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Extraction and Analysis of Volatile Oil of Foeniulum Vulgare Mill

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Abstract

The volatile oil from dry seeds of Foeniculum vulgare Mill. known in sudan as *Elshamar*, was bought from local market in Buree, Khartoum state, the plant was grown in *Elmanaseer* region in the north of the sudan, extracted by hydrodistillation, the volatile oil yield was found to be 6%(v/w), the extracted volatile oil was analyzed by gas chromatography (GC) and gas chromatography- mass spectrometry (GC-MS) to determine the chemical constituents, the retention indices of the major components were calculated from GC analysis and from GC-MS analysis 28 compounds were identified, the main constituents of volatile oil were found to be: Anethole 31.1%, Phenyl-2-methyl-3-propanol 21.25%, d-Limonene 12.13% ,β-Pinene 9.63% , γ-Terpinene8.03% and 2- Carenal 8.71% and their retention indices are 1237.04, 129-.74, 1021.11, 976.98, 1060 and 1283.33 respectively.

Keywords: Foeniculum vulgareMill; Fennel oil.

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1. Introduction

The plant Foeniculum vulgare Mill., family Umbelleferae, genus foeniculum , species Vulgare, synonyms are Fenkel, sweet fennel and wild fennel, part used are seeds, leaves and roots, the odour of fennel seed is fragrant, its taste, warm, sweet and agreeably aromatic, it yields its virtues to hot water, but more freely to alcohol, the essential oil separated by distillation with water[1], the important constituent in fennel oil in Foeniculum vulgare are Anethole (60%) and fenchone (20%) [2], monoterpenes such as α-phellandrene, α-pinene estragole, anthole and fenchone are detected in fennel oil [3], 21 compounds (12 hydrocarbons and 9 oxygenated) are obtained by steam distillation from fennel oil these 21 compounds are α-pinene, camphene, sabinene, β-pinene, myrcene, α-phellandrene, 3- carene, Ƥ- cymene, limonene, cis-ocimene, trans-ocimene, γ-terpinene, fenchone, camphor, terpnen-4-ol, methyl-carveol, trans-carveol, carvone, anisaldehyde, hexyl acetate and anethole [4].

Chromatography technique is used for the separation, isolation and identification of component of a mixture, the individual components are detect by the property of thermal conductivity this property change are recorded by a resistance thermometer, the result is a plot of signal intensity against time [5]. The retention time, the minute between the time the sample is injected and the time the chromatographic peak is recorded, agreement of retention times of two compound does not quranatee the compounds are identical, the area under the peak is proportional to the concentration and so the amount of the component can be determined. The Kovats retention index is used for identifying a compound from its retention time relative to those of similar compounds in homologous series, those that differ in the number of carbon atoms in a similar structure as in alkane chains [6].

2. Material and Methods

Dry seeds of Foeniculum vulgare Mill. (250gm) were extracted for volatile oil by hydrodistillation method using Clevenger apparatus, extraction was carried out at boiling point temperature of the water for about 4hours, the separated volatile oil was dried over anhydrous sodium sulphate, and subjected to analysis by gas chromatography (GC) and gas chromatography-mass spectrometry (GC-MS). The GC model Varian CP 3800, the analysis condition were as follow: column wall-coated open tubular fused silica, dimethyl poly siloxane 50 m length 0.12 mm inside diameter 0.39 mm out side diameter and 0.50 µm film thickness, injection temperature: 270°C, oven temperature program: 35°C for 10 min to 280°C at 12 min, pressure : 64Psi, carrier gas Helium, injection sample : 1 ul neat oil. The GC-MS model Shimadzu QP2010, column: DB-5MS, dimethyl poly siloxane, 30 m length, 0.25mm diameter and 0.25 µm film thickness, injection temperature:250°C, oven temperature programm : 40°C for 1 min to 275°C at 11 min, hold time 1 to 11 min and rate 5c/m, pressure : 100Pa, total flow: 50 ml/min, column flow 1.78 ml/min, purge flow 3ml/min, linear velocity 48.1cm/sec, carrier gas Helium with 99.9% purity, ion source temperature: 200°C, detecter temperature 250°C, solvent cut time: 2.5min and the injection sample is 1µl from 10 µl of fennel oil dilute in 1ml ethyl

alcohol.

3. Result and Discussion

The yield % of fennel oil was calculated as follow:

Yield % = 15/ 250 × 100 = 6%

The result (GC) chromatogram in figure: 1shown that the oil is a complex mixture of 28 compounds, by using retention indices, the relation used to calculate the Kovats retention index (I) is:

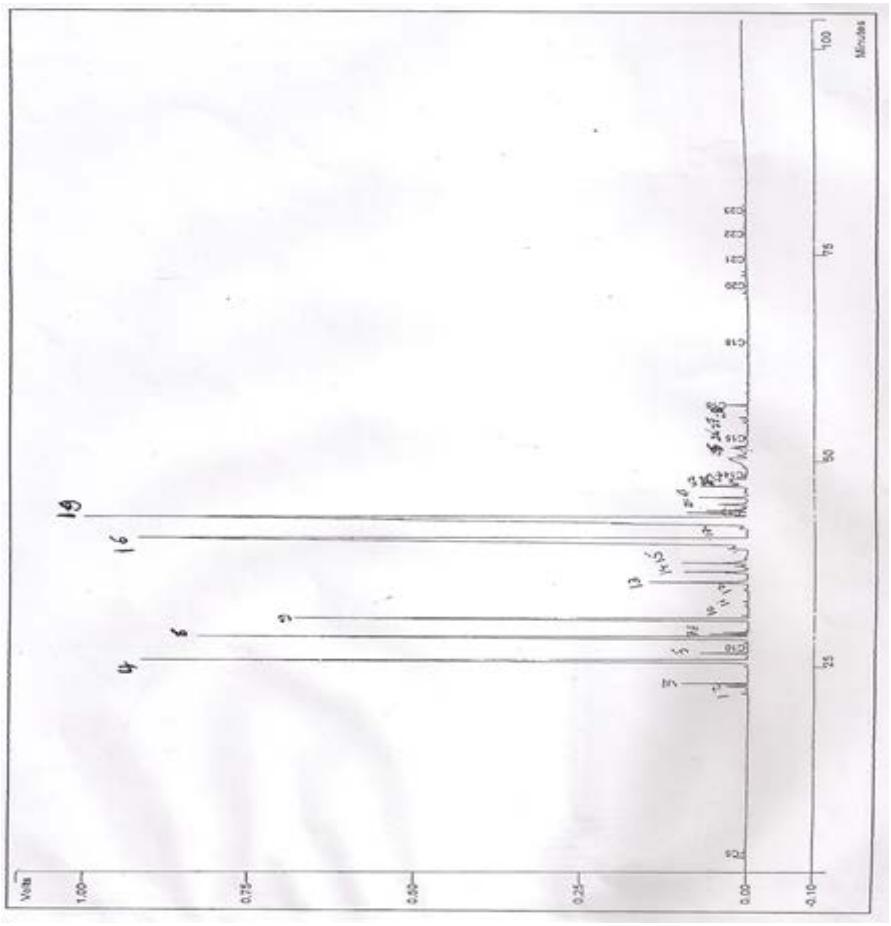

Figure 1: (GC) chromatogram of the volatile oil of foeniculum vulgare Mll.

$$I = 100 \left(n + \frac{\log t'_{R(unk)} - \log t'_{R(ns)}}{\log t_{R(nl)} - \log t'_{R(ns)}} \right)$$

n is the number of carbon atoms of the smaller hydrocarbon than the unknown log $t'_{R(unk)}$ is the adjusted retention time of the unknown compound, which is calculated from the

retention time of the unknown ($t_{R(unk)}$) by the following relation : $t_{R(unk)} - t_M$, where t_M is the time required for the mobile phase to traverse the column and is the time it would take unretained solute to appear, in this chromatogram t_M is equal to 1.779 minutes, log $t'_{R(ns)}$ is the adjusted retention time of the hydrocarbon which is smaller than the unknown log $t'_{R(nl)}$ is the adjusted retention time of the hydrocarbon which is larger than unknown.

Table 1: is retention time (t_R) and the calculated adjusted retention time (t'_R) and retention indices (I) of the volatile oil components

Peak number	t_R	t'_R	I
1	21.767	19.988	912.70
2	22.633	20.597	923.02
3	22.968	21.189	932.54
4	25.878	24.099	976.98
5	26.683	24.904	988.10
6	28.712	26.933	1021.11
7	29.018	27.239	1026.67
8	29.552	27.773	1036.67
9	30.937	29.158	1060.00
10	31.293	29.514	1065.56
11	32.981	31.202	1092.22
12	34.367	32.688	1119.12
13	35.305	33.526	1135.29
14	36.559	34.880	1161.76
15	37.623	35.844	1177.94
16	40.600	38.821	1237.04
17	41.859	40.180	1264.81
18	42.935	41.156	1283.33
19	43.262	41.483	1290.74
20	43.787	42.008	1300.00
21	44.063	42.284	1300.00
22	44.818	43.039	1317.39
23	45.608	43.829	1334.78
24	46.983	45.204	1363.04
25	50.391	48.612	1446.15
26	51.677	48.898	1451.28
27	52.792	51.013	1500.00
28	55.366	53.587	1563.64

Table 2: is retention time (t_R) and adjusted retention time (t_R) of standard hydrocarbons

Peak number	t_R	t'_R
C9	21.071	19.292
C10	27.564	25.785
C11	33.456	31.677
C12	38.809	37.030
C13	43.765	41.986
C14	48.426	46.647
C15	52.830	51.051
C16	56.893	55.114

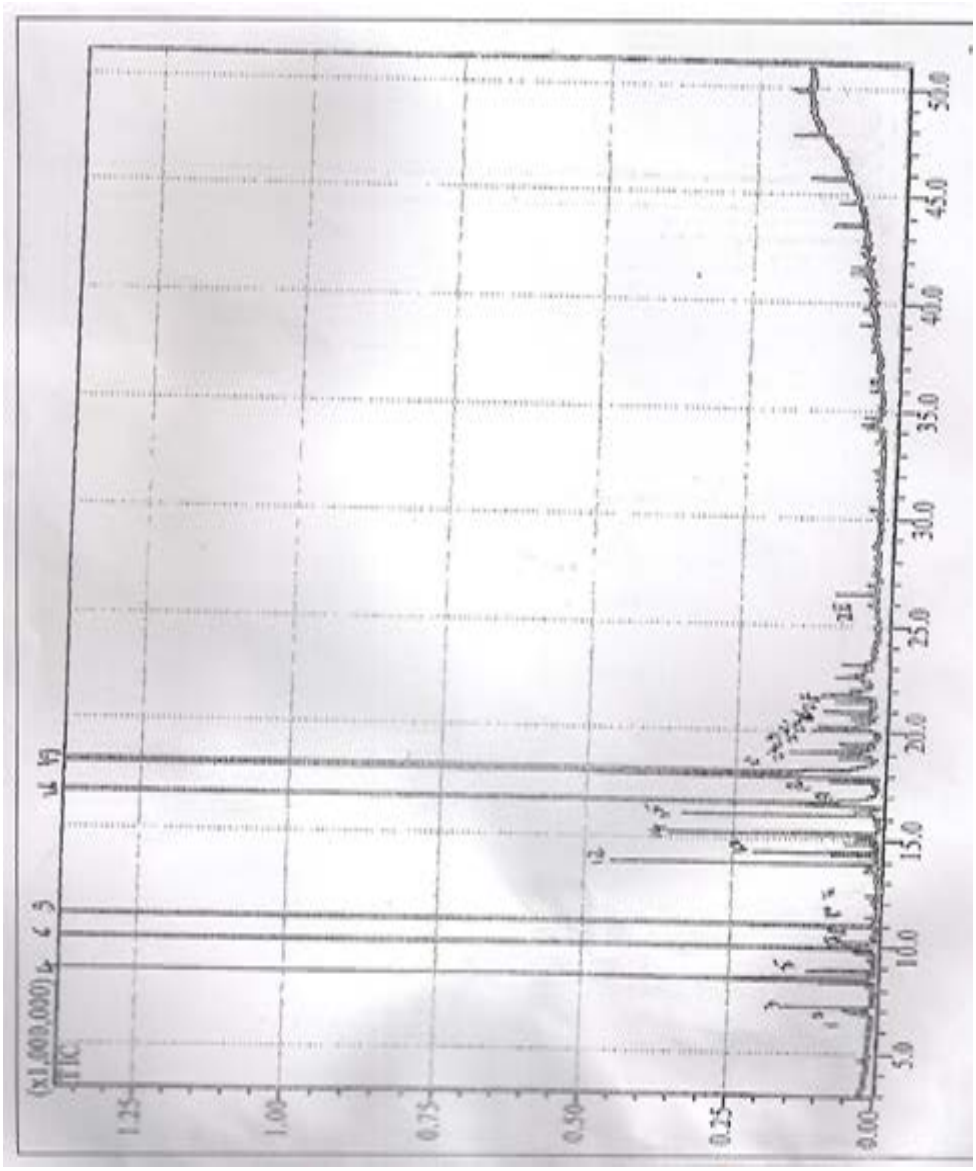

Figure 2: (GC-MS) total ion chromatogram of the volatile oil of foeniculum vulgare Mll.

From (GC-MS) 28 compounds were identified by compared the MS fragmentation pattern of these unknown compounds with those of standards compounds from the library of the machine

Table 3: Is retention time (t_R) and % area under peak of the compounds which identified by (GC-MS).

Peak number	t_R	% area	Name
1	7.050	0.13	Cyclohexane,(1-methylethyldiene)
2	7.158	0.44	Pineneα-
3	8.350	0.26	Phellandreneβ-
4	8.492	9.63	-Pineneβ
5	8.892	0.34	-Myrceneβ
6	10.100	12.13	d-Limonene
7	10.433	0.17	O-Xylene,3-ethyl
8	10.208	0.15	Cineole
9	11.017	8.03	--Terpineneγ
10	13.875	1.28	Cyclohexane,3-butyl
11	14.300	0.24	2,6-Dimethyl-3,5,7-octatriene
12	14.417	0.50	Limonene epoxide
13	15.000	0.14	1-Phenylpropane1,3-diol
14	15.300	0.95	Acetaldehyde,(3,3-dimethylcyclohexaylidene)
15	16.167	0.92	1-(Ethyl-2,3-dimethyl-cyclopent-2-enyl)ethanone
16	16.708	31.10	Anethole
17	17.742	0.19	Phellandral
18	18.000	8.71	2-Caren-10-al
19	18.525	21.25	Propanal,2-methyl-3-phenyl
20	19.275	0.51	Cyclopentane propanol,2-methylene
21	19.475	0.16	Benzene,1-(1-hydroxyethyl)-4-isobutyl
22	20.125	0.38	Nerolidol
23	20.275	0.48	Bicyclo[2.2.1]heptan-2-ol,7,7-dimethyl,acetate
24	20.675	0.32	Bicyclo[3,1,0]hex-2-ene,4-methylene-1-(1-methyethyl)
25	20.967	0.34	Butanoic acid,4-formylphenylester
26	21.467	0.76	4-Hydromethylene-2,6-dimethyl-oct-6-en-3-one
27	22.592	0.23	3-Methylene-2-fenchone
28	26.458	0.26	Carotol
		100%	

For further confirm the result of analysis of oil the Kovats retention indices (I) obtained from the (GC)

is combine with the result of (GC-MS), the total number of identified compounds is twenty eight eleven of them are hydrocarbons while seventeen are oxygenated compounds, the main constituent of volatile oil found to be Anethole 31.1%, Phenyl-2-methyl-3-propanol 21.25%, d-Limonene 12.13% ,β-Pinene 9.63% , γ-Terpinene8.03% and 2- Carenal 8.71% and their retention indices are 1237.04, 129-.74, 1021.11, 976.98, 1060 and 1283.33 respectively.

Table 4: Is compounds identified in the volatile oil of Foeniculum vulgare Mill by GC and GC-MS

Peak number	Name	I	% area
1	Cyclohexane,(1-methylethyldiene)	912.70	0.13
2	Pineneα-	923.02	0.44
3	Phellandreneβ-	932.54	0.26
4	β-Pinene	976.98	9.63
5	-Myrceneβ	988.10	0.34
6	d-Limonene	1021.11	12.13
7	O-Xylene,3-ethyl	1026.67	0.17
8	Cineole	1036.67	0.15
9	-Terpineneγ	1060.00	8.03
10	Cyclohexane,3-butyl	1065.56	1.28
11	2,6-Dimethyl-3,5,7-octatriene	1092.22	0.24
12	Limonene epoxide	1119.12	0.50
13	1-Phenylpropane1,3-diol	1135.29	0.14
14	Acetaldehyde,(3,3-dimethylcyclohexaylidene)	1161.76	0.95
15	1-(Ethyl-2,3-dimethyl-cyclopent-2-enyl)ethanone	1177.94	0.92
16	Anethole	1237.04	31.10
17	Phellandral	1264.81	0.19
18	2-Caren-10-al	1283.33	8.71
19	Propanal,2-methyl-3-phenyl	1290.74	21.25
20	Cyclopentane propanol,2-methylene	1300.00	0.51
21	Benzene,1-(1-hydroxyethyl)-4-isobutyl	1300.00	0.16
22	Nerolidol	1317.39	0.38
23	Bicyclo[2.2.1]heptan-2-ol,7,7-dimethyl,acetate	1334.78	0.48
24	Bicyclo[3,1,0]hex-2-ene,4-methylene-1-(1-methyethyl)	1363.04	0.32
25	Butanoic acid,4-formylphenylester	1446.15	0.34
26	4-Hydromethylene-2,6-dimethyl-oct-6-en-3-one	1451.28	0.76
27	3-Methylene-2-fenchone	1500.00	0.23
28	Carotol	1563.64	0.26
			100%

4. Conclusion

Fennel oil use for medicinal purposes and in the cosmetic and perfume industry stems from the presence of Limonene and Anethole in their constituent**s.**

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