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Research Article

Effects of hot water treatment effects on the essential oils of calamondin

Hsin-Chun Chen [a], Li-Wen Peng [b], Ming-Jen Sheu [c], Li-Yun Lin [d], Hsiu-Mei Chiang [a], Chun-Ta Wu [e], Chin-Sheng Wu [a], Yu-Chang Chen [f,*]

[a] Department of Cosmeceutics, China Medical University, Taichung, Taiwan, ROC
[b] Department of Research and Development, Kuai Kuai Co. Ltd., Zhongli City, Taoyuan, Taiwan, ROC
[c] Department of Horticulture, National Ilan University, I-Lan, Taiwan, ROC
[d] Department of Food Science and Technology, Hungkuang University, Taichung, Taiwan, ROC
[e] Department of Horticulture and Landscape Architecture, National Taiwan University, Taipei, Taiwan, ROC
[f] Department of Chinese Pharmaceutical Sciences and Chinese Medicine Resources, China Medical University, Taichung, Taiwan, ROC

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ABSTRACT

Volatile constituents of calamondin peel or whole fruit were obtained by cold pressing, steam distillation, or hot water treatment at 90 °C for 15 minutes followed by steam distillation. The volatile components of the essential oils were identified by direct injection coupled with gas chromatography–flame ionization detector. A total of 54 compounds were identified, including 13 monoterpenes, 7 monoterpene alcohols, 1 monoterpene oxide, 4 monoterpene aldehydes, 2 monoterpene ketones, 4 monoterpene esters, 12 sesquiterpenes, 3 aliphatic alcohols, 6 aliphatic aldehydes, and 2 aliphatic esters, with limonene and β-myrcene as the major compounds. The results showed that hot water treatment increased the yields of essential oils from both peel and whole fruit. The relative percentage of the principle constituents in the various prepared essential oils were similar, except for some minor compounds, including linalool, terpinen-4-ol, α-terpineol, and carvone, the content of which were boosted by steam distillation. Whole fruit contained higher levels of monoterpene alcohols than peel did.

1. Introduction

Calamondin (*Citrus microcarpa* Bonge) is a member of the Rutaceae family. It is widely grown in tropical and subtropical areas, including Taiwan, China, the Philippines, Vietnam, and Malaysia. The round or flat round fruit has a green/orange color with a diameter of approximate 3 cm. The peel is thin and the flesh is sour. The fruit is eaten together with the peel.

The fruit can be squeezed for juice and used as a brewed fruit tea due to its low content of sugars (glucose, fructose, and sucrose), rich aroma components and high levels of ascorbic acid (44.5 mg/100 g), dehydroascorbic acid (2.2 mg/100 g), and citric acid (3.6%) [1]. Calamondin juice is characterized by its

* *Corresponding author*. Department of Chinese Pharmaceutical Sciences and Chinese Medicine Resources, China Medical University, 91 Hsueh-Shih Road, Taichung, Taiwan, ROC.
E-mail address: yuchang@mail.cmu.edu.tw (Y.-C. Chen).

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high acidity, as reflected by the high citric acid content [1]. Thus, calamondin drinks are currently popular.

There were very few studies on the volatile compounds of calamondin. One of the first reports on calamondin peel oils was by Nigam et al [2]. In 1980, Mina [3] reported the identification of 6 aroma components in calamondin juice. Nisperos-Carriedo et al [1] identified 20 volatile flavor compounds in calamondin, including 5 aldehydes, 2 esters, 5 alcohols, and 8 hydrocarbons. Moshonas and Shaw [4] identified 56 components in calamondin peel oil extracted with hexane, and reported that limonene and β-pinene were the major components. Yo and Lin [5] analyzed the flavor components of Taiwan calamondin and Philippine calamansi by solid-phase microextraction, and identified 58 aroma components, including 8 monoterpene hydrocarbons, 3 oxygenated monoterpenes, 18 sesquiterpenes, 2 aliphatic hydrocarbons, 10 esters, 11 aldehydes, 3 alcohols, and 3 ketones. They concluded that Philippine calamansi juice has statistically higher percentages of ketones, monoterpene hydrocarbons, and sesquiterpenes than calamondin from Taiwan. Takeuchi et al [6] analyzed volatile compounds in Philippine calamondin peel and juice by gas chromatography (GC) and GC–mass spectrophotometry (GC/MS), and identified a total of 58 and 98 volatile compounds in the peel and juice concentrates, respectively. Takeuchi et al [6] reported that the major components in the peel were limonene, myrcene, γ-terpinene, β-elemene and (*E*,*E*)-α-farnesene, and the principal constituents in the juice were limonene, linalool, α-terpineol, and terpinen-4-ol. Cuevas-Glory et al [7] identified 59 and 72 compounds in the peel and leaf oils of calamondin, respectively, and found limonene was the most prominent compound that accounted for 77.0% of peel oil. Cheong et al [8] identified a total of 79 volatile components of calamansi peel from Vietnam, the Philippines, and Malaysia. Moreover, they reported that Malaysian calamansi peel has a statistically higher percentage of methyl *N*-methylanthranilate than calamansi peel from other areas. However, Cheong et al [9] studied calamansi from three countries (Malaysia, the Philippines, and Vietnam) using extraction with dichloromethane, and the extracts were analyzed by headspace solid-phase microextraction. They identified a total of 60 volatile compounds, and reported that Vietnam calamansi juice contained the highest amount of volatiles.

Calamondin fruit is used in Taiwan mainly for fruit tea and candied fruit. Fruit tea is prepared by adding boiling water into calamondin fruit. Many Taiwanese like to drink this fruit tea warm, especially in winter, due to its aroma and taste. This work was aimed to investigate the heating effects on the constituents in essential oils extracted from calamondin peels or whole fruits. Two heating methods were employed: steam distillation (SD) and hot water heating (HWH). The differences in aromatic notes caused by heating will also be discussed.

2. Methods

2.1. Plant material

Calamondins were purchased from Taipei Agricultural Products Marketing Co., Taipei, Taiwan. The calamondins were washed under running water, dried, and separated into two groups as follows: one to be hand peeled for peel analysis and the other for whole fruit analysis.

2.2. Extraction of essential oil

2.2.1. Cold pressing of peel

The calamondins were peeled by hand, and a sugarcane squeezer was used to squeeze 1 kg of peel. The vacuoles containing oil in the peel were broken, and the oil was released. The mixture of squeezed oil and pomace were left to rest in a beaker at 4 °C for 1 day to achieve perquisite lamination. The oil layer was isolated by centrifugation at 6000 × *g* at 4 °C for 40 minutes. The essential oils were obtained by further centrifugation at 6000 × *g* for 10 min and then stored in the dark at 0 °C. All experiments in this study were carried out in triplicate.

2.2.2. SD extraction of essential oils from peel

Calamondin peels (150 g) were homogenized (Waring Blender Model HGB7WTS3, Waring Co., Torrington, Connecticut 06790, USA) for 2 minutes with 600 mL deionized water and placed into a 5 L round bottom flask. The homogenate was steam distilled [10] for 2 hours to obtain the essential oils, which were then stored in the dark at 0 °C.

2.2.3. SD extraction of essential oils from whole fruit

Whole fruits (1 kg) were homogenized for 2 minutes with 1 L deionized water in a 5 L round bottom flask. The homogenate was steam distilled [10] for 2 hours to obtain the essential oils, which were then stored in the dark at 0 °C.

2.2.4. Extraction of essential oils from peel after HWH

Peels (150 g) were heated in 90 ± 3 °C water for 15 minutes, homogenized for 2 minutes with 600 mL deionized water in a 5 L round bottom flask followed by SD for 2 hours to obtain the essential oils, which were then stored in the dark at 0 °C.

2.2.5. Extraction of essential oils from whole fruit after HWH

Whole fruits (1 kg) were heated in hot water at 90 ± 3 °C for 15 minutes, homogenized for 2 minutes with 1 L of distilled water. The homogenate was steam distilled in a 5 L round bottom flask for 2 hours to obtain the essential oils, which were then stored in the dark at 0 °C.

2.3. Analysis of volatile compounds

2.3.1. Direct injection analytic method

One microliter of each essential oil sample was injected into a gas chromatography injection unit in split mode with a ratio of 1:100.

2.3.2. Analysis of the components of the essential oils by GC–flame ionization detection

Qualitative and quantitative analyses of the volatile compounds were conducted using an Agilent 6890 GC equipped with a DB-1 fused-silica capillary column (60 m × 0.25 mm internal diameter) with a film thickness of 0.25 μm and a flame ionization detector. Injector and detector temperatures were maintained at 250 °C and 300 °C, respectively. The oven

temperature was held at 40 °C for 1 minute and then raised to 200 °C at 2 °C/minutes and held for 9 minutes. The carrier gas (nitrogen) flow rate was 1 mL/minute. Kovats indices were calculated for the separated components relative to mixture of a C5–C25 *n*-alkanes [11]. Percentage composition was calculated using the peak area normalization measurements.

2.3.3. *Analysis of the components of the essential oils by GC/MS*

Identification of the volatile compounds was conducted using an Agilent 6890 GC equipped with a DB-1 fused-silica capillary column (60 m × 0.25 mm internal diameter) with a film thickness of 0.25 μm and an Agilent model 5973 N MSD mass spectrometer. The injector temperature was maintained at 250 °C. The GC conditions in the GC/MS analysis were the same as the GC analysis described above. The carrier gas (helium) flow rate was 1 mL/min. The electron energy was 70 eV at 230 °C. The constituents were identified by matching their spectra with those recorded in the MS library (Wiley 7n). In addition, the constituents were confirmed by using the Kovats indices or GC retention time data with those of authentic standards or by publication literature.

3. Results and discussion

3.1. *Constituents of calamondin essential oils*

3.1.1. *Yields of essential oils*

The essential oil yields of calamondin peel and whole fruit are shown in Table 1. The results indicated that HWH increased the yields of essential oils for both peels and whole fruits when compared to SD alone. HWH may cause tissues damage in the peel or even in the flesh, which thus releases volatile compounds. Peng et al [12] studied the effects of heat treatments on kumquat essential oils and reported that hot water heating increases the yields of essential oils. When compared with the results obtained by Maarse and Visscher [13], a high degree of aroma composition similarity was found in the calamondin and kumquat essential oils. Takeuchi et al [6] reported that the yield of essential oils extracted from calamondin peel with hexane coupled with dehydration for 24 hours is 1.92%. Umano et al [14] found that the yields of Japanese kumquat essential oil obtained by SD and solid phase extraction are 0.011% and 0.019%, respectively. Cheong et al [8] reported that the yield of volatiles obtained in hexane peel extracts is relatively higher than those in dichloromethane peel extracts. In the present study, we found that the yield of essential oils obtained by cold pressing (CP) from calamondin peel is relatively low. The thin peel of calamondin may make the destruction of peel oil droplets by extrusion or other methods difficult, thereby leading to low oil yield. The difference in yields of essential oils between the present study and those from others may be attributable to the differences in various extraction methods and extractions by polar or nonpolar organic solvents.

Table 1 – Yield of essential oils from calamondin fruits and peels extracted using different methods.

Fruit	Extraction methods	Yield (g/kg)[a]
Calamondin	CP/P	0.81 ± 0.25
	SD/P	7.11 ± 0.08
	HWH/SD/P	9.13 ± 0.12
	SD/F	0.80 ± 0.10
	HWH/SD/F	1.01 ± 0.09

CP/P = cold-pressing of peel; HWH/SD/F = whole fruit after hot water treatment followed by steam distillation; HWH/SD/P = peel after hot water treatment followed by steam distillation; SD/F = steam distilled whole fruit; SD/P = steam distilled peel.
[a] Yield: mean ± SD of triplicates.

3.1.2. *Constituents of essential oils*

3.1.2.1. *Analysis of calamondin essential oils.*

The volatile compounds in the calamondin essential oils were analyzed by direct injection (DI), coupled with both GC–flame ionization detection (GC/FID) and GC/MS. Table 2 shows a total of 54 compounds that were identified by DI/GC, including 13 monoterpene hydrocarbons, 7 monoterpene alcohols, 1 monoterpene oxide, 4 monoterpene aldehydes, 2 monoterpene ketones, 4 monoterpene esters, 12 sesquiterpenes, 3 aliphatic alcohols, 6 aliphatic aldehydes, and 2 aliphatic esters. The major volatile components of the oils extracted from peel by CP, SD, and HWH followed by SD were limonene (91.15–92.70%), myrcene (2.08–2.46%), and germacrene D (0.54–1.21%). The major compounds identified from whole fruit essential oils (HWH/SD) were limonene (89.28%), β-myrcene (2.72%), and α-terpineol (1.92%).

As shown in Table 2, the main volatile constituents of calamondin peel and whole fruit according to DI/GC/FID and GC/MS analyses were monoterpenes (93.79–95.80%; Table 3). Decanal is responsible for the important aromatic notes in sweet oranges, and linalool has a floral scent. Compared to previous studies in calamondin peel, Moshonas and Shaw [4] showed that the major component of calamondin peel extracted with hexane is limonene and the second major component is β-pinene. Takeuchi et al [6] identified that the major component of calamondin peel extracted with hexane is limonene and the second major compounds are γ-terpinene and β-elemene. In addition, Cheong et al [8] determined that the major constituents of calamondin peel isolated with hexane are limonene and β-myrcene, and the second major compounds are β-pinene, linalool, and α-pinene.

3.1.2.2. *Comparison analysis of essential oil compositions of calamondin peel and whole fruits.*

As shown in Table 2, the main constituents of calamondin peel and whole fruits essential oils according to DI/GC/FID and GC/MS analyses were monoterpenes. Table 3 shows that the peel oils had a high content of monoterpenes and sesquiterpenes but a lower content of terpene alcohols than the whole fruit oils had. Teranishi and Buttery [15] reported that certain hydrocarbon compounds, such as linalool, geraniol, and α-terpineol, as well as their hydrocarbon interactions can be interrupted by heat stress resulting in the induction of volatilization. Additionally, α-terpineol in the calamondin whole fruits essential oil was higher than that in the peel essential oil whereas citronellal in the peel essential oil was significantly higher than that in the calamondin whole fruits essential oil.

Table 2 – Means and standard deviations of volatile compounds found in the essential oils of calamondin analyzed by direct injection into gas chromatography–mass spectrometry or gas chromatography–flame ionization detection.

Compound	RI	Contents (%)			
		CP/P	SD/P	HWH/SD/P	HWH/SD/F
Monoterpene hydrocarbons					
α-Thujene	925	<0.01	<0.01	<0.01	<0.01
α-Pinene	941	0.53 ± 0.01	0.41 ± 0.06	0.42 ± 0.03	0.40 ± 0.01
Camphene	950	<0.01	0.01 ± 0.00	0.01 ± 0.00	0.04 ± 0.01
Sabinene	967	0.16 ± 0.01	0.17 ± 0.02	0.21 ± 0.04	0.09 ± 0.03
β-Pinene	971	0.23 ± 0.01	0.49 ± 0.13	0.55 ± 0.22	0.52 ± 0.49
β-Myrcene	983	2.08 ± 0.04	2.36 ± 0.11	2.46 ± 0.01	2.72 ± 0.11
α-Phellandrene	996	0.04 ± 0.01	0.04 ± 0.00	0.06 ± 0.02	0.10 ± 0.04
δ-3-Carene	1004	<0.01	<0.01	0.05 ± 0.02	0.04 ± 0.01
p-Cymene	1014	0.03 ± 0.00	0.06 ± 0.02	0.05 ± 0.01	0.12 ± 0.04
Limonene	1030	92.70 ± 0.13	91.45 ± 0.95	91.15 ± 1.78	89.28 ± 1.73
β-Ocimene	1040	0.03 ± 0.00	0.03 ± 0.01	0.04 ± 0.00	0.05 ± 0.00
γ-Terpinene	1051	<0.01	0.05 ± 0.01	0.18 ± 0.03	0.13 ± 0.01
Terpinolene	1085	—	0.02 ± 0.01	<0.01	0.30 ± 0.12
Terpene alcohols					
Linalool	1087	0.16 ± 0.06	0.98 ± 0.01	1.08 ± 0.18	0.80 ± 0.21
Terpinen-4-ol	1162	0.07 ± 0.01	0.08 ± 0.02	0.11 ± 0.02	0.31 ± 0.10
α-Terpineol	1174	0.10 ± 0.03	0.36 ± 0.04	0.42 ± 0.02	1.92 ± 0.49
Carveol	1189	—	0.05 ± 0.01	0.09 ± 0.02	0.09 ± 0.03
Nerol	1203	<0.01	<0.01	—	<0.01
Geraniol	1311	—	—	—	0.03 ± 0.01
α-Eudesmol	1653	0.18 ± 0.05	0.23 ± 0.05	0.23 ± 0.04	0.25 ± 0.03
Terpene oxide					
Limonene oxide	1134	<0.01	—	<0.01	<0.01
Terpene aldehydes					
Citronellal	1129	0.01 ± 0.00	0.05 ± 0.01	0.08 ± 0.03	—
Geranial	1197	<0.01	0.01 ± 0.00	0.07 ± 0.04	0.06 ± 0.01
Neral	1233	0.02 ± 0.00	0.02 ± 0.00	—	0.11 ± 0.03
Perillaldehyde	1241	0.03 ± 0.00	0.08 ± 0.04	0.11 ± 0.01	0.09 ± 0.02
Terpene ketones					
Camphor	1118	—	0.02 ± 0.00	0.02 ± 0.01	0.02 ± 0.01
Carvone	1209	0.03 ± 0.01	0.09 ± 0.01	0.09 ± 0.01	0.08 ± 0.02
Terpene esters					
Methyl salicylate	1195	—	<0.01	—	<0.01
Carvyl acetate	1337	<0.01	<0.01	<0.01	<0.01
Geranyl acetate	1362	0.23 ± 0.01	0.31 ± 0.09	0.41 ± 0.07	0.26 ± 0.08
Methyl *N*-methanthranilate	1380	—	<0.01	—	<0.01
Sesquiterpenes					
δ-Elemene	1331	0.11 ± 0.01	0.09 ± 0.04	0.09 ± 0.04	0.10 ± 0.03
α-Copaene	1378	0.06 ± 0.02	0.09 ± 0.03	<0.01	0.05 ± 0.01
β-Elemene	1382	0.07 ± 0.02	0.04 ± 0.01	0.05 ± 0.01	0.01 ± 0.00
β-Caryophyllene	1430	0.04 ± 0.00	0.02 ± 0.00	0.03 ± 0.01	<0.01
α-Humulene	1448	0.03 ± 0.00	<0.01	0.01 ± 0.00	<0.01
Germacrene-D	1473	1.21 ± 0.04	0.58 ± 0.21	0.54 ± 0.07	0.17 ± 0.03
Bicyclogermacrene	1489	<0.01	0.04 ± 0.01	<0.01	<0.01
α-Selinene	1490	—	0.09 ± 0.03	—	<0.01
β-Selinene	1492	—	0.08 ± 0.03	0.06 ± 0.02	0.01 ± 0.00
γ-Cadinene	1508	—	0.02 ± 0.00	<0.01	—
δ-Cadinene	1526	0.13 ± 0.01	0.07 ± 0.02	0.07 ± 0.01	0.01 ± 0.00
Valencene	1550	—	0.03 ± 0.01	<0.01	<0.01
Aliphatic alcohols					
1-Octanol	1055	0.03 ± 0.00	0.25 ± 0.05	0.14 ± 0.04	0.03 ± 0.00
1-Nonanol	1153	—	<0.01	<0.01	0.40 ± 0.10
1-Decanol	1266	<0.01	<0.01	<0.01	—
Aliphatic aldehydes					
Heptanal	878	<0.01	0.02 ± 0.01	0.01 ± 0.00	0.02 ± 0.01
Nonanal	1073	0.05 ± 0.02	0.26 ± 0.09	0.25 ± 0.08	0.31 ± 0.03
Decanal	1181	0.08 ± 0.04	0.16 ± 0.04	0.25 ± 0.06	0.25 ± 0.01
2-Decenal	1265	0.03 ± 0.00	0.03 ± 0.01	0.05 ± 0.01	0.09 ± 0.02
2,4-Decadienal	1270	<0.01	0.12 ± 0.01	0.20 ± 0.04	0.16 ± 0.04
Undecanal	1284	0.10 ± 0.01	0.02 ± 0.01	0.12 ± 0.02	0.11 ± 0.05

Table 2 – (continued)

Compound	RI	Contents (%)			
		CP/P	SD/P	HWH/SD/P	HWH/SD/F
Aliphatic esters					
Heptyl acetate	1080	—	—	<0.01	—
Octyl acetate	1189	—	0.10 ± 0.05	0.09 ± 0.03	0.05 ± 0.01

Data are presented as are means ± SD of triplicates.
— = undetectable; RI = retention indices, using paraffin (C5-C25) as references; CP/P = cold-pressing peel; HWH/SD/F = whole fruit after hot water treatment followed by steam distillation; HWH/SD/P = peel after hot water treatment followed by steam distillation; SD/P = steam distilled peel.

Thus, we speculated this compound is mainly present inside the fruit peel.

3.2. *Comparison of peel oils prepared from cold pressed and steam distilled calamondins*

Table 1 shows that CP resulted in lower essential oil yields than steam distillation did. According to Asikin et al [16], the SD system resulted in higher extraction yield and extraction of a greater number of volatile aroma compounds than the CP method did, which agrees with the present study. As shown in Table 2, the main components of calamondin essential oils extracted from peel by CP and analyzed by DI/GC/FID contained more monoterpenes compared to the calamondin essential oil extracted from peel of fruit by SD, with or without HWH. The oxygenated monoterpenes, terpene alcohol, terpene ester, terpene aldehyde, and terpene ketone, found in the essential oil extracted by CP were significantly less than those found in the essential oil extracted by SD. It is likely that monoterpenes are easily destroyed and oxygenated by heat stress so that a high amount of monoterpenes were found in the calamondin essential oil isolated by CP. Ferhat et al [17] reported that cold pressing gave lower isolation yields and lower oxygenated compounds. McGraw et al [18] observed that heat stress induced oxidative degradation of limonene resulting in the formation of monoterpene hydrocarbons or oxygenated monoterpenes. Moreover, we also noticed that a high amount of linalool was found in the essential oils from calamondin peel or the whole fruit isolated by SD compared to the calamondin essential oil isolated by CP. Likewise, Umano et al [14] showed that linalool is found in a higher concentration in the essential oil extracted by steam distillation than that by the simultaneous purging and extraction method. Additionally, Table 2 shows that the sesquiterpenes found in the essential oil extracted by CP was markedly higher than those in the essential oil extracted by SD. It is possible that compounds of relatively high molecular weight, such as germacrene D or sesquiterpenes, were found in a higher amount in the essential oil extracted by CP due to the nondistillation method.

Swisher and Swisher [19] reported that distilled oil, which is recovered from peels by SD, possesses an odor and flavor that are generally inferior to those of cold pressed oil. McGraw et al [18] observed that terpene compounds (camphene, δ-carene, limonene, and α-terpinene) degraded at high temperatures and turned into other terpene hydrocarbons and oxide terpenes. Perez-Cacho et al [20] reported that off-flavors are caused by compounds such as *p*-cymenes and carvones. The α-terpineol component is formed by oxidative degradation of limonene and is well known for its contribution to the off-flavor of orange juice [21].

Table 3 – Percentages of chemical groups of calamondin essential oils analyzed by direct GC-injection/GC/FID/GC/MS.

Compound	Contents (%)			
	CP/P	SD/P	HWH/SD/P	HWH/SD/F
Monoterpenes	95.80	95.09	95.18	93.79
Terepene alcohols	0.51	1.70	1.93	3.40
Terpene oxides	<0.01	—	<0.01	<0.01
Terpene aldehydes	0.06	0.16	0.26	0.26
Terpene ketones	0.03	0.11	0.11	0.10
Terpene esters	0.23	0.31	0.41	0.26
Sesquiterpenes	1.65	1.15	0.85	0.35
Aliphatic alcohols	0.03	0.25	0.14	0.43
Aliphatic aldehydes	0.26	0.61	0.88	0.94
Aliphatic esters	—	0.10	0.09	0.05

— = undetectable; CP/P = cold-pressing peel; HWH/SD/F = whole fruit heated in hot water followed by steam distillation; HWH/SD/P = peel heated in hot water followed by steam distillation; SD/P = steam distilled peel.

3.3. *Comparison of hot water treatment effects on the volatile constituents of calamondin essential oils*

The comparison of volatile constituents of the essential oils from the hot water-treated calamondin peel, and non–hot water-treated calamondin peel by DI/GC/FID and GC/MS is shown in Table 2. The results showed that the quantities of some monoterpenes present in these two essential oils increased under hot water treatment, such as sabinene and oxygenated monoterpene monohydrates, including linalool and α-terpineol. However, the major compositions of these essential oils were almost identical regardless of hot water treatment. The data suggested that the effects of hot water on volatile constituents of calamondin essential oils were minimal due to the short period of treatment. Schirra et al [22] determined that there was no significant difference in the volatile components of the essential oils from hot water-treated kumquat, excepting for an increase in *p*-menta-1,5-dien-1-ol. However, *p*-menta-1,5-dien-1-ol was not detected in the present study. Different samples are expected to lead to different volatile profiles.

4. Conclusion

The results indicate that hot water heating increased the yields of essential oils from both whole fruit and peel. The whole fruit oil also contained higher levels of monoterpene alcohols, such as linalool, terpinen-4-ol, and α-terpineol, which may contribute to the aroma profile of fruit tea. The peel oil had higher contents of sesquiterpenes, such as germacrene D, than whole fruit oils had. The volatile constituents in calamondin fruit tea are reported here for the first time.

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