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Extraction of essential oil from lemon and orange peel by Clevenger apparatus: Comparative GC_MS analysis of chemical composition, from Debre Berehan Market town Amahara Region Ethiopia

***Corresponding Author(s): Zelalem Zayse Zeleke**

Wachemo University, Durame campus, Department of chemistry, Ethiopia.

Email: Zelalemzayse29@gmail.com

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Abstract

The peel of orange and lemon fruits is considered as a waste. It is used as a source of essential oils that can be applicable for food flavoring agent, pharmaceuticals, cosmetics, and soap and detergent factories. In this work, essential oils were extracted from lemon (1.56%) and orange (3.55%) peels by hydro distillation. The physiochemical properties of oils were recorded. The chemical composition of the oils was also analyzed by GC-MS. Orange peel oil contained four compounds with limonene (95.19%) as major component and lemon peel oil composed of 12 compounds of which limonene (49.74%) was the main compound.

Introduction

Citrus (family *Rutaceae*) is one of the most widespread genera all over the world due to its prominent production. It comprises 140 genus and 1,300 species, *Citrus sinensis* (sweet orange), *Citrus limon* (lemon), *Citrus paradis* (grape fruits), *Citrus medica* (Citron), *Citrus aurantifolia* (lime), *Citrus grandis* (shaddock), *Citrus reticulate* (tangerine) and *citrus aurantium* (sour orange) are some of important fruits of genus citrus [1-8]. Citrus fruits are very popular in many parts of the world because of their distinctive taste and aroma as well as multiple health benefits to mankind [9]. These plants grow in tropical and subtropical regions of the world. Orange and lemon trees lemon are the three most important and abundant citrus species in the world [10,11].

Most of *citrus* plants are planted for consuming freshly at home. Orange and lemon fruits are consumed fresh or industrially processed as juice [12-15]. The juice industries produce mainly juices from the endocarp or pulp parts of citrus fruits. However, the industries discard byproducts including peels or flavedo, albedo, seeds and pulp residues as waste. The waste having more thanhalf of the original weight of the fruit is disposed outside their compound which has high potential to affect the environment. All byproducts of *citrus* plants may have importance if they are recommended to use them based on research findings [16,17].

The chemical compositions of the plants are mainly affected by the part of plant analyzed, growing condition, variation of species, plant pretreatment, extraction methods, place of ori-

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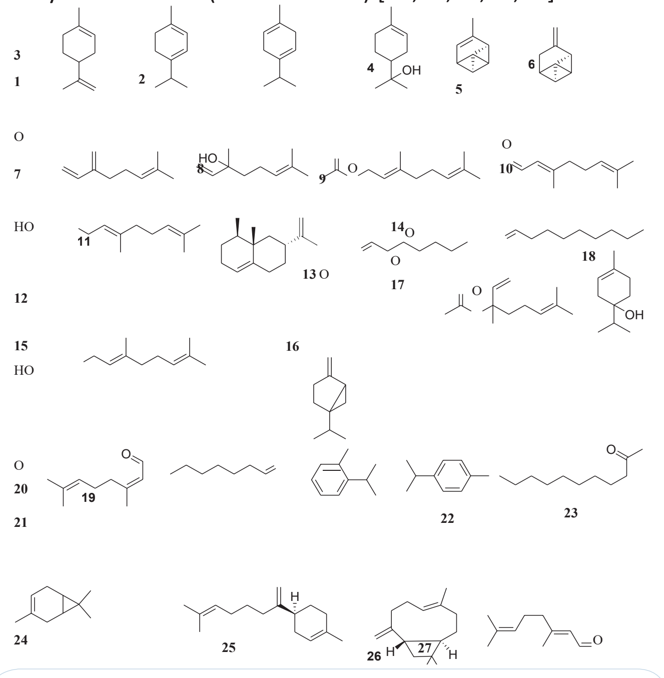
gin, maturity. The chemical constituents may present in all parts of the plant but the amount varies from one organ of plant to another [18-20].

Citrus fruits and their peels are the rich sources of vitamins, minerals, sugars, carbohydrates, fats, proteins, organic acids, dietary fibers and secondary metabolites like flavonoids, terpenoids,coumarins, alkaloids and essential oils [11]. *Citrus* plants are rich sources of citric acid (vitamin C) which causes their fruits sour due to low pH (2-3) [21-23]. The juices also contain fragrance compounds like hydrocarbons, alcohols, aldehydes, esters, ketones and some oxygenated terpenes [24]. Reportedly, the peels of *citrus* plants contain many biologically active compounds that are extensively applied for flavoring beverages, foods, perfumes, cosmetics, etc. [5,25]. Flavonoids are the major constituents of dry *citrus* fruits as well as citrus peels [17].

Essential oils are volatile odoriferous or aromatic oily liquids extracted from different plants parts. Numerous plants contain essential oils, however, parts of plants, which serve as the major source of essential oil can be different. Plant essential oils are usually the complex mixture of both polar and nonpolar compounds including terpenoids and oxygenated aromatic compounds [26]. The essential oils extracted from the peels of *citrus* peels are complex mixtures; of which 85%-99% are volatile and 1%-15% are non-volatile. The volatile components contain a mixture of monoterpene, sesquiterpene and their oxygenated derivatives but non-volatile compounds contains hydrocarbons, flavonoids, sterols, fatty acids, coumarins, waxes and carotenoids [1,27].

The main chemical components of lemon and orange essential oils are Limonene (**1**), Alpha- Terpene (**2**), Γ-Terpinene (**3**), A-Terpineol (**4**), B-Pinene (**5**), A-Pinene (**6**), Myrcene (**7**), Linalool (**8**), Geraneryl acetate (**9**), Neral (**10**), Nerol (**11**), Valencene (**12**), Octanal (**13**), Decanal (**14**), Geraniol (**15**), Sabinene (**16**), Linalyl Acetate (**17**), Terpinen-4-ol (**18**), Citronella (**19**), Heptanal (**20**), O-cymene (**21**), P-cymene (**22**), 2-Undecanone (**23**), 3-Carene (**24**), Beta-bisaboline (**25**), caryopellene (**26**), citral (**27**). Limonene (**1**) is the major constituent of lemon essential oil as wellas other citrus essential oils. Also lemon oil has some aldehyde molecules (0.13% -1.50%) [18,19,23,24,28].



Scheme 1: Structures of some compounds found in essential oils from the lemon and orange peels.

Different studies reveled that Limonene (**1**) is found in *citrus* essential oils accounting for 32%- 98% [1,27]. It is a colorless to colored liquid monoterpene at room temperature depending on the source; in some fruits it has strong odour. It takes its name from lemon. It exists as (R)- limonene in orange and (S)-limonene in lemon. It is insoluble in water but soluble in alcohol andether which has boiling point of 74^{O}C [19,29].

The peels can be used as possible sources of compounds that can be applicable for the manufacture of chemicals, flavorings and fragrances, paints, cosmetics, and animal feed supplement instead of their disposal. The aim of this study was to investigate the comparative essential oil chemical constituents of the waste peels of orange and lemon by GC-MS.

Experimental procedure

Hydro distillation of fresh orange and lemon peels

The washed lemon and orange peels were crushed in order to reduce their size. The edible portion and white spongy portion (albedo) was separated from the peel by crusher. The crushed samples were used to isolate an essential oil by hydro distillation as follows. Crushed pieces of lemon peels (225 g) were placed in to 1000 ml round bottom flask and 720 ml of distilled water was added to it. After addition of boiling chips the flask was heated slowly for two hours. The distillate forms two layers in the Clevenger apparatus with organic compounds at upper and hydrosol at bottom. The organic phase was collected by small bottles and dried with anhydrous sodium sulfate. The weight of essential oil was measured and the percentage yield of oil was calculated. Similarly crushed pieces (225g) of Lemon peel were subjected to hydro distillation by same procedure above. The same procedure was carried out twice for orange peel extraction essential oils.

GC-MS analysis of essential oils

GC-MS analyses were performed at Addis Ababa University. The instrument was fitted witha fused silica capillary column (30 m x 250 µm, i.d. 0.25 µm film thickness). The oven temperature was programmed from 60°C (2 min) to 240°C (20 min.) at 3°C/min. The injectorand detector temperatures were 250°C and 280^{o}C respectively. Oil samples of 0.2 µl were injected into the GC with a split ratio of 1:50. Helium was the carrier gas at a flow rate of 2 ml/min. The MS was obtained with an ionization voltage of 70 eV, ion source at 230°C. MS datawere acquired in the scan mode in the m/z range 40-650. Identification was made based on a comparison of their retention indices and MS data compared to those from the NIST 107 and Wiley 229 spectral libraries (National Institute of Standards and Technology) [29,30].

Results and discussion

Essential oils were isolated from fresh orange and lemon peels by hydro distillation (Clevenger apparatus) and subjected to GC-MS analysis.

The weight of the essential oils were measured and their percentage were calculated relative to mass of fresh peels of both lemon and orange (Table 1). The average yield of essential oils obtained from lemon and orange peels were determined as 1.56% and 3.55%, respectively. Orange peel has essential oil twice of lemon. Literature shows the closer percentage yield of orange essential oil (3%) [31] With this work.

Table 1: Percentage composition of essential oils.

Part	Lemon weight (g)				Orange weight (g)			
	Trial 1	Trial 2	Trial3	Aver	Trail 1	Trail2	Trail3	Aver
Fresh peel	200.0	200.0	200.0	200	200.0	200.0.	2000.0	200
EO	3.2	2.9	3.3	3.13	7.3	7.1	6.9	7.1
% Yield	1.56%				3.55%			

GC-MS analysis of essential oils of orange and lemon peels

The GC--MS data of lemon essential oil (Table 5) showed the identification of 12 components with monoterpene hydrocarbons (76.56%), aliphatic aldehyde (0.77%), monoterpene aldehydes (14.39%), monoterpene alcohols (2.3%), sesquiterpenes (4.97%) and ketone (1.02%) as main constituents. The major monoterpenes limonene (49.74%), β-pinene (17.13%), γ-terpinene (7.52), o-cymene (2.17%), sesquiterpenes of β-bisabolene (2.4%), β-caryophyllene (1.45%),

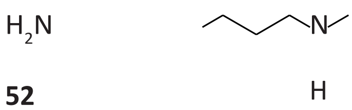
trans α-bergamotene (1.12%), carbonyls citral (14.39%), heptanal (0.77%), 2-undecanone (1.02%), and alcohols α-terpineol (1.44%) and terpinen-4-ol (0.86%) were detected. Terpenes particularly monoterpenes were dominant components in the lemon essential oils.

Table 2: Chemical composition of lemon and orange essential oils.

No	Components	Percentage composition	
		Lemon peel (%)	Orange peel (%)
1	beta- pinene (**5**)	17.13	-
2	d-limonene (**1**)	49.74	95.19
4	o-cymene (**21**)	2.17	-
5	gamma- terpinene(**3**)	7.52	-
6	terpinen-4-o1 (**18**)	0.86	-
7	Heptanal (**20**)	0.77	-
8	alpha-terpineol (**4**)	1.44	-
10	2-undecanone (**23**)	1.02	-
11	citral (**27**)	14.39	-
12	trans-alpha-bergamotene (**53**)	1.12	-
13	Caryophyllene (**26**)	1.45	-
14	beta-bisaboline (**25**)	2.4	-
15	beta-myrcene (**27**)	-	1.09
16	n-methyl-1,3-propanediamine (**52**)	-	2.88
17	3-carene (**24**)	-	0.83

Four compounds were detected in orange peel essential oil (99.99%), namely, monoterpenes (97.11%) and N-methyl-1, 3-propanediamine (2.88%). The monoterpenes were limonene (95.19), beta-myrcene (1.09) and 3-carene (0.83). The major and common component of both essential oils was limonene (32%-98%) as reported in [1].

The differences in chemical constituents of essential oil were assumed due to their genetic characteristics [1,30,32,33,34].



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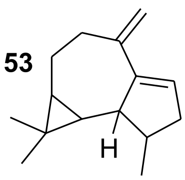

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Conclusion and recommendations

This work was intended to comparative study and chemical compositions of lemon and orange peels in the area as an alternative method to reduce municipal wastes by converting these waste materials into useful products. The percentage yield of peel essential oil from orange (3.55%) was higher than that of lemon essential oil (1.56%). The chemical compositions of the essential oils isolated from orange and lemon peels, as identified by GC-MS analysis, were dominated by limonene. The chemical composition of orange peel essential oil includes limonene (95.19%), 3,3-dimethyl-4-methylamino-2-one (2.88%), beta-myrcene (1.09%) and 3-carene (0.83%)whereas the lemon peel essential oil was compose of limonene (49.74%), beta-pinene (17.13), citral (12.39%), gamma-tepinene (7.52%), beta-bisaboline (2.4%) and o-cymene (2.17%).

Recommendation

For further study it is important to isolate the major component by different isolation technique and elucidate its structure and cover comparative bioassay studies.

Conflict of interest: The Author has no conflict of interest.

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