

Phytochemical Constituents of Leaves Essential oils of *Achillea fragrantissima* (Asteraceae) from Iraq

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Abstract – Essential oils of *Achillea fragrantissima* extract were prepared and analyzed by gas chromatography–mass spectrometry (GC-MS). A total of 57 phytochemical constituents of chemical compounds were identified in leaves of *A. fragrantissima*. The major constituents of the essential oil were camphor (34.50%), 1, 8-cineole (14.60%), artemisia ketone (10.25%), and 3-thujanone (7.82%). In addition, 43 components were present at <1%. From the 57 identified compounds, four of them was sesquiterpenes (7.01%), whereas 35 compounds were monoterpenes (61.40%).

Index Terms—*Achillea fragrantissima*, camphor, essential oils, gas chromatography–mass spectrometry.

I. INTRODUCTION

Achillea fragrantissima (Forssk.) Sch. Bip. is a wild herbaceous shrub medicinal plant belonging to the *Asteraceae* family. *Achillea* contains around 130–140 perennial species worldwide, it is a white-woolly plant, with erect stems and is widespread in Europe and temperate areas of Asia, North America, and in North Africa, it is easily found growing in fields and on roadsides (Nemeth, 2010).

A. fragrantissima known as yarrow, in Arabic called Qaysoom. Most parts (leaves, flowers, and seeds) of *A. fragrantissima* contain high percentage of volatile oils, flavonoids, tannins, sterols and triterpenes monoterpene ketones, and sesquiterpene lactones (Batanouny, et al., 1999; Bakr, et al., 2014) fatty acids: Lauric, myristic, palmitic, stearic, linoleic, linolenic, and oleic (Al-Mustafa and Al-Thunibat, 2008). So that different parts of *A. fragrantissima* including fruits, leaves and branches have been used as a folk medicine for the treatment of various diseases. It is used as anti-inflammatory, antioxidant, antiproliferative capacities, antimicrobial, antifungal, antiviral,

and anticancer activity (Shalaby and Richler, 1964; Ageel, et al., 1989; Barel, et al., 1991; Al-Mustafa and Al-Thunibat, 2008; Soltan and Zaki, 2009; Elmann, et al., 2011; Hazem, et al., 2012; Vitalini, et al., 2013; Alenad, et al., 2013; Hammad, et al., 2014; and Choucry, 2017). Comparison between the essential oils of *A. fragrantissima* (Forssk.) Sch. Bip. and *A. santolina* L. (*Asteraceae*) studying their antimicrobial activity was reported by El-Shazly, et al., 2004.

The main constituents of *A. fragrantissima* are essential oil santolina alcohol, artemisia ketone, cis-thujone, and trans-thujone (El-Shazly, et al., 2004), whereas Hazem, et al. (2012) shown that 48 components were identified in the oils, and main compounds of oils were 4-terpineol (15.65%), linalool (11%), carvone (9.42%), β -phellandrene (6.2%), γ -terpinene (5.6%), β -pinene (4.55%), verbenone (4.42%), cedrol (3.0%), and *p*-cymene (2.95%). Choucry (2017) reported that 28 compounds were identified in the *A. fragrantissima*, caryophyllene oxide (23.50%), terpinen-4-ol (11.15%), *p*-cymen-3-ol, viridiflorol, and guaiacol (9.84%). In all these studies, chemical compositions were identified and the amount of the yields of essential oils was varied. The aim of this study, *A. fragrantissima* of leaves collected from Choman region, Erbil city, Iraq to extract essential oil and its compositions were analyzed using Gas chromatography and Mass spectrometry (GC-MS).

II. MATERIALS AND METHODS

A. Plant Material

The leaves of *A. fragrantissima* were collected from plants in flowering growth stage from Choman region, 160 km northeast Erbil city, Iraq, in September 2016 at Latitude 36.636310 N, Longitude 44.886767 E, and Altitude 3607 m. The plant was identified at the Basrah University. A voucher specimen was deposited at the herbarium of the Basrah Science, Faculty of Science, Basrah University, Iraq.

The meteorology of the study location for September 2016 was as follows: 26–28°C, 56%, 28 mm, and 2.3 m/s for temperature, relative humidity, rains, and wind speed, respectively, whereas the soil texture was sandy loam.

The samples were air-dried in the shade at room



temperature until dried (2 weeks). To obtain the leaves essential oil, 150 g of dried leaves were crushed, and the method of hydrodistillation for 5 h using the Clevenger-type apparatus was used. The obtained essential oils were dried over anhydrous sodium sulfate and stored in the refrigerator (-18°C) until used (Massada, 1976).

B. GC-MS Analysis

GC-MS analysis was carried out in the University of Basrah, College of Agriculture, Iraq using Shimadzu GC-QP 2010 ultra gas chromatograph. The GC oven temperature was programmed from 40°C to 280°C at a rate of $15^{\circ}\text{C}/\text{min}$. Helium was used as carrier gas; inlet pressure was 96.1 kPa; and linear velocity was 36.1 cm/s. Column flow was 1.00 mL/min, injector temperature: 280°C ; injection mode: split. MS scan conditions was carried using a ion source temperature of 200°C , interface temperature, 280°C with detector gain 0.70 kV+0.10 kV. Scan speed was 1666, start 50 m/z, and then raised to end 800 m/z (Vandendoo and Kratz, 1963). The components of the *A. fragrantissima* were identified by comparing the spectra with those of known compounds stored in the NIST library (2005). The identification of the phytochemical compounds was confirmed based on the molecular formula, peak area, and retention time (Fig. 1-6).

III. RESULTS AND DISCUSSION

The GC-MS chromatogram of *A. fragrantissima* leaves extract (Fig. 7 and Table-1) showed 57 peaks indicate the

presence of 57 compounds (phytochemical constituents).

Most of the chemical components from *A. fragrantissima* leaves are essential oil components including camphor (34.50%), 1, 8-cineole (14.60%), artemisia (10.25%), and 3-thujanone (7.82%). In addition, 43 components were present at $<1\%$ (Table 1 and Fig. 7), from all this component, the number and percentage of sesquiterpenes component were four compounds; methyl 3,4-tetradecadienoate, beta-cubebene, beta-selinene, and benzofuran, 7-cyclohexyl-2,3-dihydro-2-methyl- which represented 7.01% from all compounds in essential oil, and monoterpenes were 35 compounds (61.40%). Our results agree with other literatures, monoterpenes are the principal components of *Achillea* essential oils (Zeedan, et al., 2014; Mottaghi, et al., 2016). Furthermore, 1, 8-cineole (20.1%), camphor (15.6%), and viridiflorol (11.8%) the three most abundant monoterpene components (Toker, et al., 2003).

Most researchers reported that santolina alcohol, artemisia ketone, cis-thujone, and trans-thujone were the major constituents in *A. fragrantissima* (Shalaby and Richter, 1964; El-Deeb, 1985; Hifnawy, et al., 2001; El-Shazly, et al., 2004; Abaas, et al., 2013; and Alshaili and Al-fawwaz, 2014). In addition, borneol and 1,8-cineole (Gohari, et al., 2011; Mazandarani, et al., 2013; Zeedan, et al., 2014). However, our results vary in the chemical composition of essential oils of *A. fragrantissima*, as well as the number and ratio of chemical components. These differences might be due to the diversity of the plant sources, different essential oil hydrodistillation procedures or growth conditions. In Egypt, the essential oil

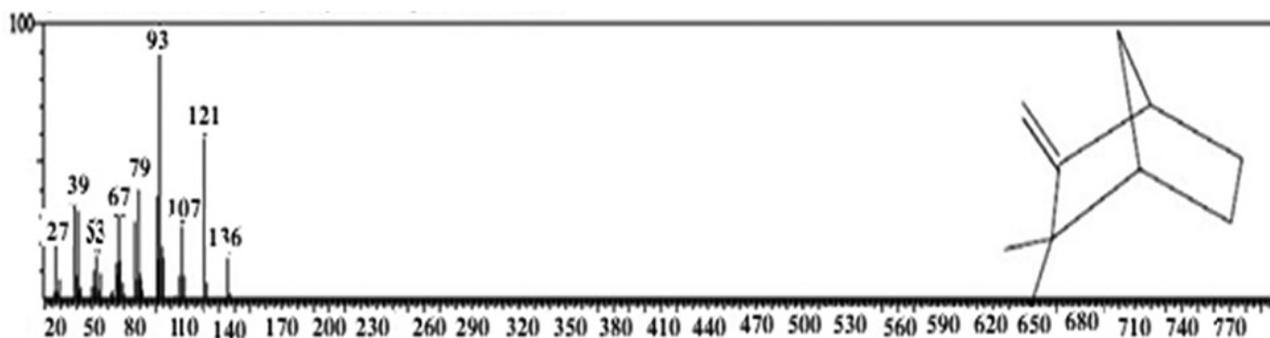


Fig. 1. A typical gas chromatogram of the camphene.

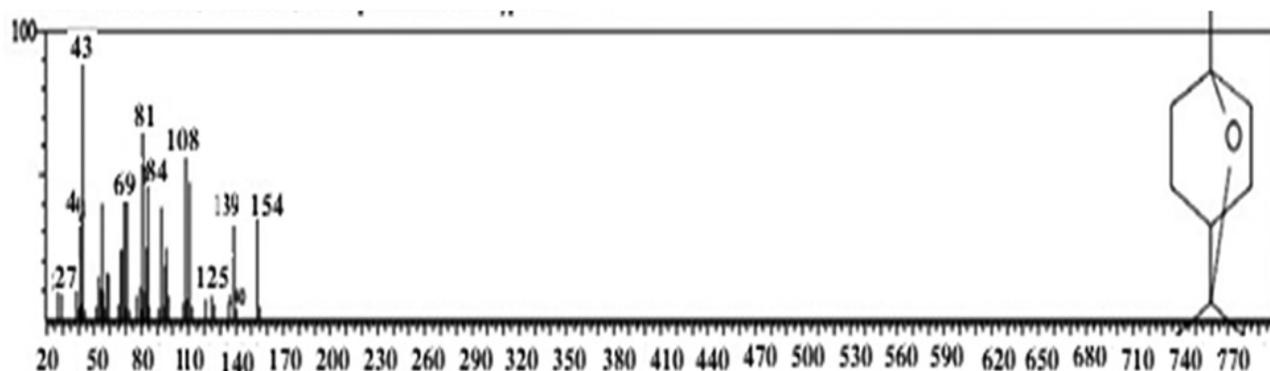


Fig. 2. A typical gas chromatogram of the 1, 8-cineole.

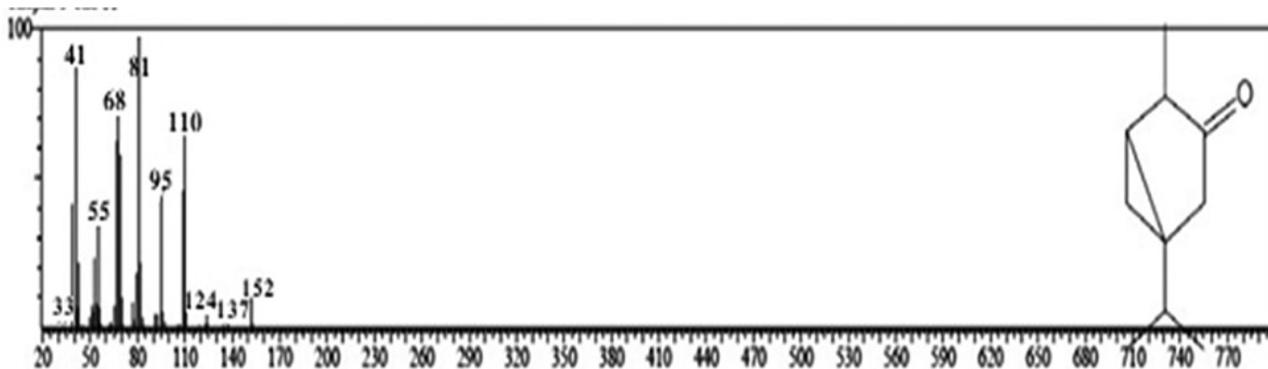


Fig. 3. A typical gas chromatogram of the 3-thujanone.

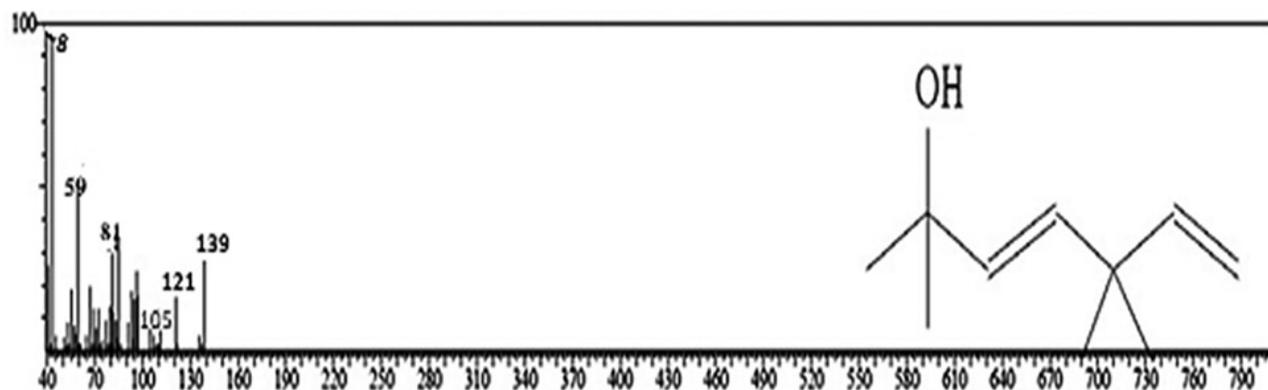


Fig. 4. A typical gas chromatogram of the yomogi alcohol.

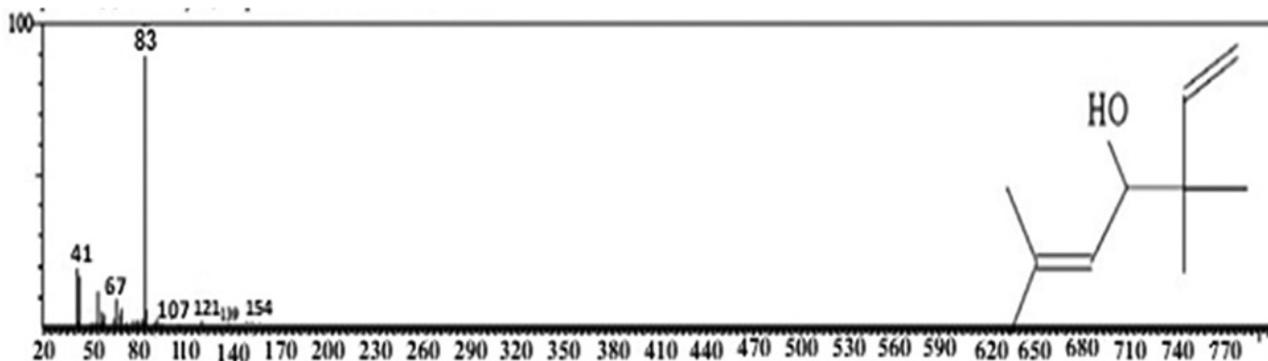


Fig. 5. A typical gas chromatogram of the artemisia alcohol.

obtained by hydrodistillation analyzed using GC-MS was found that the major components of *A. fragrantissima* were thujone (33.97%), eucalyptol 8.17, artemisia alcohol (3.49%), santolina triene (1.97%), and terpineol (0.05%) (Zeedan, et al., 2014). Alshaili (2018) revealed that the chemical composition of essential oil of *A. fragrantissima* contains trans-sabinyl acetate (0.75–10.20%), α -terpineol (3.53–9.39%), trans-menth-2-en-1-ol (6.5–13.34%), and β -thujone (11.34–22.11) using GC-MS, whereas Choucry (2017) reported that the major components were caryophyllene oxide (23.50%) and 1-terpinen-4-ol (11.15). As well as in Jordan

15 compounds were identified of *A. fragrantissima* essential oil, artemisia ketone, β -sesquiphellandrene, and carvacrol are the major observed compounds with ratios 19.87, 14.57, and 13.44%, respectively, (Alshaili, and Al-fawwaz, 2014).

Although β -phellandrene, linalool, verbenone, and cedrol were found in other studies, they could not be detected in the plants from the present study. These variations may be due to the influence of geographical differences, physiological differences, and genetic factors (Adams, 2007). Environmental factors such as climate, soil, harvest season, method of drying, storage conditions, and even the part of the plant tissue

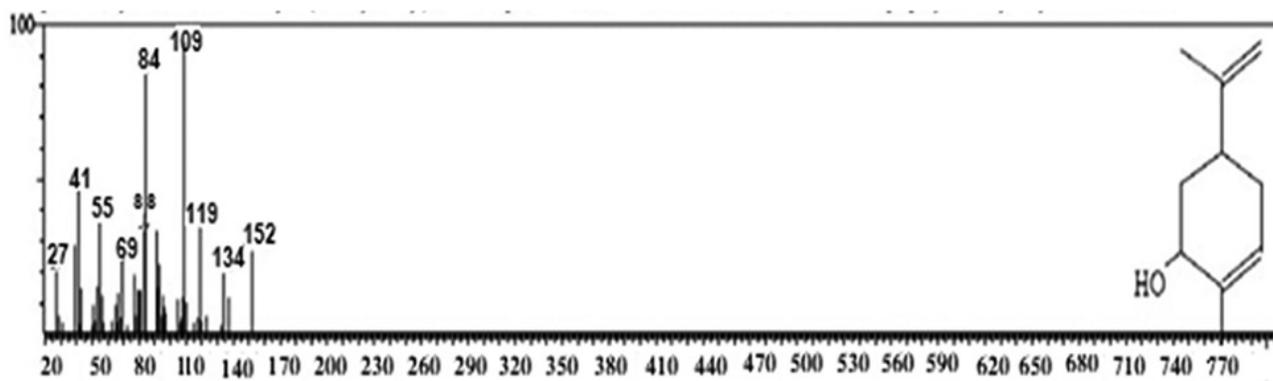


Fig. 6. A typical gas chromatogram of the trans-carveol.

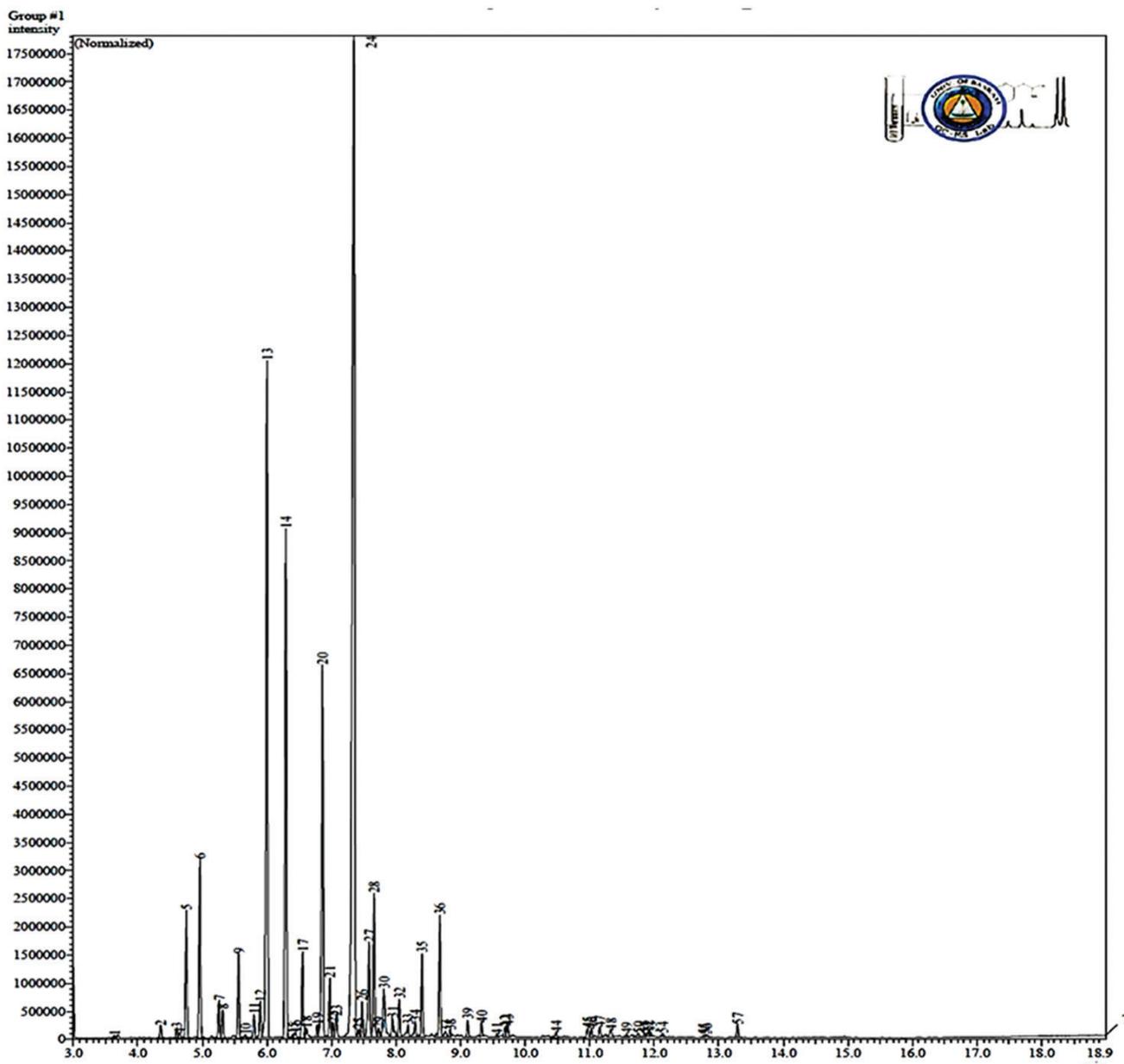


Fig. 7. Chromatogram of essential oil of *Achillea fragrantissima* leaves.

TABLE I
COMPOUNDS IDENTIFIED IN THE ESSENTIAL OIL OF *A. FRAGRANTISSIMA* LEAVES USING GC-MS

Peak number	Formula	Retention time	Retention indices	Content (%)	Components
1	C15H42P6Pd2	3.633	87409	0.06	Dipalladium (0), tris [mu bis(dimethylphosphino) methane]
2	C10H16	4.353	375108	0.26	Santolina
3	C10H16	4.598	295483	0.20	Tricyclo[2.2.1.0 (2,6)]heptane, 1,7,7trimethyl-(Tricyclene)
4	C41H39NO9	4.642	194053	0.13	1-O-p-Nitrobenzoyl-2,3,4,6-tetra-O-benzyl-betad-galactose
5	C10H16	4.749	36731873	2.54	Alpha-Pinene
6	C10H16	4.959	5219456	3.61	Camphene
7	C10H16	5.255	1085767	0.75	Sabinene or Thujene
8	C10H16	5.313	835579	0.58	Beta-pinene
9	C10H18O	5.559	2320358	1.60	Yomogi alcohol
10	C10H16	5.664	96064	0.07	Alpha-Phellandrene
11	C10H16	5.798	674863	0.47	(+)-4-Carene
12	C10H14	5.896	1154110	0.80	m-Cymene
13	C10H18O	5.997	21131715	14.60	1,8-Cineole
14	C10H16O	6.292	14835852	10.25	Artemisia ketone
15	C38H36N2O4	6.402	123812	0.09	Estradiol 17-benzoate-3-pphenylazobenzoate
16	C10H18O2	6.440	179688	0.12	alpha-Methyl-alpha- [4-methyl-3-pentenyl] oxiranemethanol
17	C10H18O	6.550	2429384	1.68	Artemisia alcohol
18	C10H16	6.609	378420	0.26	Bicyclo[4.1.0]hept-3-ene, 3,7,7trimethyl-
19	C9H14	6.779	442115	0.31	Bicyclo (3.3.1)non-2-ene
20	C10H16O	6.859	11322487	7.82	3-Thujanone
21	C10H16O	6.973	1669169	1.15	Thujone
22	C10H14O	7.020	438939	0.30	Chrysanthene
23	C10H18O	7.074	714612	0.49	p-Menth-1(7)-en-9-ol
24	C10H16O	7.346	49947783	34.50	Camphor
25	C9H14O	7.419	277954	0.19	Sabina ketone
26	C10H14O	7.472	1066587	0.74	Pinocarvone
27	C10H18O	7.582	3249579	2.24	Borneol
28	C10H18O	7.658	4070212	2.81	4-Terpeneol
29	C10H14O	7.730	323081	0.22	3,9-Epoxy-p-mentha-1,8(10)-diene
30	C10H18O	7.810	2218854	1.53	alpha-Terpeneol
31	C15H26O2	7.944	780403	0.54	Methyl 3,4-tetradecadienoate
32	C10H16O	8.050	1038638	0.71	trans-carveol
33	C11H22	8.184	560224	0.39	2-Heptene, 5-ethyl-2,4-dimethyl-
34	C10H14O	8.294	561007	0.39	Carvone
35	C10H16O	8.402	2400410	1.66	Piperitone or carvomenthenone
36	C12H20O2	8.676	3450467	2.38	Acetic acid, 1,7,7-trimethyl-bicyclo [2.2.1] hept-2-yl ester
37	C10H14O	8.765	240528	0.17	P-Cymene
38	C10H14O	8.835	208656	0.14	Carvacrol
39	C12H18O2	9.109	459884	0.32	Carvyl acetate
40	C10H12O2	9.323	386113	0.27	3-Allyl-6-methoxyphenol
41	C13H18O	9.559	74539	0.05	Damascenone
42	C10H14O	9.689	401345	0.28	2H-Inden-2-one, 1,4,5,6,7,7a-hexahydro - 7a-methyl-, (S)-
43	C11H14O2	9.727	321481	0.22	Methyl eugenol
44	C15H24	10.479	234465	0.16	beta-Cubebene
45	C19H32O	10.967	351364	0.24	1,9,12,15-octadecatetraene, 1 methoxy-
46	C10H16O	11.025	274199	0.19	beta-Pinene oxide
47	C18H30O5	11.155	393228	0.27	2-Butyloxycarbonyloxy-1,1,10trimethyl-6,9-epidioxydecalin
48	C10H16O	11.327	376046	0.26	Pinane
49	C21H30O2	11.571	134997	0.09	3-Phenylpropanoic acid, dodec-9-ynyl ester
50	C13H20O3	11.751	200159	0.14	Cyclopentaneacetic acid, 3-oxo-2-(2-pentenyl)-, methyl ester, [1 alpha, 2. Alpha (Z)]-
51	C10H18O	11.850	145938	0.10	Cyclohexanol, 2-methyl-3-(1-methylethenyl)-, (1-alpha.,2-alpha.,3-alpha.)-
52	C15H26O	11.903	155051	0.11	Eudesm-4 or beta-eudesmol) or beta-selinene
53	C15H20O	11.948	86696	0.06	Benzofuran, 7-cyclohexyl-2,3-dihydro-2methyl-
54	C16H24	12.129	118661	0.08	1,3-Di (propen-1-yl) adamantane
55	C11H16O2	12.768	80578	0.06	Jasmololone
56	C11H18N2	12.808	136055	0.09	(+)-1-Cyano-d-camphidine
57	C16H30O2	13.290	373362	0.26	Z-7-Hexadecenoic acid
			144776160	100.00	

GC-MS: Gas chromatography-mass spectrometry, *A. fragrantissima*: *Achillea fragrantissima*

evaluated are all parameters that should be considered (Skotti, et al., 2014; Bouaziz, et al., 2015). Moreover, Farhat, et al. (2001) showed major seasonal changes in the composition of the oil. Therefore, the concentration and composition of the oil in our tests may resulted from seasonal and year differences during the collection of plants essential oil content of *Achillea* species changed according to the region therewith medicinal characters of the plants are also changed (Toncer, et al., 2010).

IV. CONCLUSION

Results from this study have shown that the essential oil contains compounds with useful in pharmacological purposes. GC-MS analysis revealed that 57 different chemical components were identified in the *A. fragrantissima* leaves. The camphor compound recorded the highest amounts of 34.50%. We also showed that leaves of *A. fragrantissima* contain different amounts of monoterpenes and sesquiterpenes. All the detected compounds can inter in making different medicinal drugs.

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