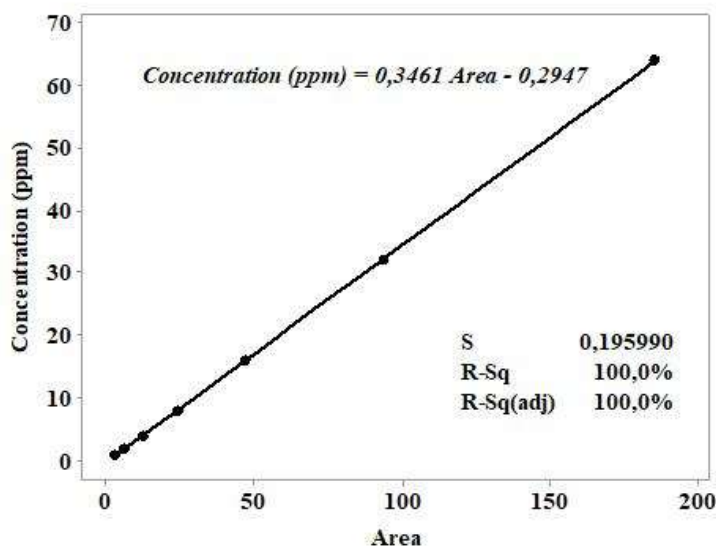


Fig 1 Calibration chart

Fractions of the citrus

The percent mass distributions of citrus fractions as juice, pulp (after squeezing) and peel were presented in Figure 2. According to the findings, the highest amount of juice fraction was determined in grapefruits (88%) where the highest portion of peel and pulp fractions in the lemon was found to be 31% and 21%, respectively.

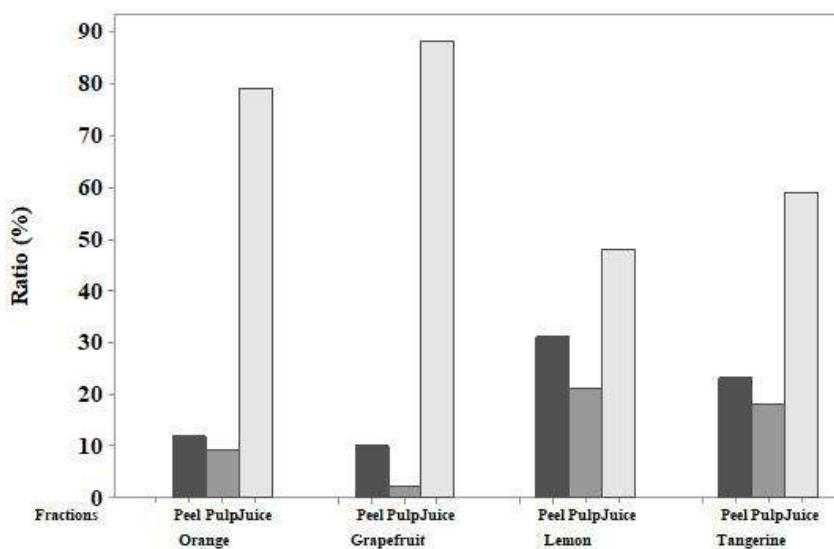


Fig 2 The percent mass distributions of citrus fractions

Obtained peel and pulp fractions of the citrus were dried prior to the extraction process and percentage reduction of weight was calculated. According to the results presented in Figure 3, the most weight reduction after drying process was found in grapefruit pulp (88%), and the least reduction was determined in tangerine peels (25%).

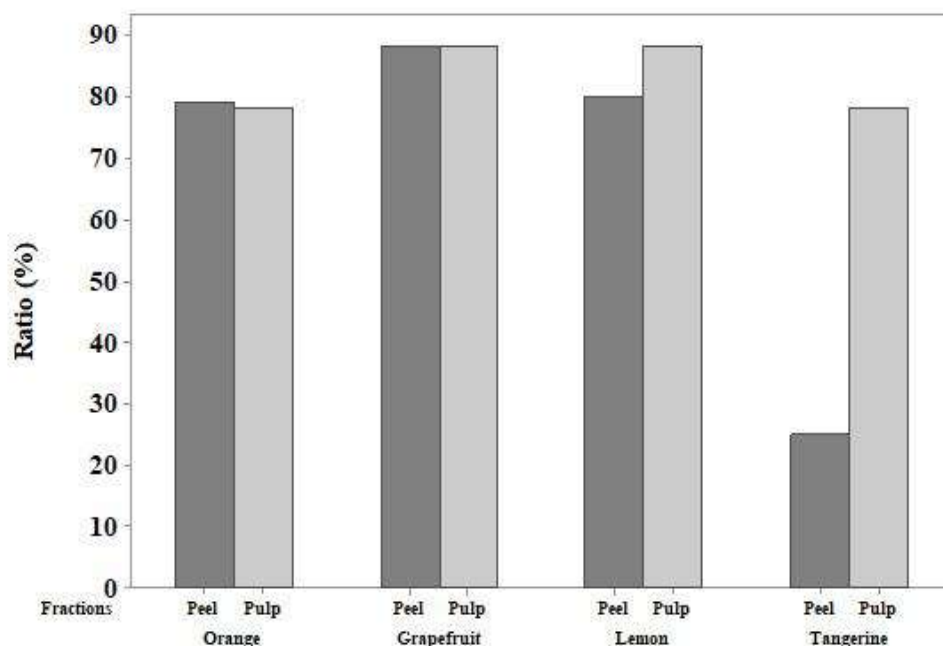


Fig 3 Weight loss of peel and pulp fractions of the citrus fruits after drying

Limonene contents of the fractions

Dried fractions of the citrus were analyzed for limonene contents by HPLC, and the results were presented in Figure 4. According to the findings the highest limonene was found in the peel fraction of lemon (58390.53 ppm), secondly in the orange peel (54768.17 ppm). Orange contains a significant amount of limonene in the pulp fraction (981.60 ppm), whereas limonene is <LOQ in fruit juices. Since fruit juices were stored at -18°C, this is thought to be due to the loss of limonene activity when citrus fruits are stored at low temperatures.

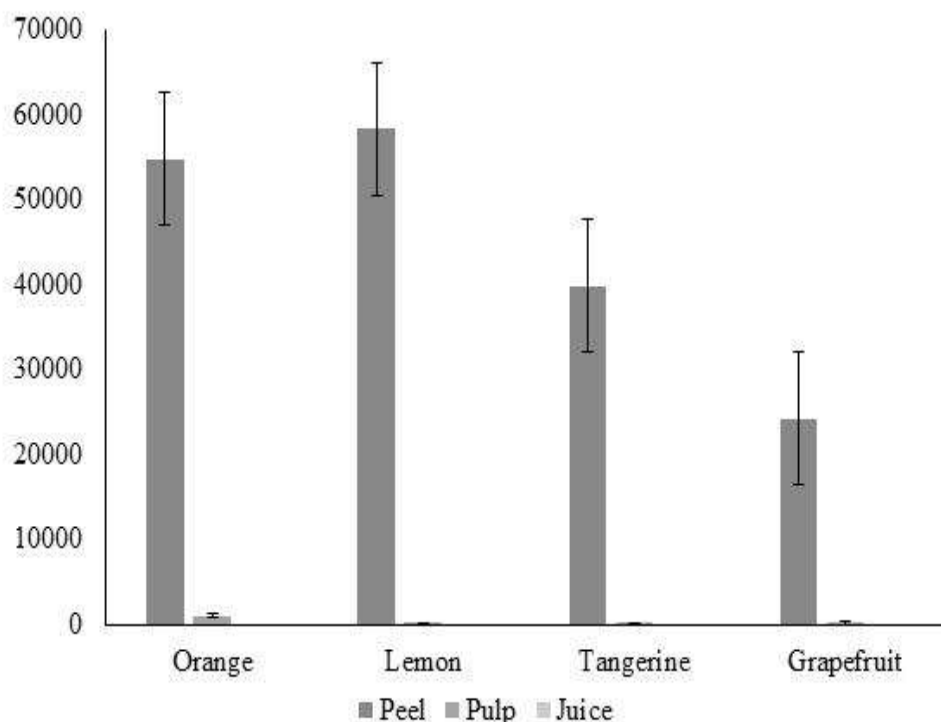


Fig 4 The fraction of limonene content of different dried citrus fruits

The study investigated the effect of a single drying method and focusing exclusively on limonene as the oil component. An overview of Limonene Content in *Citrus* spp. is illustrated in Figure 4.

Extraction of limonene from lemon peel by SFC

As shown in Figure 4, limonene was found in lemon peels at the highest level; thus, extraction was carried out with lemon peels. Lemon peels were treated like the beginning of the current study, such as dried in an oven at 40°C for 72 hours and sieved. After the experiment parameters were set up, the optimal limonene yield under different temperature and pressure conditions SFC extraction system was estimated by RSM. Obtained graph was presented in Figure 5. Optimum temperature and pressure values SFC extraction for the extraction of limonene was demonstrated in Table 2.

Table 2. Optimum conditions of SFC extraction

Limonene (ppm)	Temperature (°C)	Pressure (MPa)
450000	57	147

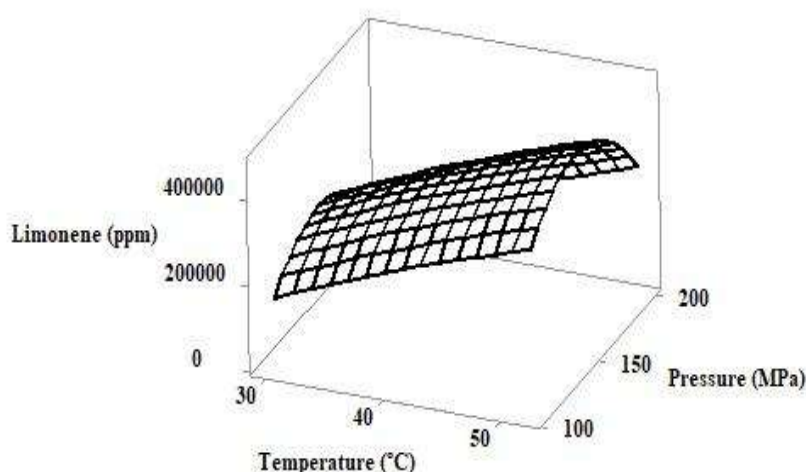


Fig 5 Optimal limonene yield in SFC

Discussion

According to a previously presented study, that worked on low density-dispersive liquid–liquid microextraction (LD-DLLME) of limonene the method demonstrated optimum value linearity with $r \geq 0.9950$ with low LOD also LOQ levels between 0.00081 and 0.00269 ppm, respectively [17]. A study revealed that limonene is the most abundant compound in essential oils extracted from lemon peels, accounting for 44.74% of the oil [18]. Also, earlier studies identified limonene as one of the major components in citrus peels [19,20]. The results showed that D-limonene was the primary component in all samples analyzed. The oven-dried peel powder samples at 40°C produced a large proportion of the primary components of limonene [21]. Earlier research showed that the chemical makeup of volatile oils in Chinese citrus peels was highly influenced by drying conditions, leading to significant variability in their composition. These studies also highlighted that the primary component in orange peel oil was limonene [22,23]. According to a study, D-limonene was detected in the MeOH extract obtained from orange peels at a concentration of 4.642 mg/mL, and the yield of D-limonene was 0.4% after the extraction process. The quality of the extract and yield can be affected by various factors, including the type of solvent, production process, and equipment utilized [24]. A research

study extracted limonene from orange peel waste using various solvents including cyclopentyl methyl ether, methyl-tetrahydrofuran, and hexane. The study found that the extraction yields of limonene from orange peel waste were 0.81%, 0.64%, and 0.53%, respectively [25].

In a previous study reveals that the highest amounts of limonene were measured as 1027 ppm in fresh lemonade in lemons and 402 ppm in limeade in limes [26]. Our results are not in agreement with the findings of this mentioned study, since lemon juices was not including limonene according to our results [27] revealed that the highest amount of limonene was detected in fresh lemon peels with 65000 ppm approximately. Dried and grinded lemon peels have the second highest limonene amount, while no grinded lemon peels have the least limonene 25000 ppm. It is suggested from mentioned study that grinding is essential for extraction of limonene. Another study indicates that limonene contents in oils of grapefruit, orange, mandarin and limonene were 96.2%, 94.9%, 74.7% and 69.9%, respectively, also in each of the four citrus oils analyzed, limonene was the most abundant constituent [28]. The chemical composition of four citrus oils, namely mandarin (*C. reticulata* L.), lemon (*C. limon* L.), orange (*C. sinensis* L.), and grapefruit (*C. paradisi* L.), was studied, the percentage of limonene in these essential oils was 74.7, 69.9, 94.9, and 96.2%, respectively [28]. However, the difference in the reported value could be due to several factors such as the chemical and biotype of the plant, climatic conditions, and the extraction process [29]. Additionally, the chemical composition of the starting material may vary depending on various factors such as the plant's health, growth stage, habitat including climatic factors, morphological factors, and harvest time [30].

Limonene extraction by using high pressure-temperature extraction (HPTE), an earlier study revealed that the least yield was found in 105 minutes at 100 °C using an HPTE approach, whereas the best limonene conditions were attained in 30 minutes at 0.6 Mpa pressure and 150 °C [27]. Telling the difference, the optimal limonene extraction demands higher pressure and lower temperature in the SFC system, as demonstrated by our studies. The research was centered on utilizing dehydrated and ground waste lemon peels to extract lemon essential oil through SFE. Various experiments were conducted at different temperatures and pressures ranging from 35-50°C and 12.5-20 MPa to determine the impact of operating conditions on total extract and D-limonene yield. The ideal conditions for extracting limonene were found to be 15 MPa and 40°C [31]. Hydro-distillation gave a higher yield in D-limonene from fresh peels than SFE from dehydrated peels, but this difference is not only due to the extraction method. Both drying and grinding pre-treatments of matrix to be extracted can significantly influence the yield on D-limonene [31], as also Asekun et al. (2007) suggested.

According to a study, limonene (61.8 %) was discovered to be the primary component of lemon peel essential oils [32]. Similarly, it was determined that the main component of the chemical composition of the essential oils of *C. limon* peel was limonene (61.68%) [33].

So far, [34] dealt with the limonene extraction from SFC method and reported that 15 MPa is the optimum pressure, also, by the pressure increases, yield decreases. According to our current study, low pressure levels were not chosen for the experiment. At a pressure of 10 MPa, 30 °C offered the highest extract recovery (95% w/w), while the highest DL concentrations could be achieved at 60 °C (99% w/w). This was attributed to increased density at higher pressures or lower temperatures [35].

In a study, SFC extraction at 60 °C at two different pressures (30 and 20 MPa) and with two different proportions (10% and 20%) of ethanol used as the extraction solvent was used to identify the compounds in lemons and their limonene. The study showed that the use of 20 MPa and 20% ethanol gives the highest percentage of limonene, which was 30.70%. In the same study, liquid CO₂ extraction was used on limonene and showed that the use of 20% ethanol with liquid CO₂ extraction increased the percentage of limonene up to 43.84% [36]. From the current study, we conclude that the use of some solvents leads to an increase in the limonene ratio, and that the high pressure may lead to a decrease in the limonene ratio extracted from lemon peels. Raising the temperature and pressure to attain CO₂'s supercritical state may cause a deterioration in the quality of limonene [19].

Conclusion

The first step in the current study was carried out with HPLC system to analyze the most abundant limonene in the fractions of some citrus fruits like orange (*Citrus cinensis* L.), lemon [*C. limon* (L.) Osbeck], grapefruit [*C. paradisi* (L.) Macfad.] and tangerine (*C. tangerina* Tanaka). The HPLC results revealed that, with a concentration of 58390.53 ppm, the lemon peel had the highest level of limonene. The following batch of orange peels contained 54768.16 ppm limonene. Fruit juices, however, did not contain any limonene that could be found; in another terms, <LOQ in fruit juices. Hence lemon peels have the highest concentration of limonene, it was extracted from lemon peels using the SFC method in the following step with an optimization chart was set up, and the most appropriate parameter was found in the SFC technique at a temperature of 52 °C and a pressure of 147 Mpa.

Limonene is a by-product of the fruit juice industry that is useful for commercial use. Moreover, citrus waste can be recovered for limonene compound. If it is handled properly, it can be utilized in numerous applications. Since our resources in the world is limited, it is predicted that the findings of the current study can be used for recovering of in citrus wastes.

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Data Availability Statement

The author confirms that the data supporting this study are cited in the article.

Compliance with ethical standards

Conflict of interest

The author declare no conflict of interest.

Ethical standards

The study is proper with ethical standards.

Authors' contributions

Tuğba Pelin TOKER: Formal analysis, Investigation, Writing – original draft, Mustafa Hamza Mawlood Al Bayati: Formal analysis, Methodology, Writing – original draft, Mariem Bouali: Formal analysis, Validation, Ümit Babacan: Formal analysis, Validation, Mehmet Fatih Cengiz: Writing – review & editing, Supervision, Investigation.

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