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Chemical composition for hydrodistillation essential oil of mentha longifolia by gas chromatography-mass spectrometry from north regions in Kingdom of Saudi Arabia

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ABSTRACT

Essential oil are very interesting natural plant products and among other qualities they posses various properties depend on the composition in the essential oil, so it is important to know that composition, which give idea for the toxicology and safety of essential oil. Therefore, the chemical characterization of the essential oil is very important for the understanding of its properties. The literatures show and to our knowledge no previous reported on the Al-Habak (Mentha longifolia) have been study for different location in KSA by GC-MS. Al-Habak (Mentha Longifolia) were collected from different location in Kingdom of Saudi Arabia (KSA) were subjected to the hydrodistillation extraction for the essential oils, the oil submit to qualitative and quantitative analysis using Gas chromatography-Mass Spectrometer (GC-MS) techniques equipped with a fuse silica capillary column. The instrument will be optimized in term of the temperature programme for the GC oven and the effective parameter for MS. The identification will carried out by computer matching with the NIST library as well as by comparison the retention time with standards. A total of 49 compounds were identified in the essential oils extracted from Al-Habak plant represented 99.96-100% of the total oil compositions, with pulegone (11.92- 62.54%), menthone (7.84-34.13%) as the main constituents found in all samples.

Keywords: Mentha longifolia, Gas Chromatography - Mass Spectrometer, essential oils analysis, Hydrodistillation.

INTRODUCTION

Menthalongifolia (L) Hudson (wild mint, Englishhorsemint) belongs to the genus Mentha, Lamiaceaefamily and is associated with medicinal and aromaticherbs. It is widespread throughout the Mediterranean,Central and Northern Europe, Asia and Africa. It is fast growing perennial herb [1,2]. Mint is used as traditional medicine for stomach ache, anti asthmatic anti spasmodic [3-5]. It is mostly the leaves that are used, usually to make a tea that is drunk for coughs, colds, stomach cramps, asthma, flatulence, indigestion and headaches[6-7]. Mint extracts areapplications may be found in the cosmetic industry, as ingredients of fragrances, decorative cosmetic, fine fragrances and flavoring, in the food industry, as aromas and flavors, in the pharmaceutical industry, as active components of medicines and as antibacterial/antimicrobials, are generally considered safe to use as the provide good defense against oxidative damage and health benefits [8].

The most common methods used for the extraction of essential oils are hydrodistillation. Their selection will depend on the characteristics of the material from which the oil will be extracted, since they can be present in different parts of the plant, like the roots, the stem, the leaves, the fruits and/or the seeds [9].

Essential oil composition of plants varies and is due to genetic and environmental factors that influence genetic expression.The essential oil content of plant tissue also varies with developmental stage and can vary by extraction methods. Chemical composition of the essential oil of wild mint herb is very variable depending on the habitat and climate where the species grow [10].

Gas chromatography-mass spectrometry (GC-MS) is the most popular method for the determination of essential composition. Components existing in the essential oil can be identified by comparison of their mass spectra with the MSdata reported in a library for a single compound, with different common names, or systematic name [11].

The chemical composition of essential oil derived from *M. longifolia*was investigated by Mkaddem et al.,[12].Many researchers describe the chemicalcomposition of the essential oil using gas chromatography-mass spectrometry techniques [13-15].

The aim of present study to extract the essential oil of Menthalongifolia from different locations in KSA and analysis the chemical compositions of the oil by GC-MS and care was taken to optimize the chromatography conditions in order to obtain the most accurate results.To our knowledge, no previous reports on Menthalongifoliaessential oil in this area have been made.

MATERIALS AND METHODS

2.1 Experiments

2.1.1 Reagents and Plant Material

The plant materials of Al Habak (Menthalongifolia) were collected in march 2014 from northparts in kingdom of Saudi Arabiasuch as Al-Ahsa, Al-Qassim, Al-Madinah-Ibear Ali, Al-Madinah-IbearAlmashy, as shown in the map below. Where people frequently use this plant in traditional medicine and as flavor in tea, same fresh drinks and food. The collection plant was taxonomically kindly identified at the department of Biology, faculty of Sciences, Taif university. All regents were GC grade.

Figure 1: The regions that have been collecting sample of Al-Habak

2.1.2 Essential Oil Extraction

The samples preparation and Essential oil extraction from the collected fresh plant samples, the leaves were released, typically 300 grams of the fresh leaves were cut into smaller pieces and hydro distilled for 3 h using Clevenger type apparatus producing a colorlessessential oils at a yieldof 0.5-0.9 %. Oils were dried over 2 grams of anhydrous sodium sulfate and subsample were taken for analysis of the constituents. Then a quantity of 10 µl from the dried essential oil was mixed with 1 ml of GC grade n- hexane. The new mixture was agitated for one min, and 1 µl was injected into the gas chromatography twice by using the autosampler injector. The GC-MS analysis were performed on the same day with the extraction.

2.1.3 Gas Chromatography–Mass Spectrometry System

The aim of the selection and definition of chromatographic condition is to achieve a proper separation of the components of the oil, both for the qualitative analysis, and for the proper quantification.

The analysis of the samples was performed using gas chromatograph (GC, Model CP 3800, Varian,USA) coupled with a mass spectrometer (MS, Model Saturn 2200, Varian) and auto sampler (Model CombiPal, Varian) system. The separation was done using a VF-5ms fused silica capillary column (5% phenyl-dimetheylpolysiloxane, 30 m × 25 mm i.d., film thickness 0.25 mm, Varian). For MS detector, electron impact (EI) ionization system with ionization energy of 70 eV was used. Helium gas was used as a carrier gas at a constant flow rate of 1 ml/min. Injector and mass transfer line temperature were set at 250 and 300^{o} C, respectively. The Optimization condition for oven temperature was programmed for 10 min at 40 ^{o}C, 40-200 ^{o}C at 3 ^{o}C/min and 200-220 ^{o}Cat 2 ^{o}C/min then held for 5 min at 290 ^{o}C, solvent delay time 3 min. The injection of the samples was carried out with the auto-sampler for 1 µl with a split ratio 1/20. The conditions of analysis and specification of the instrument were optimized for a better separation and resolution. Identification of components was based on matching with Wiley and NIST electronic library.

RESULTS AND DISCUSSION

A total of 49 compounds were identified in the essential oils extracted from differentMenthalongifolia plants collected in Kingdom of Saudi Arabia. The composition together with the percentage and retention time of Menthalongifolia are shown in(Table 1). The major components of KSA Menthalongifolia oil, which generally found in all eight samples were pulegone (11.92-62.54%), menthone (7.84-34.13%).

In Al-Ahsa sample, thirty-four compounds of the total oil were identified. The most abundant constituents (≥ 1%) in the essential oil of Menthalongifolia were found to be pulegone (33.21%), menthone (28.53%), menthol (11.12%), eucalyptol (5.96%), borneol (3.13%), δ-terpineol (2.20%), β-caryophyllene (1.77%), isomenthone (1.69%), δ-cadinene (1.46%), β-pinene (1.02%).

From Al-Madinah Al-Munawarah two samples were collected from two different regions, Ibear Ali and Ibear Al-Mashy. Twenty-nine compounds were identified from the oil of Ibear Ali. The most abundant constituents (≥ 1%) in the essential oil of Menthalongifolia were found to be menthone (34.13%), eucalyptol (20.07%), isomenthone (14.64%), pulegone (11.92%), menthol (6.60%), δ-terpineol (1.83%), β-pinene (1.23%), α-terpineol (1.16%).

Twenty-six compounds were identified from the oil of Ibear Al-Mashy. The most abundant constituents (≥ 1%) in the essential oil of Menthalongifolia were found to be pulegone (62.54%), eucalyptol (9.92%), menthone (7.84%), isomenthone (5.28%), limonene (1.81%), piperitone (1.69%), β-pinene (1.53%), isopulegone (1.52%), β-sabinene (1.04%).

Twenty-nine compounds were identified from the oil of Al-Qassim sample. The most abundant constituents (≥ 1%) in the essential oil of Menthalongifolia were found to be pulegone (47.74%), menthone (15.57%), isomenthone (10.62%), eucalyptol (10.57%), menthol (2.97%), β-pinene (1.30%), borneol (1.01%).

For all four samples, the chromatograms resulting of the analysis process by GC–MS as shown below

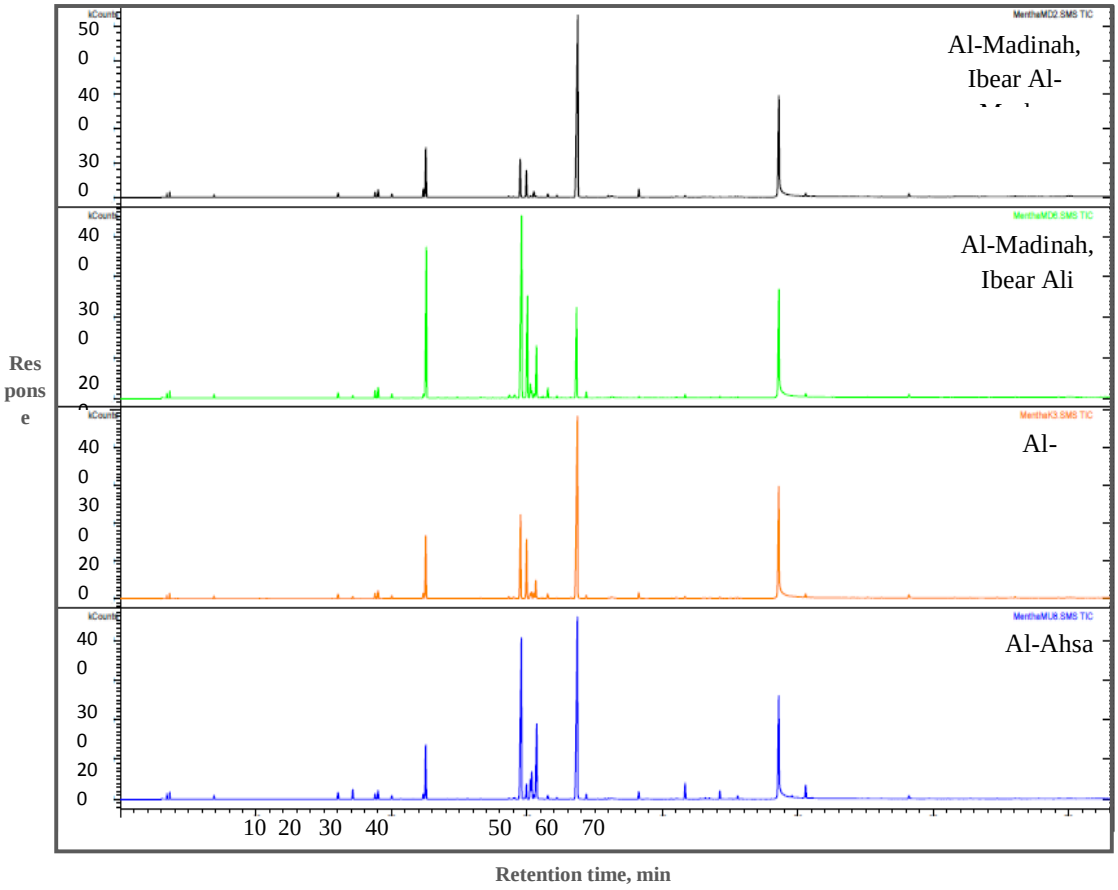

Figure 2 : Collected GC-MS chromatograms for essential oil collected from north parts locations in KSA

There are many reports in the literature showing the variation in the yield and chemical composition of the essential oil with respect to geographical regions. About the chemical composition of Menthalongifolia, the most close result to our results was performed by Hafedh et al.,[16] who reported that the most important components of essential oil from Menthalongifolia were: Menthol (32.51%), Menthone (20.71%) and Pulegone (17.76%). Also, there are some similarities in some of the results of the studies presented in this object. Mohkami et al., [17] reported that Menthe longifolia was constituted by pulegone (22.37%), Isomenthone (11.56%), β-Pinene (7.02%), Piperitenone oxide (6.75%) and 1,8-Cineole (6.60%). Iqbalet al., [18] reported that the most abundant constituents in the essential oil of Menthe longifolia were found to be piperitenone oxide (28.3%), piperitenone (24.9%), germacrene D (8.16%), borneol (5.96%), and β-caryophyllene (5.94%). Golparvar et al., [19]reported that the 37 and 33 compounds were identified in dried aerial parts Isfahan and Lorstan province, respectively. The compounds identified in dried aerial parts in Isfahan province were; 1,8-Cineole (15.58%), Piperitenone oxide (15.05%), Pulegone (9.58%), Sabinen (9.52%), 2,6-Dimethyl-2,4,6-octatriene (6.768%), α-Pinene (5.519%), β-Caryophyllene (4.636%) and β-Pinene (3.568%). The compounds identified in dried aerial parts in Lorstan province were; p-Mentha-3,8-diene (10.531%), 2,6-Dimethyl-2,4,6-octatriene (10.132%), Sabinene (6.98%), β-Caryophyllene (6.971%), Piperitone oxide (6.7%), Pulegone (6.6%), Pulespenone (6.462%), Menthofuran (6.42%), α-Terpinolene (5.84%), α-Pinene (5.46%), 1,8-Cineole (5.272%) and Isopulegone (4.4%). Saeidi et al., [20] reported that the 23 identified compounds of Menthalongifolia (L.) Hudson grown wild in Iran, the major compounds were; Piperitenone oxide (7.41 to 59.67%), Pulegone (3.61 to 49.43%), 1,8–Cineole (7.25 to 24.66%), α-Terpineol (2 to 6%) and β-pinene (1.32 to 4.19%). Sharopov et al., [21] reported that the major components of Tajikistan Menthe longifoliaoil were *cis*-piperitone epoxide (7.8-77.6%), piperitenone oxide (1.5-49.1%), carvone (0.0-21.5%), menthone (0.0-16.6%), thymol (1.5-4.2%), pulegone (0.3-5.4%), β-thujone (0.2-3.2%), (*E*)-caryophyllene (0.9-2.5%), myrcene (0.3-2.5%), carvacrol (0.0-2.7%), borneol (0.9-1.8%), and *p*-cymene (0.2-1.9%). The chemical composition of volatile oil derived from

Menthe longifolia was investigated by Mkaddem et al., [12] their research has been conducted on populations from different regions of Gabes, Tunisia. Compounds identified in significant quantities in the essential oil were pulegone (54.41%), isomenthone (12.02%), 1,8-cineole (7.41%), borneol (6.85%), and piperitone oxide (3.19%). Finally, Asekun et al., [22] reported that in the oil from the fresh plant. Pulegone (35.0%), menthone (31.1%) and 1,8-cineole (13.0%) were the most abundant.

Table1 : Chemical Composition of Al-Habak (MenthaLongifolia) Essential Oils from North Parts Locations in KSA

Oil constituent	% of oil from sample of				
	RT	Al-Ahsa	Al-Madinh, Ibear Ali	Al-Madinh, Ibear Al-Mashy	Al-Qassim
		%	%	%	%
Butane, 2-methyl	3.41	0.28	0.27	0.33	0.26
Hexane, 3-methyl	3.62	0.37	0.37	0.45	0.35
Octane	6.89	0.33	0.33	0.40	0.32
α-Thujene	15.61	-	-	-	-
α-Pinene	16.04	0.85	0.61	0.92	0.74
Camphene	17.13	1.16	0.26	0.07	0.33
β-Sabinene	18.77	0.63	0.87	1.04	0.85
β-Pinene	19.00	1.02	1.23	1.53	1.30
α-Phellandrene	20.01	0.38	0.43	0.61	0.44
3-Octanol	20.67	-	-	-	-
α-Terpinene	21.58	-	-	-	-
β-Phellandrene	22.08	-	-	-	
Limonene	22.34	0.69	0.57	1.81	0.94
Eucalyptol	22.51	5.96	20.07	9.92	10.57
γ-Terpinene	24.09	-	-	-	-
Sabinene Hydrate	24.84	0.05	0.10	0.23	0.05
α-Terpinolene	25.61	0.08	-	-	0.05
Linalool	26.57	0.05	0.09	-	0.07
P-menth-2-ene-1-ol	27.82	-	-	-	-
Cis-Sabinol	28.67	0.14	0.48	-	0.29
Verbenol	29.08	0.17	0.64	0.16	0.20
Menthone	29.58	28.53	34.13	7.84	15.57
Isomenthone	29.95	1.69	14.64	5.28	10.62
δ-Terpineol	30.24	2.20	1.83	0.50	0.77
Borneol	30.35	3.13	0.73	-	1.01
Isopulegone	30.51	0.67	0.37	1.52	0.90
Menthol	30.71	11.12	6.60	0.27	2.97
α-Terpineol	31.53	0.47	1.16	0.75	0.81
Verbenone	32.20	0.19	0.15	0.41	-
Pulegone	33.72	33.21	11.92	62.54	47.74
P-menth-4-ene-3-one	34.37	0.55	0.65	0.14	0.48
bornyl Acetate	35.76	0.08	-	-	-
Menthyl Acetate	36.02	0.11	-	-	-
Thymol	36.22	0.23	0.31	0.32	0.28
Piperitenone	38.25	0.90	0.16	1.69	0.91
Methyl Eugenol	41.03	0.10	0.14	0.25	0.20
β-Caryophyllene	41.67	1.77	0.38	0.35	0.32
α-Humulene	43.17	-	-	-	-
γ-Muurolene	44.24	-	-	-	-
Germacrene	44.24	0.92	-	-	-
β-Germacrene	44.84	-	-	-	
β-Elemene	45.32	-	-	-	-
γ-Cadinene	45.56	0.35	-	-	-
Nerolidol	47.44	-	-	-	-
Spathulenol	48.08	-	-	-	-
Caryophyllene oxide	48.27	-	-	-	-
Cubenol	49.56	-	-	-	-
δ-Cadinene	50.58	1.46	0.33	0.55	0.51
tau.-Muurolol	51.07	-	-	-	-

CONCLUSION

Essential oils are important natural products used for their flavour and fragrances in food, pharmaceutical and perfumery industries. They are also sources of aroma chemicals, particularly of enantiomers and useful chiral building blocks in syntheses. Biological and pharmacological activities of essential oils and their constituents have been gathering momentum in recent years. Essential oils therefore will continue to be indispensable natural ingredients. Moreover, the continuous demand for new synthetic compounds reproducing the sensations elicited by natural flavors triggered analytical investigations toward the attainment of information on scarcely known properties of well-known matrices.As evidenced by the numerous techniques described in the present contribution, chromatography, especially GC, has evolved into the dominant method for essential oil analysis. This is to be expected because the complexity of the samples must be unraveled by some type of separation, before the sample constituents can be measured and characterized; in this respect, GC–MS provides the greatest resolving power for most of these volatile mixtures.

Menthalongifolia (Al-Habak) is very famous plant in KSA, commonly used as a flavor agent with tea and some food and because of the interesting biological activities of it, we were interested to study the essential oil composition of Al-Habak from different regions. According to our results which proved that there is obvious difference in the chemical composition between samples of which is Regarding to the difference in environmental and geographical factors which are known to have significant influence on the essential oil composition of the plants. From these results it can be concluded that the chemical composition of the essential oil obtained from the leaves of Al-Habakcollected from fournorth regions of KSA have different qualitative and quantitative properties.

In conclusion, there are some studies have shown the possibility of using essential oil of Menthalongifolia not only as flavor agents but also as safe antioxidant, antimicrobial, antifungal and antiseptic supplements in preventing deterioration of foodstuff, beverage products, pharmaceuticals, folk remedy and natural therapies of infectious diseases in human, and management of plant diseases. Menthalongifolia has these properties because its essential oil contain oxygenated monoterpenes as major component So, the essential oil from our study can be use in some of this applications. Of course, a further study is necessary to further elaborate the biological activities ofMentha essential oils for various applications and some test should be conducted in future, in order to evaluate the safety of these oils before human uses.

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