VOLATILE CONSTITUENTS OF THE AERIAL PARTS OF *TERATAENIUM LASIOPENTALUM* (BOISS.) MANDEN., STEMS AND LEAVES OF *DOREMA AMMONIACUM* D.DON. AND LEAVES, FRUITS AND STEMS OF *LEUTEA PETIOLARE (DC.)* M.PIMEN FROM IRAN

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ABSTRACT

The Composition of the essential oils from three Umbelliferae species of Iran , Tetrataenium lasiopetalum (Boiss.) Mandem ., Dorema ammoniacum D.Don., which is endemic to Iran, and Leutea petiolare (DC.) M.Pimen. obtained by hydrodistillation were analyzed by GC and GC/MS. Germacrene D (17.72%) and (E,Z) farnesol (12.58%) were the main components in the oil of aerial parts of T. lasiopetalum. The major compound in the stem oil of D. ammoniacum was α-muurolol (13.68%), whereas the leaf oil contained (E)-β-ocimene (30.94%), γ-terpinene (11.09%), p-cymene (10.03%) and (Z)-β-ocimene (7.11%). Terpinolene (13.93%, 8.28% and 10.27%) , (E)-β-ocimene (10.24%, 24.63% and 10.46%) and endo-fenchyl acetate (15.1%, 7.42% and 7.57%) were the main constituents in the leaf, fruit and stem oils of L. petiolare respectively.

Keywords: Tetrataenium lasiopetalum, Dorema ammoniacum, Leutea petiolare, Umbelliferae, Essential oil

INTRODUCTION

The genus Tetrataenium is represented in Iran by two species. T. lasiopetalum (Boiss.) Manden. and T. nephrophyllum (Leutea) Manden.[1,2] No studies on the chemical composition or oils of Tetrataenium species have previously been reported.

The genus Dorema is represented in the flora of Iran by six species, among which two are endemic: D. aucheri Boiss. and D. ammoniacum D. Don.[1,2] D. ammoniacum is a perennial plant growing up to 2.5 m in height in arid and semi-arid regions of central Iran, such as Yazd, Isfahan and Semnan provinces.[1]

This species excudes a medicinal gum resin, commonly known as "Ushaq" or "Vasha" in Iranian traditional medicine and also as "Persian ammoniacum" in Greek and Latin medicinal literature.[3-5] In Iranian folk medicine, this gum resin has been considered useful in treatment of spastic pains, gastric disorders, intestinal parasitic infections and skin inflammations and as analgestic, stimulant, expectorant and laxative.[3,6-8] It has also been traditionally used in Western and Indian medicines as antispasmodic, expectorant, diaphoretic and emmeragogue and also for treatment of catarrh, asthma, chronic bronchitis and persistent coughing.[9,10] D. ammoniacum gum resin has been reported to exhibit some bioactivities including antibacterial, antifungal and acetylcholinesterase inhibitory effects.[11-13].

Free salicylic acid, ammoresinol, ashamirone and some sesquiterpene chroman diones have also been isolated and identified from Persian ammoniacum from Iran. [12-14] Presence of phenolic compounds such as sesquiterpene coumarins, phenols, flavonoids and phloroacetophenone glycosides have been reported from the other Dorema species. [15-18.]

There are also some reports about the essential oils obtained from D. ammoniacum from Iran.[19-23].

The major constituents of the fruits oil of D. ammoniacum, collected from Semnan road toward Firooskuh, were (Z)-ocimenone (22.3%) and (E)-ocimenone (18.1%). The results of the antimicrobial assay of the oil indicated that the oil exhibited moderate to high antimicrobial activity, especially against Bacillus subtillis and Staphylo coccusepidermidis.[19-20.]

The oil from the leaves of the plant contained α-gurjunene (49.5%) and β-gurjunene (19.0%) as the major components.[21]

The essential oils obtained by hydrodistillation of the flowers, stem and roots of D. ammoniacum collected from Hezar mountain in Rayen area, Kerman Province, has been reported. The major components in the flower oils were δ-cadinene (11.58%) and α-himachalene (7.71%). The stem oils contained δ-cadinene (16.24%), liguloxide (8.69%) and δ-amorphene (8.43%) as the major components, while the root oil had 3-n-butyl phthalide (62.49%), benzyl butanoate (6.57%) and liguloxide (5.15%) as the major components. The oils of flower, stem and root were richer in sesquiterpenes than monoterpenes.[22] Recently antioxidant and antibacterial activities of the essential oils and extracts from the aerial parts and roots of D. ammoniacum, collected from Kashan region have been reported. β-Himachalene (9.3%) and β-chamigrene (8.7%) were the main constituents in the aerial parts of the plant whereas β-bisabolene (15.1%) and hexadecanal (13.2%) were dominated in the roots oil. Ethyl acetate extract of the roots showed the highest antioxidant activity in both DPPH and FRAP assays. In antibacterial assay, the ethyl acetate and chloroform extracts of the roots exhibited strong antibacterial activity against Bacillus subtilis, Pseudomonos aeruginosa and Staphylococcus aureus. Essential oils were also found to be active against Shigella dysenteriae.[23] According to M. Pimen point of view the Leutea genus is represented in Iran by five species: L. cupularis (Boiss.) M. Pimen., L. gracillima M. Piman., L. nematoloba (Rech. F.)., L. petiolare (DC.) M. Pimen and L. polycias (Boiss.) M. Pimen.[1.]

In this work, we report the analysis of the essential oils of the aerial parts of Tetrataenium lasiopetalum, stems and leaves of Dorema ammoniacum and leaves, fruits and stems of Leutea petiolare, all found growing wild in Iran.

EXPERIMENTAL

Plant material: The aerial parts of Tetrataenium lasiopetalum was collected from Kellar mountain, Province of Chaharmahal-Bakhtiyari, west of Iran, in July 2013. The stems and leaves of Dorema ammoniacum, which is endemic to Iran, and the leaves, fruits and stems of Leutea petiolare both were collected from Shahroud, Province of Semnan in July 2013. Voucher specimens have been deposited at the Herbarium of the Research Institute of Forests and Rangelands (TARI), Tehran, Iran. Isolation of the essential oils: Air-dried aerial parts of Tetrataenium lasiopetalum (120 g), stems (85 g) and leaves (70 g) of Dorema ammoniacum and leaves (70 g), fruits (60 g) and stems (90 g) of Leutea petiolare were separately subjected to hydrodistillation using a Clevenger-type apparatus for 3h. After decanting and drying of the oils over anhydrous sodium sulfate, the corresponding yellowish coloured oils were recovered [in a yield of 0.2%, 0.3%, 0.7%, 0.4%, 0.2% and 0.1% (w/w)], respectively. Gas chromatography : GC analysis was performed on Schimadzu 15 A gas chromatograph equipped with a split/splitless injector (25 °C) and a flame ionization detector (250 °C). Nitrogen was used as a carrier gas (1mL/min) and the capillary column used was DB-5 (50 m × 0.2 mm, film thickness 0.32 μm). The column temperature was kept at 60 °C for 3 min and then heated to 220 °C with a 5 °C/min rate and kept constant at 220 °C for 5 min. Relative percentage amounts were calculated from peak area using a Schimadzu C-R4A chromatopac without the use of correction factors. Gas chromatography-mass spectrometry : GC/MS analysis was performed using a Hewlett-Packard 5973 MSD detector with a HP-5 MS column (30 m × 0.25 mm, film thickness 0.25 μm). The column temperature was kept at 60 °C for 3 min and programmed to 220 °C at a rate of 5 °C/min and kept constant at 220 °C for 5 min. The injector and GC/MS interphase were maintained at 270 °C. The flow rate of helium, as carrier gas, was 1(mL/min), with a split ratio of 1/50. The ionization voltage was 70 eV. The ion source temperature was 250 °C, and the transfer line temperature was 280 °C. The mass range (m/z) was 45-465 amu at a speed of 2.8 scan/s. The retention indices for all the components were determined according to the Van Den Dool method, using n-alkanes as standards. The compounds were identified (RRI, DBS) by comparison with data reported in the literature and by comparison of their MS with either the Wiley library or with published MS.[24,25]

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RESULTS AND DISCUSSION

The composition of the essential oils from aerial parts of Tetrataenium lasiopetalum, stems and leaves of Dorema ammoniacum and leaves, fruits and stems of Leutea petrolare are listed in Table 1, 2 and 3, respectively, in which the percentage and relative retention indices of compounds are given.

As it is shown from Table 1, about 90.81% (48 components) of the oil of T. lasiopetalum were identified. The oil of the plant consists of five monoterpene hydrocarbons (5.79%), eight oxygenated monoterpenes (6.75%), fifteen sesquiterpene hydrocarbons (43.21%), thirteen oxygenated sesquiterpenes (22.34%) and seven non terpenoid compounds (12.72%.

Germacrene D (17.72%) and (E,Z)-farnesol (12.58%) were the major components in this oil. As can be seen from the above information in the aerial parts oil of T. lasiopetalum sesquiterpenes (65.55%) predominated over monoterpenes (12.54%) and other compounds (12.72%).

As it is shown from the Table 2, sixty-four constituents 89.2% were identified in stem oil of D. ammoniacum: three hydrocarbon monoterpenes (0.62%), five oxygenated monoterpenes (6.93%), ten sesquiterpene hydrocarbons (14.34%), thirteen oxygenated sesquiterpenes (37.89%) and thirty-two non terpenoid compounds (29.42%). α-Muurolol (13.68%) was the most abundant constituent followed by hexadecanoic acid (6.81%) and (E)-nerolidol (5.09%).

Twenty-five constituents 100% were identified in leaf oil: eight monoterpene hydrocarbons (69.49%), five oxygenated monoterpenes (10.45%), six sesquiterpene hydrocarbons (10.26%), four oxygenated sesquiterpenes (7.94%) and two non terpenoid compounds (1.86%).

It was characterized by (E)-β-ocimene (30.94%), γ-terpinene (11.09%), p-cymene (10.03%). Other notable constituents were (Z)-β-ocimene (7.11%), terpinolene (6.19%) and endo-fenchyl acetate (5.25%).

The qualitative and quantitative variation between our results and previous reports for the concentration of the oils from stems and leaves of the plant may be attributed to the different environmental conditions.[21-22]

Previously we reported the main compounds among the 35 constituents characterized in D. aucheri, representing 89.2% of the total components detected.[26] Essential oil of D. glabrum roots have previously reported to contain δ-cadinene (12.8%), β-bisabolene (7.5%) and α-fenchyl acetate (6.3%) as the main compounds.[2]

From Table 3, it is evident that forty-four components representing 91.05%, fifty-three constituents representing 98.03% and thirty-six compounds representing 94.6% were identified in the oils of leaf, fruit and stem of Leutea petiolare respectively.

The main components in all oils were terpinolene (13.93%, 8.28% and 10.27%), (E)-β-ocimene (10.24%, 24.63% and 10.46%) and endo-fenchyl acetate (15.1%, 7.42% and 7.57%), respectively. Other notable compounds were in leaf oil: limonene (9.16%) and (Z)-β-ocimene (7.79%), in fruit oil: p-cymene (11.5%), γ-terpinene (9.72%) and (Z)-β-ocimene (8.32%) and in stem oil: p-cymene (15.43%), hexadecanoic acid (9.01%) and γ-terpinene (5.27%).

As can be seen from the above information, the composition of the leaf, fruit and stem oils of L. petiolare were quite similar. All three oils were rich in regard to monoterpenes (71.85%, 82.07% and 73.53%), respectively. The sesquiterpene fraction of the oils was relatively small, representing (16.98%, 14.51% and 7.96%) of the total oils, respectively.

Previously we reported the essential oil from the aerial parts of L. elbursensis Mozaffarian. Thirty-three compounds were identified in the oil of L. elbursensis, representing 98.0% of the total oil, with α-pinene (37.3%) and β-pinene (36.1%) as the main constituents. The oil was rich in monoterpenes rather than sesquiterpenes.[28]

CONCLUSIONS

This paper presents the chemical composition of the essential oils of the aerial parts of Tetrataenium lasiopetalium, stems and leaves of Dorema ammoniacum and leaves, fruits and stems of Leutea petiolare. In the aerial parts oil of T.lasiopetalum sesquiterpenes predominated over monoterpenes. The composition of the stem oil of D. ammoniacum showed differentially to the leaf oil for the concentration of main compounds, also the stem volatile was rich in sesquiterpenes, while the leaf volatile was rich in monoterpenes. The leaf, fruit and stem oils of L. petiulare were rich in regard to monoterpenes.

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Table 1: Chemical composition of essential oil from aerial parts of Tetrataenium lasiopetalum.

Compound	RI[a]	T. lasiopetalum
Hexanal	800	0.15
3-Nonene	893	0.10
Nonane	899	0.17
α-Pinene	939	1.14
Sabinene	976	0.32
β-Pinene	980	1.06
Car-3-ene	1011	1.58
1,8-Cineole	1033	1.85
Terpinolene	1088	1.69
iso-Pentyl isovalerate	1103	8.53
Hexyl isobutyrate	1150	1.73
Pinocarvone	1162	0.18
Lavandulol	1166	0.12
Terpin-4-ol	1177	1.86
α-Terpineol	1189	0.48
Carvone	1242	1.06
Lavandulyl acetate	1289	1.06
Thymol	1290	0.14
δ-Elemene	1339	0.18
Neryl acetone	1365	0.24
α-Copaene	1376	0.16
β-Bourbonene	1397	3.13
-α Gurjunene	1409	2.17
β-Cedrene	1415	4.08
β-Gurjunene	1432	1.96
Aromadendrene	1439	0.26
(E)-β-Farnesene	1458	0.65
Germacrene D	1480	17.72
Viridiflorene	1493	3.59
α-Muurolene	1499	0.52
(E,E)-α-Farnesene	1508	3.29
δ-Cadinene	1524	1.86
β-Sesquiphellandrene	1526	3.00
(E)-γ-Bisabolene	1533	0.64
(E)-Nerolidol	1534	0.61
Elemol	1549	0.20
Geranyl isovalerate	1597	4.16
Cubenol	1642	0.62
α-Muurolol	1645	0.66
α-Cadinol	1653	2.45
Ar-Turmerone	1664	0.38
(Z,E)-Farnesol	1697	12.58
(E,E)-Farnesol	1772	0.12
(E,Z)-Farnesol	1742	0.10
Tetradecanoic acid	1771	0.33
(Z,E)-Farnesyl acetate	1818	0.12
(E,E)-Farnesyl acetate	1843	0.10
Hexadecanoic acid	1973	1.71
Monoterpene hydrocarbons		5.79
Oxygenated monoterpenes		6.75
Sesquiterpene hydrocarbons		43.21
Oxygenated sesquiterpenes		22.34
Other compounds		12.72
Total		90.81

[a] retention indices as determined on a DB-5 column using a homologous series of n-alkanes.

Table 2: Percentage composition of stem and leaf oils of Dorema ammoniacum.

Compound	RI[a]	Stem	Leaf
4-methylene-5-Hexenal	893	0.17	_
α-Pinene	935	_	2.37
Myrcene	991	_	0.70
6-methyl-5-Hepten-2-one	1010	0.17	_
Allyl tiglate	1022	0.22	_
p-Cymene	1024	0.21	10.03
Limonene	1031	_	1.06
(Z)-β-Ocimene	1040	_	7.11
(E)-β-Ocimene	1050	0.23	30.94
γ-Terpinene	1060	1.09	
Terpinolene	1086	0.18	6.19
endo-Fenchol	1112		0.55
(E)-2-Nonenal	1150	0.16	
Borneol	1163	_	2.59
Dodecane	1199	0.26	
trans-Carveol	1217	_	1.48
endo-Fenchyl acetate	1220	0.75	5.25
Decanol	1272	0.37	
Bornyl acetate	1284	_	0.58
Undecanal1304	0.24		
2-Undecanol	1312	0.83	_
(E)-2-Decen-1-ol	1375	1.09	_
4-ethyl-1-ethylen Benzene	1392	0.21	_
Tetradecane	1399	0.31	_
2-Dodecanol	1410	0.39	_
Dodecanal1415	0.96		
β-Caryophyllene	1418	4.39	0.90
Neryl acetone	1434	0.40	
2-methyl-butyl Benzoate	1436	_	0.89
trans-α-Bergamotene	1438	0.12	_
1-methoxy-Naphthalene	1441	0.34	_
Geranyl acetone	1453	3.89	_
Neryl propanoate	1456	0.59	_
(E)-β-Farnesene	1458	0.61	4.42
β-Acoradiene	1464	0.70	_
γ-Gurjunene	1473	0.31	_
Germacrene D	1480	0.51	2.29
(Z)-8-Dodecen-1-ol	1483	0.88	_
2-Tridecanone	1492	0.87	_
Bicyclogermacrene	1494	_	1.16
2-Pentadecanol	1502	0.98	_
β-Bisabolene	1509	3.04	0.85
γ-Cadinene	1511	2.38	_
δ-Cadinene	1524	2.03	_
(Z)-Nerolidol	1532	0.18	_
α-Calacorene	1542	0.25	_
cis-Sesquisabinene hydrate	1545	3.35	_
Germacrene B	1554	_	0.64
Geranyl-n-butyrate	1562	1.30	_
(E)-Nerolidol	1564	5.09	0.57
Spathulenol	1576	0.89	4.23
Caryophyllene oxide	1581	3.41	0.91
Tetradecanal	1598	3.58	_
iso-amyl Nerolate	1601	0.25	_
γ-Eudesmol	1628	0.17	_
α-Muurolol	1645	13.68	2.23
α-Eudesmol	1650	2.66	_
α-Cadinol1653	4.47		
α-Bisabolol	1680	0.42	_
Acorenone	1685	1.59	_
1,4-dimethoxy Naphthalene	1690	0.75	_
2-Pentadecanone	1695	2.07	_
2-Pentadecanol	1705	0.30	_
(E,E)-Farnesol	1722	1.73	_
(E,E)-3,7,11-trimethyl, 2,6,10-Dodecatrienal	1740	1.02	_
benzyl Benzoate	1762	0.32	_
Tetradecanoic acid	1770	0.64	_
Octadecane	1800	0.33	_
cis-9-Hexadecanal	1812	0.73	_
Hexadecanol	1820	2.40	_
6,10,14-trimethy l-2- Pentadecanone	1840	0.17	_
Pentadecanoic acid	1868	0.21	_
(E)-5-Octadecene	1876	0.42	
Hexadecanoic acid	1976	6.81	0.97
(Z,Z)-9,12-Octadecadienoic acid	2140	1.22	_
Monoterpene hydrocarbons		0.62	69.49
Oxygenated monoterpenes		6.93	10.45
Sesquiterpene hydrocarbons		14.34	10.26
Oxygenated sesquiterpenes		37.89	7.94
Other compounds		29.42	1.86
Total	89.2	100	

[a] retention indices as determined on a DB-5 column using a homologous series of n-alkanes.

Table 3 : Comparative percentage composition of the leaf, fruit and stem oils of Leutea petiolare.

Compound	RI[a]	Leaf	Fruit	Stem
α-Pinene	939	4.22	3.34	1.83
Camphene	951	0.67	0.28	_
Sabinene	976	0.66	0.24	_
β-Pinene	980	_	0.45	
Myrcene	991	2.23	1.07	1.96
p-Cymene	1024	_	11.50	15.43
Limonene	1028	9.16	2.39	2.35
(Z)-β-Ocimene	1040	7.79	8.32	4.08
(E)-β-Ocimene	1050	10.24	24.63	10.46
γ-Terpinene	1061	0.96	9.72	5.75
Terpinolene	1086	13.93	8.28	10.27
Linalool	1096	_	0.11	_
cis-Thujone	1101	0.25	0.13	_
endo-Fenchol	1111	_	_	4.32
exo-Fenchol	1114	1.82	0.75	_
allo-Ocimene	1127	0.88	0.66	_
trans-Sabinol	1140	0.26	0.10	_
4,8-epoxy-p-Menth-1-ene	1145	_	_	0.45
trans-epoxy Ocimene	1147	0.16	_	_
Menthofuran	1164	0.16	_	
Borneol	1165	_	_	3.78
p-Mentha-1,5 dien-8-ol	1166	0.40	_	_
Terpin-4-ol	1176	_	0.21	_
p-methyl Acetophenone	1182	0.49	0.16	_
p-Cymene-8-ol	1183	1.71	0.59	0.96
methyl Salicylate	1189	_	_	0.52
trans- dihydro Carvone	1200	_	0.18	_
trans- Carveol	1215	_	0.79	0.48
endo-Fenchyl acetate	1220	15.11	7.42	7.57
Thymol methyl ether	1232	_	_	0.46
Pulegone	1235	0.19	_	_
Cumin aldehyde	1239	_	_	0.88
Bornyl acetate	1285	0.99	0.42	0.62
Thymol	1290	_	_	0.37
Carvacrol	1296	0.22	0.19	1.19
Menth-1-ene-4,8-diol	1305	_	0.14	_
δ-Elemene	1336	0.28	_	_
α-Copaene	1374	0.31	_	_
β-Cubebene	1390	0.46	_	
Dodecanal	1406	_	_	0.32
β-Caryophellene	1418	2.11	0.88	_
β-Gurjunene	1430	0.12	_	_
γ-Elemene	1432	_	0.18	_

Compound	RI[a]	Leaf	Fruit	Stem
2-methyl-buthyl Benzoate	1436	_	0.46	_
Geranyl acetone	1453	_	_	0.8
α-Humulene	1454	0.91	0.15	_
(E)-β-Farnesene	1456	0.67	2.78	0.27
Germacrene D	1480	3.08	1.84	_
Bicyclogermacrene	1494	_	0.4	_
trans-β-Guaiene	1500	0.23	_	_
β-Bisabolene	1506	0.77	0.61	0.98
γ-Cadinene	1510	0.46	0.12	_
δ-Cadinene	1521	0.88	0.48	_
iso-penthyl Salicylate	1531	_	0.19	_
Elemol	1543	_	0.16	_
Germacrene B	1552	_	0.36	_
(E)-Nerolidol	1564	_	0.30	0.49
1,5-epoxy Salvial-4(14)-en	1565	0.14	_	_
(Z)-3-hexenyl Benzoate	1570	0.49	0.29	_
Spathulenol	1576	2.99	2.88	0.63
Caryophyllene oxide	1581	_	0.60	0.66
Salvial-4(14)-en-1-one	1589	0.27	0.19	_
β-Oplopenone	1604	0.27	0.17	_
(Z)-Sesquilavandulol	1606	_	_	0.68
Tetradecanal	1611	_	_	0.23
(E)-Sesquilavandulol	1632	0.29	0.23	_
Isospathulenol	1642	_	0.19	_
α-Muurolol	1644	2.06	0.86	3.47
β-Eudesmol	1647	_	0.18	_
α-Cadinol	1653	_	0.76	_
Khusinol	1674	0.68	0.19	_
Acorenone	1682	_	_	0.40
(E,E)-Farnesol	1720	_	_	0.38
benzyl Benzoate	1730	0.12	0.11	0.45
6,10,14-trimethyl-2-Pentadecenone	1872	0.16	_	0.77
Hexadecanoic acid	1973	0.96	0.24	9.01
(Z,Z)-9,12-Octadecadienoic acid	2142	_	_	1.81
Monoterpene hydrocarbons		50.74	70.88	51.65
Oxygenated monoterpenes		21.11	11.19	21.88
Sesquiterpene hydrocarbons		10.28	6.80	2.05
Oxygenated sesquiterpenes		6.70	7.71	5.91
Other compounds		2.22	1.45	13.11
Total		91.05	98.03	94.06

[a] retention indices as determined on a DB-5 column using a homologous series of n-alkanes.

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