SHORT REPORT

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Chemical Composition of Vegetative Parts and Flowers Essential Oils of Wild *Anvillea garcinii* Grown in Saudi Arabia

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Abstract: The flowers and vegetative parts essential oils of *Anvillea garcinii* were analyzed by GC-FID and GC-MS using polar and nonpolar columns which led to the identification of total 140 compounds from both oils, among which 130 compounds were identified for the first time in the genus *Anvillea*. In the flowers oil 126 compounds were identified, whereas 119 compounds were identified in the vegetative parts oil of *A. garcinii* representing 95.7% and 94.9% of the total oil composition, respectively. The major components in the flowers oil were bornyl acetate (33.7%), *cis*-nerolidol (7.3%) and camphene (6.1%). In contrast, the major compounds in the vegetative parts oil were *cis*-nerolidol (16.0%), terpinen-4-ol (10.4%) and cabreuva oxide B (6.4%).

Keywords: Asteraceae; essential oils; bornyl acetate; cabreuva oxide; *Anvillea garcinii*.

1. Plant Source

Anvillea is a small genus of plants that belong to the Asteraceae daisy family, which comprises four species distributed in the region from North Africa to Iran, including several Middle Eastern countries, such as Egypt, Palestine and Saudi Arabia [1]. In Saudi Arabia, the *Anvillea* genus is represented by two species: *A. garcinii* and *A. radiata*. The former species, *A. garcinii* DC (Arabic name: Nuqd), is also called Arabian oxeye.

The whole wild *A. garcinii* plant was obtained from Wadi Al-Hair, central region of Saudi Arabia during the flowering stage in March 2011. The identification of the plant species was confirmed by Dr. Jacob Thomas Pandalayil (plant taxonomist) from the Herbarium Division, King Saud University, Riyadh, Saudi Arabia. A voucher specimen (no. KSU-21599) of the plant material is kept in our laboratory and also deposited in the herbarium division of King Saud University.

2. Previous Studies

Phytochemical investigations on the various parts of *A. garcinii* have led to the isolation of various biologically active classes of compounds, including germacranolides [2-6], flavanoids and their glycosides [7].

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Germacranolides such as 9-α-hydroxyparthenolide, 9-α-hydroxy-1β,10-α-epoxyparthenolide and their isomers isolated from this plant have been shown to exhibit anti-tumor, anti-HIV and cytotoxic activity in both *in vitro* and *in vivo* assays [3,5,6]. Furthermore, hypoglycemic activity has also been reported for this plant [8].

Although the literature contains several reports on the isolation of phytoconstituents from *A. garcinii* and their biological activities [2-8], no studies on the volatile constituents of the genus *Anvillea* have been reported, with the exception of one short report [9] that did not provide a detailed identification of the volatile components. To the best of our knowledge, no reports have been published on the essential oil constituents of *A. garcinii* grown in Saudi Arabia. As a result of this and in continuation of our interest in the chemistry of volatile constituents of aromatic plants found in Saudi Arabia [10,11], we decided to perform a detailed chemical characterization of essential oils derived from the flowers and vegetative parts of wild *A. garcinii* grown in Saudi Arabia using GC-FID and GC-MS analyses as well as linear retention indices (LRI) measurements performed on polar and nonpolar columns.

3. Present Study

This study provides a detailed characterization of the chemical constituents of essential oils derived from *A. garcinii* in Saudi Arabia. The hydrodistillation of flowers and vegetative parts (leaves and stems) of *A. garcinii* produced light-yellowish oils in yields of 0.05 and 0.04%, v/w, respectively, based on the fresh weight. The phytochemical analysis of the obtained essential oils was performed using GC–MS and GC–FID with polar and nonpolar columns (*cf.* experimental detail in supporting information, S1, S2). These experiments resulted in the identification of 140 compounds from both oils. Among these compounds, 130 were identified for the first time in the genus *Anvillea*. In the oil derived from flowers of *A. garcinii*, 126 compounds were identified, whereas 119 compounds were identified in the oil derived from vegetative parts; these compounds account for 95.7% and 94.9% of the total oil compositions, respectively. The identified compounds and their relative contents are listed in Table 1 according to their elution order on a nonpolar column (HP-5MS). While, GC–FID chromatograms with identified peaks of major components of *A. garcinii* flowers and vegetative parts essential oils on HP-5MS column are shown in Figure S3 and Figure S4 (supporting information), respectively.

Table 1 shows that the oil from flowers of *A. garcinii* was dominated by oxygenated monoterpenes (51.0%) followed by oxygenated sesquiterpenes (27.1%) and monoterpene hydrocarbons (9.0%). Oxygenated aliphatic hydrocarbons (3.8%), sesquiterpene hydrocarbons (2.2%), aromatics (1.3%), aliphatic hydrocarbons (0.5%), and diterpenes (0.8%) were found in low to minute concentrations. In contrast, the oil from the vegetative parts was dominated by oxygenated sesquiterpenes (46.2%) followed by oxygenated monoterpenes (28.3%), monoterpene hydrocarbons (5.7%) and oxygenated aliphatic hydrocarbons (5.0%). Sesquiterpene hydrocarbons (4.9%), diterpenes (2.9%), aliphatic hydrocarbons (1.2%) and aromatics (0.7%) were present in low to minute concentrations (Figure 1). The main constituents of the oil from the flowers included bornyl acetate (33.7%), *cis*-nerolidol (7.3%), camphene (6.1%), lavanduly-2-methylbutanoate (3.8%), terpenen-4-ol (3.3%) and borneol (3.1%), whereas the oil from the vegetative parts included *cis*-nerolidol (16.0%), terpenen-4-ol (10.4%), cabreuva oxide B (6.4%), cabreuva oxide D (5.2%) and bornyl acetate (3.4%).

A comparison of the two oils based on the data in Table 1 reveals that the oxygenated mono- and sesquiterpenes were the most prevalent groups in both oils, accounting for 78.1% and 74.5% of the total oil compositions of flowers and vegetative parts oil, respectively. At the same time, the content of each component in the two oils differed significantly. In the oil from flowers, the oxygenated monoterpenes (51.0%) were found at a higher concentration than were the oxygenated sesquiterpenes (27.1%; Figure 1); the reverse was true in the case of the oil from vegetative parts, wherein the oxygenated sesquiterpenes were the major component (46.2%) relative to the oxygenated monoterpenes (28.3%; Figure S5).

The results in Table 1 indicate that the oils from flowers and vegetative parts were similar in terms of their chemical constituents, with as many as 105 constituents observed in both oils. However, the two oils differed significantly with one another in terms of their relative compositions. For example, the linalool (LRI[a]; 1099), *β*-maaliene (LRI[a]; 1386) and khusimone (LRI[a]; 1603) contents were 2-3 times greater in the oil from flowers than in the oil from vegetative parts, whereas the camphene hydrate (LRI[a]; 1150), 2-methyl butyl benzoate (LRI[a]; 1440) and *α*-sinensal (LRI[a]; 1754) contents were 4-5 times greater. In contrast, the content of *α*-pinene (LRI[a]; 933), *p*-cymene (LRI[a]; 1024), 1,8-cineole (LRI[a]; 1031) and *cis*-dracunculifoliol (LRI[a]; 1556) was approximately 2-3 times greater in the oil from vegetative parts.

Notably, the amounts of major constituents such as camphene (LRI[a]; 948), borneol (LRI[a]; 1167), bornyl acetate (LRI[a]; 1289) and lavanduly-2-methylbutanoate (LRI[a]; 1514) were 5-10-fold greater in the oil from flowers than in the oil from vegetative parts. At the same time, the amount of major constituents such as terpenen-4-ol (LRI[a]; 1179), cabreuva oxide B (LRI[a]; 1465), cabreuva oxide D (LRI[a]; 1483) and *cis*-nerolidol (LRI[a]; 1539) was 2-6 times greater in the oil from vegetative parts than in the oil from flowers. In addition to the previously mentioned qualitative similarities and differences between the two oils, 21 compounds (3.5% of oil composition) were found only in the oil from flowers, whereas 14 compounds (3.2% of oil composition) were found only in the oil from vegetative parts. Moreover, *δ*-guaiene (LRI[a]; 1512), which is a sesquiterpene detected in appreciable quantities in the oil from vegetative parts, was not observed in the flowers oil. Conversely, himachalol (LRI[a]; 1650), which is an oxygenated sesquiterpenes detected in a significant amount in the oil from flowers, was not present in the oil from vegetative parts.

A comparison of the results of our present study and that of the leaves and flowers oils of *A. garcinii* reported by Rustaiyan *et al.* [9] from Iran revealed that among the 140 constituents identified in this study and the 24 identified in the Iranian study, only four constituents were common: *α*-pinene (LRI[a]; 933), limonene (LRI[a]; 1029), 1,8-cineole (LRI[a]; 1031) and bornyl acetate (LRI[a]; 1289). In addition, with the exception of bornyl acetate and 1,8-Cineole the contents of the other two constituents (i.e., *α*-pinene and limonene) differed considerably between the different oil samples. Notably, bornyl acetate and 1,8-cineole were the compounds that were present in noticeable amount in all of the *A. garcinii* oil samples analyzed till date (*cf.* Table 2 of supporting information). Hence, bornyl acetate and 1,8-cineole may be considered to be marker compounds of essential oils derived from *A. garcinii*.

Table 1. Composition of essential oils derived from flowers and vegetative parts of *Anvillea garcinii* grown in Saudi Arabia.

SL. No.	Compounds*	LRI[a]	LRI[p]	Flowers (%)	Leaves & Stems (%)	Identification[b]
1	1, 3, 5,7-Cyclooctatetraene	890	1255	0.1	0.1	LRI, MS
2	*α*-Thujene	927	1023	t	0.1	LRI, MS
3	*α*-Pinene	933	1018	0.9	1.7	LRI, MS, Cs
4	Camphene	948	1060	6.1	0.5	LRI, MS, Cs
5	Sabinene	973	1118	0.1	0.3	LRI, MS
6	*β*-Pinene	976	1103	0.9	0.7	LRI, MS, Cs
7	6-Methyl-5-hepten-2-one	987	1339	t	t	LRI, MS
8	Dehydro-1,8-cineole	991	-	0.1	0.1	LRI, MS
9	*β*-Myrcene	992	1164	t	-	LRI, MS
10	*α*-Phellandrene	1008	-	t	-	LRI, MS, Cs
11	*p*-Cymene	1024	1268	0.6	1.9	LRI, MS, Cs
12	Limonene	1029	1197	0.3	0.3	LRI, MS, Cs
13	1,8-Cineole	1031	1208	1.4	2.9	LRI, MS
14	*γ*-Terpinene	1059	1245	-	t	LRI, MS
15	*α*-Terpinolene	1089	-	t	0.1	LRI, MS, Cs
16	Linalool	1099	1550	0.4	0.2	LRI, MS, Cs
17	Nonanal	1104	1393	0.1	0.1	LRI, MS
18	2,6-Dimethyleneoct-7-en-3-one	1116	-	t	0.1	LRI, MS
19	*cis-p*-Ment-2-en-1-ol	1122	1613	0.3	0.6	LRI, MS
20	*α*-Campholenal	1127	1495	t	t	LRI, MS
21	*trans-p*-Mentha-2,8-dien-1-ol	1132	1641	0.1	0.1	LRI, MS

22	*p*-Mentha-1,4,8-triene	1135	-	0.1	0.1	LRI, MS
23	*trans*-Pinocarveol	1140	1661	0.3	0.7	LRI, MS
24	Camphor	1146	1518	0.2	0.4	LRI, MS
25	Camphene hydrate	1150	1611	0.9	0.2	LRI, MS
26	*cis*-Tagetone	1152	1525	-	0.1	LRI, MS
27	Nerol oxide	1155	1472	0.1	-	LRI, MS
28	*iso*-Borneol	1158	1670	0.3	-	LRI, MS
29	Pinocarvone	1165	1568	0.1	-	LRI, MS
30	Borneol	1167	1707	3.1	0.5	LRI, MS
31	Lavandulol	1170	1677	0.1	0.1	LRI, MS
32	Terpinen-4-ol	1179	1607	3.3	10.4	LRI, MS, Cs
33	*p*-Cymen-8-ol	1186	1852	0.3	0.5	LRI, MS
34	*α*-Terpineol	1191	1702	0.8	1.3	LRI, MS, Cs
35	Myrtenal	1194	1633	-	0.1	LRI, MS
36	Myrtenol	1198	1799	0.2	0.3	LRI, MS
37	*n*-Decanal	1205	-	0.1	0.1	LRI, MS
38	Verbenone	1211	1727	-	0.1	LRI, MS
39	*iso*-Dihydrocarveol	1217	1793	0.1	-	LRI, MS
40	*trans*-Carveol	1220	1840	0.2	0.2	LRI, MS
41	*endo*-Fenchyl acetate	1223	1474	0.1	0.2	LRI, MS
42	*cis*-Carveol	1227	1866	t	0.2	LRI, MS
43	Nerol	1228	-	t	-	LRI, MS
44	*cis*-1(7),8-*p*-Menthadien-2-ol	1231	1891	0.1	0.1	LRI, MS
45	(3*Z*)-4,8-Dimethyl-3,7-nonadien-2-one	1237	1601	0.3	0.7	LRI, MS
46	*trans*-Tagetenone (*E*-Ocimenone)	1242	1871	0.1	0.1	LRI, MS
47	Cuminaldehyde	1245	1782	0.1	0.1	LRI, MS
48	Geraniol	1254	-	0.3	0.1	LRI, MS
49	Linalool acetate	1257	1558	t	0.1	LRI, MS
50	*cis*-Chrysanthenyl acetate	1261	1561	-	0.1	LRI, MS
51	Geranial	1265	1732	t	-	LRI, MS
52	*cis*-Verbenyl acetate	1271	-	0.1	0.1	LRI, MS
53	4,8-Dimethyl-nona-3,8-dien-2-one	1275	1656	0.7	1.5	LRI, MS
54	Perilla aldehyde	1280	1787	0.1	0.1	LRI, MS
55	Bornyl acetate	1289	1586	33.7	3.4	LRI, MS
56	*p*-Cymen-7-ol	1291	2102	0.3	-	LRI, MS
57	Thymol	1294	2163	t	-	LRI, MS, Cs
58	(4*R*, 8*S*)-*p*-Menth-1-en-9-ol	1297	-	0.1	0.1	LRI, MS
59	(4*R*, 8*R*)-*p*-Menth-1-en-9-ol	1298	-	-	t	LRI, MS
60	Perilla alcohol	1300	2002	0.2	0.3	LRI, MS
61	Thujyl acetate	1302	1528	0.2	0.2	LRI, MS
62	Carvacrol	1303	2218	-	0.2	LRI, MS, Cs
63	*cis*-Pinocarvyl acetate	1307	1653	0.1	-	LRI, MS
64	*iso*-Ascaridole[c]	1309	-	0.1	0.1	LRI, MS
65	Chrysanthenone epoxide	1321	-	0.1	0.2	LRI, MS
66	Myrtenyl acetate	1328	1691	0.3	0.1	LRI, MS
67	*iso*-Butyl benzoate	1330	-	0.2	-	LRI, MS
68	*trans*-Carvyl acetate	1336	-	0.1	0.2	LRI, MS
69	Piperitenone	1342	1899	t	-	LRI, MS
70	*α*-Terpinyl acetate	1346	-	0.1	0.1	LRI, MS
71	*nor*-Chrysanthemic acid methylester	1349	2438	t	0.1	LRI, MS
72	Dehydro-*ar*-ionene	1356	-	0.1	-	LRI, MS
73	Eugenol	1361	-	-	0.3	LRI, MS, Cs
74	*cis*-Carvyl acetate	1366	-	0.7	-	LRI, MS
75	*α*-Copaene	1379	-	0.1	-	LRI, MS
76	*β*-Maaliene	1386	-	0.2	0.1	LRI, MS
77	*cis*-*β*-Damascenone	1387	1825	-	0.1	LRI, MS
78	*cis*-Jasmone	1394	1949	0.2	0.2	LRI, MS
79	*α*-Funebrene[c]	1401	-	0.1	0.2	LRI, MS
80	*β*-Funebrene[c]	1412	-	-	0.1	LRI, MS
81	*trans*-Caryophyllene	1425	-	0.1	0.3	LRI, MS
82	2-Methyl butyl benzoate	1440	1913	0.5	0.1	LRI, MS
83	Cabreuva oxide A	1448	1686	0.2	0.5	LRI, MS
84	*exo*-Arbozol[c]	1453	-	0.1	0.1	LRI, MS
85	*α*-Patchoulene	1460	2182	0.1	0.1	LRI, MS
86	Cabreuva oxide B	1465	1712	2.2	6.4	LRI, MS
87	Cabreuva oxide C	1470	1737	0.3	0.5	LRI, MS
88	Geranyl propanoate	1478	1814	t	-	LRI, MS
89	*γ*-Selinene	1480	-	-	0.3	LRI, MS
90	Cabreuva oxide D	1483	1754	1.6	5.3	LRI, MS
91	*trans*-*β*-Ionone	1486	1944	t	-	LRI, MS

92	*β*-Selinene	1490	1723	0.4	0.5	LRI, MS
93	*α*-Selinene	1493	1734	0.2	0.4	LRI, MS
94	*β*-Dihydroagarofuran	1496	1698	0.3	0.6	LRI, MS
95	*δ*-Guaiene (*α*-Bulnesene)	1512	1727	-	1.5	LRI, MS
96	Lavandulyl-2-methylbutanoate	1514	1781	3.8	0.8	LRI, MS
97	*δ*-Cadinene	1521	-	0.2	0.2	LRI, MS
98	7-*epi*-*α*-Selinene	1524	1767	0.1	0.2	LRI, MS
99	*trans*-Calamenene	1528	1836	0.1	0.1	LRI, MS
100	Kessane	1535	1773	0.2	0.4	LRI, MS
101	*cis*-Nerolidol	1539	2018	7.3	16.0	LRI, MS
102	Liguloxide	1544	1796	0.2	0.4	LRI, MS
103	*cis*-Dracunculifoliol	1556	-	0.4	1.1	LRI, MS
104	*trans*-Nerolidol	1565	2037	0.9	1.6	LRI, MS
105	Dodecanoic acid	1571	2486	0.1	0.1	LRI, MS
106	*cis*-3-Hexenyl benzoate	1574	2119	0.3	0.4	LRI, MS
107	*n*-Hexyl benzoate	1580	2075	0.2	0.2	LRI, MS
108	Caryophyllene oxide	1591	1990	1.2	2.0	LRI, MS, Cs
109	Helifolen-12-al B[c]	1598	-	0.3	0.6	LRI, MS
110	Khusimone	1603	-	0.3	0.1	LRI, MS
111	Tetradecanal	1614	1924	0.6	0.8	LRI, MS
112	Sesquithuriferol	1619	-	0.8	0.9	LRI, MS
113	Caryophylla-4-(14),8(15)-dien-5-*α*-ol	1627	-	0.3	0.5	LRI, MS
114	*γ*-Eudesmol	1632	2171	0.1	0.1	LRI, MS
115	*epi*-*α*-Cadinol	1638	2167	0.1	0.2	LRI, MS
116	*α*-Muurolol	1641	2178	0.2	-	LRI, MS
117	*cis*-Methyl jasmonate	1644	2311	0.3	0.3	LRI, MS
118	Himachalol	1650	2238	1.2	-	LRI, MS
119	*β*-Eudesmol	1652	2233	0.9	1.3	LRI, MS
120	*α*-Eudesmol	1658	2221	0.8	1.6	LRI, MS
121	Intermedeol	1667	2243	0.2	0.6	LRI, MS
122	*Z*-*α*-Santalol	1670	-	0.3	0.4	LRI, MS
123	*β*-Bisabolol	1676	2142	0.9	1.3	LRI, MS
124	1-Tetradecanol	1679	2154	0.3	0.8	LRI, MS
125	Eudesma-4(15),7-dien-1-*β*-ol	1688	2387	0.8	1.5	LRI, MS
126	Geranyl tiglate	1697	2099	0.3	0.6	LRI, MS
127	*n*-Heptadecane	1700	1700	0.4	0.9	LRI, MS, Cs
128	Pentadecanal	1714	2050	0.2	0.2	LRI, MS
129	Methyl tetradecanoate	1721	1998	0.4	1.1	LRI, MS
130	(*Z*, *E*)-Farnesol	1728	2366	0.3	0.7	LRI, MS, Cs
131	*α*-Sinensal	1754	2305	0.4	0.1	LRI, MS
132	*γ*-Curcumen-15-al	1758	-	0.3	0.1	LRI, MS
133	Tetradecanoic acid	1764	2693	0.3	0.3	LRI, MS
134	8,8-Dimethyl-9-methylene-1,5-cycloundecadiene	1774	-	0.6	0.9	LRI, MS
135	Palmitic acid	1959	-	1.6	1.4	LRI, MS
136	*n*-Heneicosane	2100	2100	t	-	LRI, MS, Cs
137	Phytol	2125	2619	0.8	2.9	LRI, MS, Cs
138	*cis*-13-Octadecen-1-yl acetate	2195	-	0.1	0.1	LRI, MS
139	*n*-Docosane	2200	2200	-	0.1	LRI, MS, Cs
140	*n*-Tricosane	2300	2300	-	0.1	LRI, MS, Cs
Chemical class composition						
Monoterpenehydrocarbons				*9.0*	*5.7*	
Oxygenated monoterpenes				*51.0*	*28.3*	
Sesquiterpene hydrocarbons				*2.2*	*4.9*	
Oxygenated sesquiterpenes				*27.1*	*46.2*	
Aliphatic hydrocarbons				*0.5*	*1.2*	
Oxygenated aliphatic hydrocarbons				*3.8*	*5.0*	
Aromatics				*1.3*	*0.7*	
Diterpenes				*0.8*	*2.9*	
Total identified				**95.7**	**94.9**	
Oil yield (%, v/w, fresh weight basis)				***0.05%***	***0.04%***	

*Compounds are listed in their order of elution from HP-5 MS column; LRI[a]: Determined linear retention index against mixture of *n*-alkanes (C8-C27) on HP-5 MS column; LRI[p]: Determined linear retention index against mixture of *n*-alkanes (C8-C27) on DB-wax column; [b]Identification: LRI: Linear Retention index indentical to literatures (*cf.* exp. part); MS: Comparison of mass spectra with the library entries of mass spectra databases (*cf.* exp. part); [c]Tentatively identified; Cs: coinjection/comparison with the LRI and mass spectra of standards; t: Trace (<0.05%).

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Supporting Information

Supporting Information accompanies this paper on http://www.acgpubs.org/RNP

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