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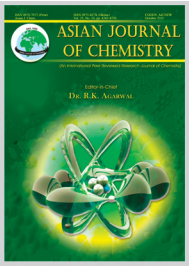

NOTE

Gas Chromatography/Mass Spectrometry for Separation and Identification of Cold-Pressed Compounds in Grapefruit Essential Oils from Iran

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The grape fruits, (*Citrus paradisi* macfad.), were collected in December from Jiroft, Kerman province (eastern south of Iran). The essential oils after cold-press were analyzed by gas chromatography/mass spectometry. Twenty-seven compounds present in the non-volatile residue of the essential oils of grape fruit were analyzed by a gas chromatography/mass spectrometry. Use of hyphenated techniques, such as gas chromatography/mass spectometry provides great information about the content and nature of constituents of natural complex matrices, such as essential oils. The different responses of the identified compounds have been also evaluated and the Quvats index of these components was compared with the standard samples. This method is used to confirm the identified components of essential oils. Limonene was the main constituents of the essential oils (95 %). Some new compounds were found, together with many others already identified in different grape fruit essential oils.

Key Words: Grapefruit, Essential oil, Limonene, GC/MS, Cold-press.

Grape fruit is a fruit (*Citrus paradisi* macfad.), which belongs to citrus (Rutaceas) family[1]. The fruit and its extracted compounds contain active components such as flavonoids, fruit acids, emulsion and large amount of vitamin C, K and also is a good source of Fe, Ca and other inorganic materials. Pink and red kinds of grape fruit have large amount of β-carotene, pro-vitamin A and is helpful to cure the cellulite[2,3].

In this work, a satrun model 3400 gas chromatography/mass spectrometry at 70 eV ionization voltages and also a Shimadzu GC model A9 (FID) were used. A centrifuge set model BHG 502, 0-7000 μ/min was also used.

The column used was a DB-1 (60 m × 0.25 mm, film thickness 0.25 μm), which was programmed from 50-250 ºC at 4 ºC/min, with helium as carrier gas. Injector and detector temperatures were 250 ºC and 265 ºC, respectively. The remaining steps in the production of cold-pressed essential oil are: (1) Extraction of oil by mechanical pressure on the oil vesicles; (2) Separating the oil from the peel by water spray; (3) Centrifugation of the water/oil emulsion with acceleration 2000 X g for 15 min at 4 ºC; (4) Clarification of the crude oil in a centrifuge; (5) Settling the waxy crystals present in the oil by holding at low temperatures for several weeks (winterizing) and 6-separating the wax from the oil by centrifugation.

In the present work, retention time and Quvats index of identified compounds have been studied. Mass spectra and all the above parameters are compared with standard ones. Twenty-seven compounds were identified in the emulsion of *Citrus paradisi* prepared by cold-press method in this laboratory. Among all compounds, limonene, myrcene and caryophyllen all have high concentrations and constitute almost 98 % of the oil. Sixteen compounds were identified with analysis of industrial sample prepared by cold-press method. Retention time and Quvats index of identified compounds of both industrial and laboratory samples are given in Tables 1 and 2. In addition, in order to compare the industrial and laboratory samples, their constituting components are summarized in Table-3. It is obvious that aldehyde and alcohol contents in laboratory samples are more than industrial sample. Data for the physical and chemical properties of laboratory sample were: specific gravity (25 ºC): 0.8534, refractive index (25 ºC): 1.4712, optical rotation (25 ºC): +94.00, aldehyde content: 0.24 % and moisture: 72.4 %

Components of grape fruit may impair the activity of intestinal enzymes resulting in clinically important drug interactions. Limonene was a major constituent in both oil emulsions. Limonene has several applications, for example in

TABLE-1
CHEMICAL COMPOSITION OF COLD-PRESS GRAPEFRUIT PEEL OIL (LABORATORY SAMPLE)

No	Compounds	Retention time	Quvats index	(%)
1	α-Pinene	613	941	0.39
2	Sabinene	685	971	0.12
3	β-Pinene	695	975	0.05
4	Myrcene	720	985	1.62
5	α-Phellandrene	751	998	0.04
6	Limonene	821	1026	95.31
7	(E)-β-Ocimene	842	1036	0.08
8	Linalool	944	1079	0.07
9	Decanal	1170	1177	0.21
10	α-Copaene	1584	1370	0.22
11	β-Cubebene	1606	1380	0.28
12	β-Caryophyllene	1670	1412	0.61
13	α-Humulene	1735	1445	0.10
14	Germacrene D	1785	1470	0.22
15	δ-Cadinene	1858	1508	0.28
16	γ-Cadinene	1844	1500	0.02
17	Bicyclogermacrene	1815	1486	0.10
18	Geranyl acetat	1548	1352	0.07
19	Octanol	877	1050	0.02
20	Terpinolene	935	1075	0.01
21	Limonene Oxide (trans)	1028	1115	0.02
22	Nonyl acetate	1190	1186	0.02
23	α-Terpineol	1144	1166	0.02
24	Elemol	1895	1526	0.03
25	Nerolidol (trans)	1918	1538	0.01
26	α-Cadinol	1954	1558	0.03
27	Citronellal	1051	1125	0.03

TABLE-2
CHEMICAL COMPOSITION OF COLD-PRESS GRAPEFRUIT PEEL OIL (INDUSTRIAL SAMPLE)

No	Compounds	Retention Time	Quvats Index	(%)
1	α-Pinene	615	941	0.72
2	Sabinene	687	971	0.22
3	β-Pinene	697	975	0.08
4	Myrcene	721	985	2.25
5	α-Phellandrene	753	998	0.04
6	Limonene	815	1024	94.94
7	(E)-β-Ocimene	843	1036	0.07
8	Linalool	947	1080	0.09
9	Decanal	1173	1179	0.2
10	α-Copaene	1587	1370	0.18
11	β-Cubebene	1609	1381	0.22
12	β-Caryophyllene	1672	1412	0.50
13	α-Humulene	1737	1445	0.07
14	Germacrene D	1787	1470	0.15
15	Valencene	1817	1486	0.07
16	δ-Cadinene	1860	1502	0.20

TABLE-3
COMPARISON BETWEEN COMPOUNDS IN BOTH OIL GRAPEFRUIT SAMPLES (%)

No	Compounds	Industrial sample (%)	Laboratory sample (%)
1	α-Pinene	0.72	0.39
2	Sabinene	0.22	0.12
3	β-Pinene	0.08	0.05
4	Myrcene	2.25	1.62
5	α-Phellandrene	0.04	0.04
6	Limonene	94.94	95.31
7	(E)-β-Ocimene	0.07	0.08
8	Linalool	0.09	0.07
9	Decanal	0.20	0.21
10	α-Copaene	0.18	0.22
11	β-Cubebene	0.22	0.28
12	β-Caryophyllene	0.50	0.61
13	α-Humulene	0.07	0.10
14	Germacrene D	0.15	0.22
15	Valencene	0.07	-
16	δ-Cadinene	0.20	0.28
17	γ-Cadinene	-	0.02
18	Bicyclogermacrene	-	0.10
19	Geranyl acetat	-	0.07
20	Octanol	-	0.02
21	Terpinolene	-	0.01
22	Limonene Oxide (trans)	-	0.02
23	Nonyl acetate	-	0.02
24	α-Terpineol	-	0.02
25	Elemol	-	0.03
26	Nerolidol (trans)	-	0.01
27	α-Cadinol	-	0.03
28	Citronellal	-	0.03

medicine, cosmetic, hygienic industries and also has an important role in food industry.

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