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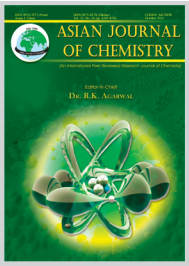

Effect of Harvest Years on Chemical Composition of Essential Oil of Bitter Fennel (*Foeniculum vulgare* subsp. *piperitum*) Leaves

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The essential oils of leave of *Foeniculum vulgare* subsp. *piperitum* (Apiaceae) growing wild in south Anatolia, Turkey were extracted by hydrodistillation and analyzed by GC and GC-MS. The percentage yields of the essential oils from leaves of bitter fennel harvested in different years (from 2003 to 2008) were 1.3, 3.09, 2.1, 1.95, 1.7 and 1.9 %, respectively. The major constituents varied depending on harvest years. Methyl chavicol, fenchone, limonene and fenchyl acetate (exo) were established as the major components of bitter fennel leaves collected in different years. The main constituents of leave oil were methyl chavicol (24.67-69.93 %), fenchone (7.82-14.50 %), limonene (2.39-9.85 %) and fenchyl acetate (exo) (0.17-14.21 %) among the years.

Key Words: Bitter fennel, Leave, *F. vulgare* subsp. *piperitum*, Harvest years, Essential oil, Composition, Methyl chavicol.

INTRODUCTION

Fresh or dried herb and fruits of bitter Fennel (*Foeniculum vulgare* subsp. *piperitum*) (called "malotra or melotra" in Turkish are used as a flovouring agent for some food such as salad, cacik and yoghurt soup[1,2]. It is a perennial or annual herbaceous and a typical aromatic plant that grows in several regions all over the world[3]. Fennel oil is used in cooking and for correcting less pleasant odours and flavours in oral and medicinal preparations[4,5]. Many of spices and herbs were valued for their preservative and medical powers besides their flavor and odor qualities[6-9]. Fennel has been prescribed as a muscle relaxant, a weak diuretic, carminative and a mild stimulant[10].

There are usually considerably variations in the major components of *Foeniculum vulgare* Mill. subsp. *piperitum* (Ucria) count fruits in north of France were reported to be limonene (52.4 and 56.9 %), piperitenone oxide (21.5 and 14.2 %) and γ-terpinene (12.1 and 5.1 %)[11]. In previous investigation on the essential oil of *F. vulgare* subps. *piperitum* fruits, methyl chavicol (47.09 %), limonene (29.07 %), fenchone (13.43 %) and fenchyl acetate (exo) (1.95 %) were found to be the major components[2].

The objective of this study is to determine chemical composition of the essential oils from wild fennel (*F. vulgare* subsp. *piperitum*) collected in six different collection year period.

EXPERIMENTAL

Bitter fennel leaves were collected from Mersin (Büyükeceli-Gülnar), Turkey from August 2003 to 2008 years. A voucher specimen is kept in the herbarium of the Department of Food Engineering, Faculty of Agriculture, University of Selçuk and identified by Dr. Bagci.

Idendification of components

Recovery of the essential oils: Dried parts of the plants (200 g) were ground and submitted to hydrodistillation for 4 h using a Clevenger-type apparatus and the oils obtained were dried over anhydrous sodium sulfate. The oil yields on a weight basis were established as 1.3, 3.09, 2.1, 1.95, 1.7 and 1.9 %, respectively.

Gas chromatographic analysis: The essential oil were analyzed on a Agilent gas chromatograph Model 6890, equipped whith a DB5 MS column (30 m × 0.25 mm, 0.25 μm), programming from 50 ºC (5 min) to 300 ºC at 5 ºC/mn, 5 min hold. Hydrogen as carrier gas (1.0 mL/min); injection in split mode (1: 60); injector and detector temperature, 280 and 300 ºC, respectively. The essential oil is diluted in hexane : 1/30.

GC-MS analysis: The essential oil were analyzed on a Agilent gas chromatograph Model 7890, coupled to a Agilent MS model 5975, equipped whith a DB5 MS column (30 m × 0.25 mm, 0.25 μm), programming from 50 ºC (5 min) to 300 ºC

at 5 ºC/mn, 5 min hold. Helium as carrier gas (1.0 mL/min); injection in split mode (1:100); injector and detector temperature, 250 and 280 ºC, respectively. The MS working in electron impact mode at 70 eV; electron multiplier, 1200 V; ion source temperature, 230 ºC; mass spectra data were acquired in the scan mode in m/z range 33-450. The essential oil is diluted in hexane :1/30. The components were identified by comparing linear Kovats indices, their retention times and mass spectra with those obtained from the authentic samples and/or the MS library. The library search was carried out using a Wiley GC/MS library of essential oil constituents.

The percentage composition of the essential oil was computed from GC peak areas without correction factors. Qualitative analysis was based on a comparison of retention times and mass spectra with corresponding data in the literature[12].

RESULTS AND DISCUSSION

The names and percentages of components identified by GC-MS in bitter fennel leave oils are given in Table-1. The samples depend on years contain a high percentage of essential oil, which is devoid of methyl chavicol, limonene and fenchone as the main constituents. The essential oils exhibited light yellow colour and typical fennel odor. The yields of the essential oil of dried ripe fruits from 2003 to 2006 years were 1.3, 3.09, 2.1, 1.95, 1.7 and 1.9 %, respectively. In this study, a total of 22, 17, 22, 22, 21, 21 and 20 compounds accounted for about 85.54, 95.88, 99.25, 95.41, 88.19 and 99.13 % of the essential oils of bitter fennel, respectively.

Methyl chavicol, fenchone, limonene and fenchyl acetate (exo) were established as the major components of bitter fennel fruits collected in different years. The main constituents of leave oil were methyl chavicol (24.67-69.93 %), fenchone (7.82-14.50 %), limonene (2.39-9.85 %) and fenchyl acetate (exo) (0.17-14.21 %) among the years.

Methyl chavicol, fenchone and limonene were identified as the highest-level main constituents for bitter fennel leave oil. But, all the oils consisted of monoterpenic hydrocarbons, oxygenated monoterpenes and sesquiterpenes. α-Phellandrene content of 2008 year was found high compared with other years.

The oil obtained from air dried fruits of bitter fennel contained methyl chavicol (47.09 %), limonene (29.07 %), fenchone /13.43 %), fenchyl acetate (exo) (1.95 %), *cis*-β-

TABLE-1
CHEMICAL COMPOSITION OF ESSENTIAL OILS OF BITTER FENNEL (*Foeniculum vulgare* subspp. *piperitum*)FRUIT COLLECTED AT THE DIFFERENT HARVEST YEARS (%)

RT	Constituents	Harvest years					
		2003	2004	2005	2006	2007	2008
10.26	α-Thujene	0.03	–*	–	0.03	–	0.10
10.52	α-Pinene	0.34	0.90	0.99	0.20	0.17	0.60
11.13	Camphene	0.09	0.12	0.07	0.11	0.08	0.19
11.92	Sabinene	0.04	–	0.40	–	0.04	0.10
12.11	β-Pinene	0.04	–	0.09	–	–	0.06
12.49	Myrcene	0.39	0.49	0.25	0.46	0.36	1.09
13.09	α-Phellandrene	0.33	0.55	0.15	0.78	0.07	13.99
13.70	*p*-Cymene	2.56	0.88	0.61	1.57	3.61	1.95
13.85	Limonene	7.23	8.43	9.85	5.37	2.39	5.35
13.91	β-Phellandrene	0.30	1.75	0.25	0.23	0.25	1.48
13.98	Eucalyptol	–	–	1.53	–	–	–
14.06	(Z)-β-Ocimene	0.08	0.49	0.17	0.55	0.09	1.23
14.42	(E)-β-Ocimene	–	–	–	–	–	0.07
14.82	γ-Terpinene	–	–	1.07	–	–	0.06
15.23	*cis*-Sabinene hydrate	–	–	0.04	–	–	–
15.71	Terpinolene	–	–	0.04	0.16	–	0.26
15.87	Fenchone	7.82	12.88	14.50	10.29	12.82	10.49
16.14	Linalool	0.07	0.70	–	–	0.09	–
16.35	Isovalerate d'isoamyl	–	–	–	0.09	–	–
16.95	Menth-2-en-1-ol-*cis-para*	0.09	–	–	0.10	–	–
17.69	Camphre	0.33	0.31	0.30	0.22	0.79	0.19
18.67	Terpinene-4-ol	–	–	0.10	–	–	–
18.82	Cymene-9-ol-*para*	–	–	–	0.26	–	–
18.86	Cryptone	0.3	0.13	–	–	0.46	
19.24	Estragol	48.340	65.39	24.67	69.93	59.41	59.57
19.75	Fenchyl acetate (endo)	1.79	0.86		0.57	0.23	0.18
20.16	Fenchyl acetate (exo)	14.21	1.57	0.17	4.09	2.45	1.99
20.6	*cis*-Ascaridole	–	–	–	–	3.63	–
20.72	Anethole Z	–	–	0.07	0.15	–	–
20.79	Anisaldehyde	–	–	0.41	–	0.56	–
21.07	*cis*-Ascaridol	0.13	–	–	–	0.39	–
21.62	Bornyl acetate	0.63	0.34	–	0.17	0.18	0.19
21.74	Anethole E	–	–	43.51	–	0.15	–
21.95	Carvacrol	0.39	0.08	–	0.11	–	–
Total		85.54	95.88	99.25	95.41	88.19	99.13

*Nonidentified [a]Compound listed in the order of elution from a HP-5MS column. [b]Each compound is mean of two values. [c]non identified.

ocimene (1.41 %), α-pinene (1.22 %) and myrcene (1.08 %) as the main constituents[2]. Özcan and Chalchat[13] also determined 21.69- 40.49 % methyl chavicol, 12.98-16.90 % fenchone, 17.66-22.24 % limonene in fruits of bitter fennel collected in 2001 and 2002 harvest years.

It was reported that the chemical composition of bitter fennel oils are variable according to harvest years. The environmental conditions and nutritional status of the plant caused to this fact. Therefore, variation in the essential oil contents and compositions is due to variation of ecological conditions (temperature, rainfall, humidity, soil, *etc*.) in different years and plants ages. Therefore, variation in the essential oil contents and compositions is due to variation of ecological conditions (temperature, rainfall, humidity, soil *etc*.) in different years and plants ages. When the results were compared with the literature, the oils showed partially differences and similarities[2,14,15]. Our results showed that the oil of Turkish bitter fennel belonged to methyl chavicol (estragole) rich type.

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