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RESEARCH ARTICLE

Chemical Composition of Essential Oil and Mineral Contents of *Pulicaria inuloides*

Nabil Qaid M. Al-Hajj[1,2], Chaoyang Ma[1], Riyadh Thabit[1], Ammar Al-alfarga[1], Mohammed A.A. Gasmalla[1], Abubakr Musa[1], Waleed Aboshora[1] and Hongxin Wang[1*]

[1]School of Food Science and Technology, Jiangnan University, Wuxi 214122, PR China;
[2]Marine Science and Biological Research Authority, Aden, P.O. Box-1231, Aden, Yemen
whx1964@126.com*; Tel.: +8651085917795; fax: +8651085876799

Abstract

Essential oils are volatile, natural, complex mixtures of compounds characterized by a strong odour and formed by aromatic plants as secondary metabolites. The chemical composition of the essential oil obtained by hydrodistillation from the whole plant of *Pulicaria inuloides* grown in Yemen and collected at full flowering stage were analyzed by Gas chromatography-Mass spectrometry (GC-MS). Several oil components were identified based upon comparison of their mass spectral data with those of reference compounds. The main components identified in the oil were 47.34% of 2-Cyclohexen-1-one, 2-methyl-5-(1-methyl with Hexadecanoic acid (CAS) (12.82%) and Ethane, 1,2-diethoxy- (9.613%). In this study, mineral contents of whole plant of *P. inuloides* were determined by atomic absorption spectroscopy. Highest level of K, Mg, Na, Fe and Ca of 159.5, 29.5, 14.2, 13.875 and 5.225 mg/100 g were found in *P. inuloides*.

Keywords: Essential oils, *Pulicaria inuloides*, mass spectral data, atomic absorption spectroscopy.

Introduction

Genus *Pulicaria* belonging to the tribe Inuleae of the Asteraceae family consists of ca. 100 species distributed in Europe, North Africa and Asia and five species of this genus reported from Yemen (Dubaie and El-Khulaidi, 2005). Essential oils are volatile, natural, complex compounds characterized by a strong odour and formed by aromatic plants as secondary metabolites. In nature, essential oils play an important role in the protection of the plants as antibacterials, antivirals, antifungals and insecticides. Pulicaria genus is an annual herb producing small bright yellow flowers (Ezoubeiri *et al.,* 2005). The oil of *P. arabica* was characterized by the presence of a high percentage of sesquiterpene hydrocarbons and alcohols, whilst that of *P. undulata* was rich in phenolic compounds and monoterpene hydrocarbons. The oil of *P. undulata* was shown to have insecticidal properties (Elegami *et al.,* 1994). The oil of another Saudi Arabian Pulicaria species has also been studied (Al-Yahya *et al.,* 1989) and the major components were P-caryophyllene and its oxide. *Pulicaria jaubertii* indigenous to Yemen, locally known as Anssif is traditionally used in the Yemeni folk medicine to reduce the symptoms of flu and common cold (Huanga *et al.,* 2010), treat back-pain, intestinal disorders (Ezoubeiri *et al.,* 2005), treat inflammation and also as an insect repellent (Stavri *et al.,* 2008). The flower of *P. jaubertii* was also used as spice to make various delicious foods. Various biological activities have been reported for some species of Pulicaria, such as cytotoxic activity of *P. crispa* and *P. orientalis* (Awadh *et al.,* 2001), antibacterial activity of *P. undulata* and *P. dysenterica* (Bohlmann *et al.,* 1982), antispasmodic activity of *P. glutinosa* (Tanira *et al.,* 1996) and antihistaminic effect of *P. dysenterica* (Mahfouz *et al.,* 1973). No previous phytochemical work has been done on the essential oil of *P. inuloides* up to now. In this study, we have investigated the composition of the essential oil of *P. inuloides* and the composition of whole plant of *P. inuloides* (from Yemen) were investigated by GC-MS. In this study, for the first time, the concentration of macro-minerals (Ca, K, Mg, Na and P) and micro-minerals (Fe, Cu, Zn and Mn) and toxic minerals (Pb and Cd) were estimated in *P. inuloides*.

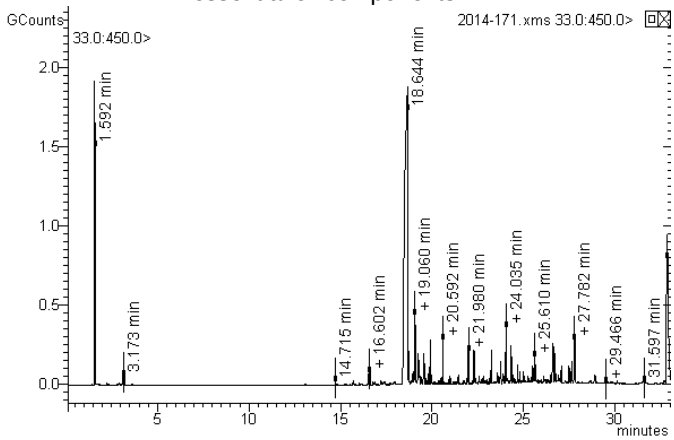
Materials and methods

Plant collection and identification: The whole plant of *P. inuloides* was collected in March 2014 from Bany Mater, province of Sana'a at flowering stage (Fig. 1). The sample was air-dried and taxonomically identified by Prof. Abdellah Amine (College of Agriculture, Sana'a University, Yemen). A voucher specimen of the plant material was deposited at the Dept. of Biology (Sana'a University) of College of Agriculture.

Oil isolation: The whole plant of *P. inuloides* (100 g) was separately subjected to hydrodistillation for 6 h using a Clevenger-type apparatus according to the method recommended in the European Pharmacopoeia (Strasbourg Cedex, 2004). The obtained oils were dried over anhydrous sodium sulphate and stored in air-tight, amber coloured glass vials at 4°C.

Fig. 1. *Pulicaria inuloides.*

Fig. 2. Typical chromatogram of *P. inuloides* essential oil components**.**



GC-MS analysis: The components of the essential oils were identified by GC-MS analysis (Kumar *et al.,* 2010). Gas chromatography-mass spectrometry (Varian 1200L) was incorporated with a relatively no polar capillary column (DB-5, 30 m length, 0.25 mm film thickness, 0.25 internal dia). The injection port and interface were held at 220 and 260°C respectively. The temperature was programmed from 50-220°C at 15°C per min and a hold at 220°C for 25 min with helium as the carrier gas. Mass spectra with electronic impact, ionisation potential of 70 eV, ion source temperature of 200°C and mass range of 35-500 Da was carried out. The identification of individual compounds was based on comparison of their relative retention times with those of authentic samples on HP-5MS capillary column and by matching of their mass spectra of peaks with those obtained from authentic samples and/ or the Wiley NIST7 library spectra and published data (Adams, 1995).

Mineral determination: Minerals were determined in *P. inuloides* from the ash which was prepared and dissolved in 6 M hydrochloric acid and made up to 10 mL. Calcium content was estimated by the titrimetric method of Clark and Collip (1925) and iron content was estimated by UV-Visible spectrophotometer (Shimadzu, UV-160A model) at 480 nm (Clark and Collip, 1925). Phosphorus was analyzed by Ranganna method (AOAC, 1995). The blue colour developed was read at 650 nm in UV-Visible spectrophotometer and expressed as phosphorus (mg/100 g meal). Other minerals were estimated by atomic absorption spectroscopy (Shimadzu AA 6701F, Atomic absorption flame emission spectrophotometer) equipped with hollow cathode lamp.

Results and discussion

The yield of volatile oil of *Pulicaria inuloides* obtained by hydrodistillation of the finely powdered whole plant was 0.5 (v/w). The oil was light yellow and with a perfumery odour. The chemical composition of the oil is presented in Table 1 and Fig. 2. Identification of the constituents was based on comparison of their mass spectra and retention indices with those obtained from authentic samples and Wiley NIST7 library spectra and published data (El-Khh *et al.,* 2009).

Quantification of the components was performed on the basis of their GC peak area. About 60 components were identified in the oil of *P. inuloides*, which represented about 100% of the oil. This oil contained about 47.34% of 2-Cyclohexen-1-one, 2-methyl-5-(1-methyl with Hexadecanoic acid (CAS) (12.82%), Ethane, 1,2-diethoxy- (9.613%) as the main component. High content of oxygenated compounds might explain the characteristic and fragrant odour of the oil. Our results of some components of essential oil of *P. inuloides* showed minor differences when compared with literature (El-Khh *et al.,* 2009). This differences might be due to growth conditions, genetic factors, geographical variations and analytical procedures. In addition, according to previous phytochemical studies, this plant is a considerable source of eudesmanolide, sesquiterpene lactones of the guaianolide and xanthanolides family Asteraceae (Dubaie and El-Khulaidi, 2005). In this study, mineral contents of *P. inuloides* used in Yemen were determined by atomic absorption spectroscopy. The concentrations were calculated on a dry weight basis. Table 2 shows difference between content of plant from minerals. This may be due to various fractions of dissolved organic matter. Highest levels of K, Mg, Na, Fe and Ca were found in *P. inuloides* to be 159.5, 29.5, 14.2, 13.875 and 5.225 mg/100 g respectively. The results were compared with those from the *P. undulata* from Iran (Mehdi *et al.,* 2011). However, toxic mineral concentrations of the studied plant were lower. This work attempts to contribute to the knowledge of nutritional properties of these plants.

Conclusion

The chemical composition and mineral elements in *P. inuloides* suggested that it can be used as an effective natural source of antioxidant and food additives and also a good candidate for phytochemical and pharmacological investigations to discover new broad spectrum bioactive compounds. The major minerals in *P. inuloides* were K, Mg, Na, Fe and Ca which can be considered as a good source of nutrition.

Table 1. Chemical composition of the essential oil of whole plant of *P. inuloides*.

Peak	RT (min.)	Area (%)	Compounds	RT index	Percentage
1	1.592	4.57	Ethane, 1,2-diethoxy-	772	9.613
2	3.173	1.77	No match	-	0.372
3	14.715	6.96	Filifolone	942	0.146
4	15.699	5.73	Dihydroedulan II	909	0.12
5	16.508	1.33	Linalool L	917	0.28
6	16.602	1.91	Carvomenthone	955	0.402
7	16.79	3.39	Terpineol, cis-.beta.-	888	0.071
8	17.206	7.03	No match	-	0.148
9	18.644	2.25	2-Cyclohexen-1-one, 2-methyl-5-(1-methyl	926	47.34
10	18.927	2.03	2-Cyclohexen-1-one, 6-methyl-3-(1-methyl	885	0.004
11	19.06	1.31	Cyclohexanol, 2-methyl-5-(1-methylethyl)	938	2.755
12	19.244	4.89	2-Cyclohexen-1-ol, 2-methyl-5-(1-methyle	883	1.029
13	19.293	3.06	Bicyclo[3.1.1]hept-3-en-2-ol, 4,6,6-trim	842	0.643
14	19.414	1.05	.delta.-Cadinene	882	0.221
15	19.548	5.43	Benzene, 1-(1,5-dimethyl-4-hexenyl)-4-me	892	1.142
16	19.676	8.21	Benzaldehyde, 4-(1-methylethyl)-	883	0.173
17	19.822	1.72	3-Hexadecyloxycarbonyl-5-(2-hydroxyethyl	792	0.361
18	19.92	5.32	Carvotanacetol, cis-	905	1.117
19	20.358	2.57	1-(1'-Acetyl)-2-(2-Oxopropyl)Cyclopentan	743	0.054
20	20.484	8.73	Geranyl propionate	882	0.184
21	20.592	5.32	Thymohydroquinone dimethyl ether	950	1.117
22	20.955	1.60	Thymyl acetate	897	0.337
23	21.185	2.54	Trans-2-Undecen-1-OL	921	0.053
24	21.28	3.31	3,5-Heptadienal, 2-ethylidene-6-methyl-	771	0.07
25	21.424	1.40	.beta.-Ionone	860	0.293
26	21.744	3.89	Dihydro-.alpha.-ionone	797	0.082
27	21.98	5.31	(-)-Caryophyllene oxide	936	1.117
28	22.291	4.83	E-2-Tetradecen-1-ol	947	1.014
29	22.562	1.12	1-Hydroxy-1,7-dimethyl-4-isopropyl-2,7-c	918	0.235
30	22.74	1.13	9-Chloro-8-oxatetracyclo[7.3.1.0(2,7).0(	770	0.237
31	22.813	9.26	cis-Z-.alpha.-Bisabolene epoxide	831	0.195
32	22.916	7.67	trans-Z-.alpha.-Bisabolene epoxide	826	0.161
33	23.02	3.20	Nerolidol	782	0.067
34	23.141	4.82	zingiberenol	842	0.101
35	23.224	3.33	2-Pentadecanone, 6,10,14-trimethyl-	916	0.7
36	23.557	1.20	Alpha Sinensal	816	0.253
37	23.652	8.39	Phenol, 2-methoxy-4-(2-propenyl)- (CAS)	795	0.176
38	23.742	3.26	Thymyl acetate	893	0.685
39	23.903	2.11	No match	-	0.443
40	24.035	7.07	2-Cyclohexen-1-one, 2-methyl-5-(1-methyl	889	1.486
41	24.109	6.41	No match	-	0.135
42	24.314	4.51	.alpha.-Cadinol	911	0.947
43	24.422	7.66	No match	-	0.161
44	24.512	4.99	No match	-	0.105
45	24.677	1.87	1-Hydroxylinalool	806	0.392
46	24.803	1.17	Tetracosane (CAS)	961	0.245
47	24.993	1.96	9,17-Octadecadienal, (Z)-	903	0.412
48	25.251	9.35	Allopregnane-7.alpha.,11.alpha.-diol-3,2	735	0.196
49	25.504	2.90	1-Hexadecanol (CAS)	869	0.61
50	25.61	3.93	cis,cis,cis-7,10,13-	913	0.827
51	25.927	2.83	Hexadecatrienal	805	0.06
52	26.115	1.11	trans-.alpha.-Bergamotene	796	0.233
53	26.197	3.11	Sabinene	787	0.065
54	26.313	4.79	Nerolidol-Epoxyacetate	808	0.101
55	26.481	6.77	2-Methyl-Z,Z-3,13-octadecadienol Phenol, 3-(1,1-dimethylethyl)-4-methoxy-	779	0.142
56	26.593	7.30	Tetracosane	964	1.535
57	26.712	4.41	Acetic acid, 3,7,11,15-tetramethyl-hexad	918	0.927
58	26.901	2.18	Ethyl linoleate	818	0.458
59	27.055	2.59	1,2-Benzenedicarboxylic acid, bis(2-meth	927	0.543
60	27.468	3.07	Hexadecen-1-ol, trans-9-	946	0.646
61	27.62	3.34	Methyl (Z)-5,11,14,17-eicosatetraenoate	872	0.702
62	27.782	8.98	2-Hexadecen-1-ol, 3,7,11,15-tetramethyl-	962	1.887
63	28.886	2.03	1,2-Benzenedicarboxylic acid, butyl 8-me	794	0.427
64	29.466	7.17	Cyclopropaneoctanoic acid, 2-[[2-[(2-eth	773	0.151
65	30.099	5.80	No match	-	0.122
66	31.597	7.01	(12Z)-Abienol	785	0.147
67	32.848	6.10	Hexadecanoic acid (CAS)	947	12.82
				Total = 99.993	

Table 2. The mineral elements in whole plant of *P. inulodies.*

Number	Elements	mg/100 g
1	Pb	0.081
2	Cd	0.09
3	Zn	0.3
4	Fe	13.875
5	Cu	0.144
6	Mn	0.645
7	K	159.5
8	Na	14.2
9	Mg	29.5
10	Ca	5.225
11	P	0.0024

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