



RESEARCH PAPER

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Comparative volatile components examination of various citrus peel essential oils

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Key words: Essential oil, Citrus peels, Limonene, α -terpineol.

<http://dx.doi.org/10.12692/ijb/13.4.262-266>

Article published on October 30, 2018

Abstract

Volatile components of various citrus peel essential oils were determined in four varieties of citrus i.e. *citrus limetta* var. mitha, *citrus aurantifolia* var. kaghzi nimbu, *citrus reticulata* var. tangerine and *citrus sinensis* variety mosammi were extracted by hydro distillation method. Eleven constituents were identified in *citrus sinensis*, eleven in *citrus reticulata*, sixteen in *citrus limetta* and thirteen in *citrus aurantifolia*. The % age of limonene in *citrus limetta*, *citrus aurantifolia*, *citrus reticulata* and *citrus sinensis* were 95.97%, 82.84%, 93.70% and 94.78% respectively. The other components i.e. β -pinene, α -terpineol were also present in all four varieties.

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Introduction

Citrus is one of the most important fruit crops grown in all over the world (Tao *et al.*, 2007). Citrus belongs to family "Rutaceae" which has 150 genera and 1600 species distributed through out the mediterranean, tropical and sub-topical region of the world (Swingle., 1967, Tasneem 1995). The genus citrus has 17 species Distributed throughout the tropical and temperate regions (Davis & Albrigo, 1994, Shaw, 1977, Chutia M. *et al.*, 2009). Essential oil is one of the major by product of citrus which attracting the people (Njoroge *et al.* 2005, Neng-guo *et al.*, 2008). Volatile oils known as essential oils are lipophilic compounds containing volatile aroma compounds (Ayoola *et al.* 2008, Fayed *et al.*, 2009).

Essential oil constituents are basically complex mixture of terpenic hydrocarbons and oxygenated derivatives such as aldehydes, alcohols and esters. The citrus fruit peels are the most familiar and rich source of essential oils. (Ahmad, M. *et al.*, 2006). Peels of citrus fruits comprise of two layers, red outer layer as flavedo and inner white layer as albedo (Nagi *et al.*, 1977). The flavedo layer contains essential oils in the range of 0.5 to 3.0 Kg/ton of fruit (Sattar *et al.*, 1992).

Essential oils of the fruits are grouped into two fraction a volatile fraction constituents 90-95% of the whole oil and contains monoterpenes, aliphatic sesquiterpenes and oxygenated derivatives, bicyclic terpenes and sesquiterpenes, aromatic hydrocarbons, aliphatic aldehydes, alcohols and esters. Non-volatile residue constitutes from 1-10% of the whole oil and contains fatty acids, esters, carotenoids, coumarines psoralens, paraffine waxes, flavonoids and steroids, which accumulate in ballon like cells (oil glands) granular trichomas and oil or resin ducts (Clifford *et al.*, 1999, Ahmad *et al.*, 2006, Diaz *et al.* 2004).

The composition of the essential oil is effected by the ripens of fruits, vegetative stage of plants, storage conditions and extraction method (Njoroge, *et al.*, 2006, Venkateshwarlu, *et al.*, 2000). The main objective of this study was to determine and compare the volatile components of the various citrus peel essential oil found in Pakistan.

Materials and methods

Extraction of essential oils

The fresh citrus fruits were collected from citrus garden near Renala khurd in the Punjab province of Pakistan. The peels were off from the fruits and cut into small pieces.

The essential oil was extracted through hydro-distillation by reverse Dean Stark assembly. The steam distilled essential oil was removed, dried over anhydrous sodium sulphat and stored in an ambered colored bottle at low temperature (Sattar A.1989).

Gas chromatographic analysis

Volatile constituents of the essential oils were determined by Jeol model IMX-AX500H mass spectrometer combined with Hewlett Packard 5890 gas chromatograph was used for GC-MS analysis Oil sample was injected on a 25mx0.22mm WCOT BP5 (5% phenyl, 95% dimethyl siloxane) fused silica column, using Helium as a career gas with split ratio 1:10, EI positive mode, electron energy 70 + ev, ionization currant 300µA, ionization source temperature 250°C, interface temperature 230°C, column temperature programmed at 60°C for 4 min., with a 6°C/min rise to 220°C.

Data acquisition and processing were performed by JEOL JMA-DA 5000 system various components were identified by their retention time and peak enhancement with standard samples in gas chromatographic mode and Nist library search from the derived fragmentation pattern of the various components of the essential.

Results and discussion

The volatile components which are identified in various citrus peel essential oil by GC-MS are described in table 1. The % age yield of essential oil in mosammi, tangerine, mitha and kaghzi nimbu is 0.10%, 1.31%, 0.15% and 0.51% respectively which is quite encouraging as compared to the yield reported by G.A Ayoola 2008 (0.005%), Rondeau *et al* 2003 (0.03-0.08%), Sattar *et al.* 1992 (0.014%), Ahmad *et al.* 2006 (0.98%) in these species.

Table 1. GC analysis of volatile components of Kaghzi Nimbu, Mosammi, Tangerine and Mitha

SN	Components	Citrus aurantifolia var kaghzi nimbu	Citrus sinensis var mosammi	Citrus reticulata var tangerine	Citrus limetta var mitha.
1.	α -pinene	-	0.13%	-	0.38%
2.	β -pinene	0.86%	1.41%	2.00%	0.02%
3.	α -terpinene	-	-	0.47%	0.33
4.	β -terpinene	0.61%	-	0.46%	-
5.	Myrcene	-	0.36%	0.32%	-
6.	Limonene	82.84%	94.78%	93.70%	95.97%
7.	ρ -cymene	-	-	0.19%	0.38%
8.	α -terpineol	0.39%	0.062%	0.44%	0.31%
9.	Linayl acetate	-	-	0.12%	-
10.	δ -Cadinene	0.18%	-	0.60%	-
11.	α -thujene	0.16%	0.29%	-	0.06%
12.	Geraniol	0.09%	0.29%	-	0.36%
13.	γ - terpinene	-	0.21%	-	0.03%
14.	3- carene	0.01%	-	-	0.17%
15.	4-terpineol	0.39%	-	-	-
16.	α - cedrene	0.18%	-	-	-
17.	Octanol	-	0.17%	-	-
18.	Decanol	-	0.14%	-	-
19.	Neval	-	-	-	0.29%
20.	α -humulene	-	-	-	0.05%
21.	β -bisabolene	-	-	-	0.12%
22.	trans carveol	0.33%	-	-	-
23.	Gernayl alcohol	0.11%	-	-	-
24.	7-octadecanal	-	-	0.01%	-
25.	geranial	-	-	0.03%	-
26.	citronellol	-	0.21%	-	0.09%
27.	moslene	-	-	0.43%	-

In Mosammi oil 11 components are identified. The oil has high contents of monoterpene hydrocarbons (97.18%) with limonene (94.78%), α -pinene (0.13%), β -pinene (1.41%), Myrcene (0.36%), α -thujene (0.29%), γ - terpinene (0.21%). The major oxygenated component (1.74%) of the oil were found; aldehyde components (0.31%); decanal (0.14%), octanal (0.17%); alcohol components (0.56%); α -terpineol (0.06%), geraniol (0.29%), citronellol (0.21%).

In Tangerine oil 11 components are identified .The oil has high contents of monoterpene hydrocarbons (97.14%) with limonene (93.70%), β -pinene (2.00%), Myrcene (0.32%), α -terpinene (0.47%), β -terpinene (0.46%), ρ -cymene (0.19%); sesquiterpene components (1.03%); δ -Cadinene (0.60%), moslene (0.43%). The major oxygenated component (1.14%) of the oil were found; aldehyde components (0.01%); 7-octadecanal (0.01%); alcohol components (0.44%); α -terpineol (0.44%); ester components (0.12%); linyl acetate (0.12%). In Mitha oil 16 components are identified .The oil has high contents of monoterpene

hydrocarbons (96.99%) with limonene (95.97%), α -pinene (0.03%), β -pinene (0.02%), α -thujene (0.06%), γ - terpinene (0.33%), ρ -cymene (0.38%), γ -terpinene (0.03%), 3-carene (0.17%); sesquiterpene components (0.17%); α -humulene (0.05%), β -bisabolene (0.12%). The major oxygenated component (1.08%) of the oil were found; aldehyde components (0.32%); gernal (0.03%), neral (0.29%); alcohol components (0.76%); α -terpineol (0.31%), geraniol (0.36%), citronellol (0.09%).

In kaghzi nimbu oil 13 components are identified. The oil has high contents of monoterpene hydrocarbons (%) with limonene (82.84%), β -pinene (0.86%), α -thujene (0.16%), β -terpinene (0.61%), 3-carene (0.01%); sesquiterpene components (%); α -cedrene (0.18%), δ -Cadinene (0.18%); The major oxygenated component (1.31%) of the oil were found following; alcohol components (1.31%); α -terpineol (0.39%), geraniol (0.09%), 4-terpineol (0.39%), trans carveol (0.33%), Gernayl alcohol (0.11%). The limonene is the major component in Mosammi, Tangerine, Mitha and

Kagzi nimbu and the % age of Limonene is 94.78%, 93.70 %, 95.97%, and 82.84% respectively. Mitha has a high percentage of limonene as compared to other species. In previous studies the limonene is reported as major component in all citrus oils (Minh *et al.*, 2002, Choi, H.S. *et al.*, 2002, Njorge *et al.*, 1994, Masayoshi *et al.*, 2004, Mao, Y *et al.*, 2004 and Sattar *et al.* 1987).

The oxygenated components contributes to the flavor of oil .the alcohol,aldehyde and ether may contribute to the aroma of extracted oil (chisolam *et al.*, 2003). The % age of oxygenated components present in Mosammi, Tangerine, Mitha and Kagzi nimbu is 1.74%, 1.14%, 1.08%, 1.31% respectively.

Conclusion

Essential oils from the four citrus varieties i.e. *citrus limetta* var. mitha, *citrus aurantifolia* var. kaghzi nimbu, *citrus reticulata* var. tangerine and *citrus sinensis* variety mosammi were extracted by hydro distillation method. Their chemical constituents were determined by GC-MS and found eleven constituents in *citrus sinensis*, eleven in *citrus reticulata*, sixteen in *citrus limetta* and thirteen in *citrus aurantifolia*.

The % age of limonene in *citrus limetta*, *citrus aurantifolia*, *citrus reticulata* and *citrus sinensis* were 95.97%, 82.84%, 93.70% and 94.78% respectively. Due to the presence of oxygenated components the oil is recommended for flavoring agents in beverages and food products.

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