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CHEMICAL COMPOSITION OF ESSENTIAL OILS AND LIPOPHILIC EXTRACTS OF *Silphium integrifolium* AND *S. trifoliatum* INFLORESCENCES

R. Kowalski

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The purpose of the present paper is a qualitative and quantitative evaluation of essential oils and constituents present in lipophilic extracts from *Silphium integrifolium* Michx., and *Silphium trifoliatum* L. (Asteraceae) inflorescences.

Inflorescences of *S. integrifolium* and *S. trifoliatum* contained up to 0.35 % and 0.37 % v/w DM of essential oil, respectively (Table 1). Inflorescences of *S. perfoliatum* studied earlier contained 0.28 % v/w DM of oil, on average [1].

After petroleum ether evaporation, extracts were brown and were characterized by greasy consistence (Table 1). Dry remains from *S. integrifolium* and *S. trifoliatum* extracts were respectively up to 4.28 % and 4.56 % DM of examined organs. Literature data indicate that the dry ether extract content in *S. perfoliatum* was 4.53 % DM, on average [2].

The following compounds dominated in the essential oils and extracts from inflorescences of the studied species: α-pinene, germacrene D, and caryophyllene oxide (Table 2). α-Pinene (13.4±0.6 %) was the main component of the oil from *S. integrifolium* inflorescences, and caryophyllene oxide (19.0±2.2 %) along with germacrene D (13.1±4.4 %) dominated in the oil from *S. trifoliatum*. Earlier research [3] showed that α-pinene (to 20.9 %) and caryophyllene oxide (to 13.4 %) were the dominating constituents of oil from *S. perfoliatum* inflorescences. Oil from *S. integrifolium* was characterized by quite a high percentage of camphene (5.3±0.4 %), limonene (3.4±0.5 %) with the presence of bornyl acetate (6.5±0.5 %), and allo-aromadendr-9-ene (5.6±0.5 %) that were absent in essential oil made from the other taxon studied. β-Caryophyllene (4.4±0.9 %) and silphiperfol-6-en-5-one (7.3±0.7 %) were worth mentioning in oil from *S. trifoliatum*. Besides volatile compounds in the studied lipophilic extracts, the following sterols were present in large amounts: γ-sitosterol and stigmasterol, as well as triterpene alcohols such as β-amyrine and α-amyrine. Furthermore, higher alkanes, free fatty acids, their derivatives, and vitamin E were identified in ether extracts.

It can be supposed that some of the identified components of the studied extracts and essential oils from inflorescences are produced by *Silphium* plants as pollinating insects. Earlier research [8, 9] revealed that verbenone, carveol, bornyl acetate, or (*Z,Z,Z*)-9,12,15-octadecatrienoic acid methyl ester, which were also identified in the present study, attracted honey-bee *Apis mellifera* abundantly visiting flowering plants of the studied species.

The performed study revealed similarity of leaf anatomical structure and, to some extent, essential oils and ether extracts from these organs of *S. integrifolium* and *S. trifoliatum* to the earlier described *S. perfoliatum*. However, among the tested oils and extracts, significant amounts of bornyl acetale and allo-aromadendr-9-ene were found only in *S. integrifolium* rhizomes, which were absent in previously examined *S. perfoliatum*.

TABLE 1. Percentage of Essential Oil in Inflorescences and Petroleum Ether Extract of *S. integrifolium* and *S. trifoliatum*

Species	1999		2000		2001		Mean	
	A	B	A	B	A	B	A	B
S. integrifolium	0.31	3.81	0.31	4.02	0.35	4.28	0.32	4.04
S. trifoliatum	0.27	3.85	0.30	4.42	0.37	4.56	0.31	4.28

A: Percentage of the essential oil (% v/v DM); B: Content of *Silphium* petroleum ether extract (% DM).

R. Kowalski Department of Analysis and Evaluation of Food Quality, Central Apparatus Laboratory, University of Life Sciences in Lublin, 13 Akademicka St., 20-950 Lublin, Poland, fax +48 81 533 37 52, e-mail: radoslaw.kowalski@ar.lublin.pl.
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TABLE 2. Percentage Composition of the Essential Oils (EO) and Lipophilic Extracts (EX) of *S. integrifolium* and *S. trifoliatum* Inflorescences (1999-2001)

Compound	RI	Percentage				Reference
		S. integrifolium		*S. trifoliatum*		
		EO	EX	EO	EX	
Tricyclene	919	0.7±0.0	0.1±0.0	0.4±0.0	0.2±0.0	4
α-Pinene	931	13.4±0.6	3.0±0.8	8.0±0.8	3.2±1.0	4
Camphene	946	5.3±0.4	1.0±0.1	2.7±0.3	1.0±0.2	4
Sabinene	972	0.4±0.0	Tr.[b]	0.1±0.0	Tr.	4
β-Pinene	975	2.1±0.0	0.4±0.0	1.0±0.1	0.3±0.1	4
α-Terpinene+1,4-Cineole	1016	Tr.	-[c]	-	-	4
p-Cymene	1024	0.2±0.1	Tr.	0.1±0.0	Tr.	4
Limonene	1029	3.4±0.5	0.5±0.0	0.2±0.1	Tr.	4
1,8-Cineole	1035	Tr.	Tr.	Tr.	Tr.	4
p-Cymenene	1091	0.2±0.1	Tr.	-	-	4
α-Pinene oxide	1100	-	Tr.	Tr.	0.2±0.0	4
α-Campholenal	1130	1.3±0.1	Tr.	0.5±0.1	0.1±0.0	4
cis-Verbenol	1142	0.8±0.1	Tr.	0.4±0.1	Tr.	4
trans-Pinocarveol	1143	1.4±0.1	0.1±0.0	0.5±0.1	0.1±0.0	4
trans-Verbenol	1150	5.2±1.1	0.5±0.0	2.0±0.1	0.3±0.0	4
Pinocarvone	1168	1.0±0.1	Tr.	0.5±0.0	Tr.	4
Borneol	1170	0.8±0.1	Tr.	-	-	4
p-Mentha-1,5-dien-8-ol	1171	1.1±0.0	Tr.	0.5±0.2	Tr.	4
p-Cymen-8-ol	1193	Tr.	-	Tr.	-	4
Myrtenal + Myrtenol	1202	1.9±0.2	0.3±0.0	0.8±0.2	0.2±0.0	4
Verbenone	1217	2.0±0.3	0.5±0.0	0.7±0.0	0.2±0.1	4
trans-Carveol	1223	0.7±0.1	Tr.	0.1±0.0	Tr.	4
Carvone	1251	0.5±0.1	Tr.	-	-	4
Bornyl acetate	1290	6.5±0.5	1.4±0.1	-	-	4
7-α-H-Silphiperfol-5-ene	1328	-	-	Tr.	Tr.	4, 5
7-β-H-Silphiperfol-5-ene	1344	-	-	Tr.	Tr.	4, 5
α-Ylangene	1375	-	-	-	0.1±0.0	4
α-Copaene	1380	0.2±0.0	0.1±0.0	0.3±0.0	0.6±0.0	4, 5
Silphiperfol-6-ene	1381	Tr.	Tr.	-	-	4, 5
Modhephene	1385	Tr.	Tr.	-	-	2, 4
β-Bourbonene	1389	Tr.	Tr.	0.1±0.0	Tr.	4, 5
β-Elemene	1396	0.2±0.0	Tr.	0.2±0.0	Tr.	2, 4
β-Caryophyllene	1425	1.0±0.1	0.7±0.2	4.4±0.9	6.2±0.2	2, 4, 5
trans-α-Bergamotene	1439	0.4±0.1	0.3±0.0	0.4±0.0	0.2±0.0	4, 5
α-Humulene	1460	0.5±0.0	0.3±0.0	2.3±0.3	1.5±0.8	2, 4
Germacrene D	1488	3.8±0.3	3.1±1.3	13.1±4.4	17.4±5.5	4, 5
allo-Aromadendr-9-ene	1490	5.6±0.5	2.6±0.7	-	-	5
(*E*)-β-Ionone	1491	0.3±0.1	Tr.	-	-	4
Germacrene A	1499	-	-	-	0.2±0.1	4, 5
β-Bisabolene	1513	0.4±0.1	Tr.	-	-	4, 5
γ-Cadinene	1517	0.2±0.1	0.1±0.0	0.2±0.0	Tr.	4, 5
δ-Cadinene	1530	0.2±0.1	0.1±0.0	0.6±0.1	0.5±0.1	4, 5
α-Calacorene	1548	0.2±0.0	Tr.	0.1±0.0	Tr.	4
N.i.[a]	1562	0.6±0.1	Tr.	1.3±0.4	Tr.	3, 6
(*E*)-Nerolidol	1568	0.9±0.2	0.1±0.0	0.9±0.0	0.2±0.0	4
N.i.	1576	0.8±0.1	0.1±0.0	0.6±0.1	0.2±0.0	3, 6

TABLE 2 (continued)

Compound	RI	Percentage				Reference
		S. integrifolium		*S. trifoliatum*		
		EO	EX	EO	EX	
4-β-Hydroxygermacra-1(10),5-diene	1584	Tr.	0.1±0.0	Tr.	0.1±0.0	3
Spathulenol	1588	1.3±0.0	0.3±0.0	3.5±0.4	1.0±0.1	4
Caryophyllene oxide	1593	5.0±0.2	1.8±0.7	19.0±2.2	7.0±1.1	4
N.i.	1596	0.5±0.0	0.2±0.0	0.5±0.0	0.8±0.2	3, 6
Salvial-4(14)-en-1-one	1604	0.6±0.0	Tr.	1.1±0.0	0.4±0.0	3, 4
Silphiperfol-6-en-5-one	1629	2.8±0.1	0.8±0.0	7.3±0.7	2.4±0.4	4
N.i.[a]	1640	-	-	0.5±0.1	Tr.	2, 3
τ-Cadinol	1646	1.9±0.5	0.7±0.2	0.1±0.0	Tr.	4
α-Cadinol	1661	1.2±0.2	Tr.	2.5±0.0	Tr.	4
N.i.	1664	5.2±0.0	Tr.	0.4±0.1	Tr.	6
N.i.	1693	-	-	2.1±0.3	Tr.	3, 6
Oplopanone	1752	Tr.	0.4±0.0	Tr.	0.6±0.1	4
N.i.	1757	0.7±0.3	Tr.	1.2±0.1	0.5±0.3	
β-Acoradienol	1769	-	-	1.7±0.2	Tr.	4
N.i.	1782	0.4±0.2	Tr.	0.8±0.1	Tr.	3, 6
6,10,14-Trimethyl-2-pentadecanone	1847	-	0.4±0.0	-	0.4±0.1	7
N.i.	1854	0.4±0.0	0.4±0.0	1.0±0.1	0.9±0.4	3, 6
Hexadecanoic acid	1970	-	0.4±0.2	-	2.9±1.1	7
N.i. JEOR	2113	5.2±0.7	Tr.	-	-	6
Phytol	2116	-	1.0±0.0	-	0.6±0.0	2, 4, 7
(*Z,Z,Z*)-9,12,15-octadecatrienoic acid methyl ester	2156	-	0.2±0.0	-	0.6±0.2	7
Pentacosane	2500	-	3.4±0.3	-	1.2±0.2	7
Docosanoic acid methyl ester	2531	-	0.9±0.0	-	0.2±0.0	7
Hexacosane	2600	-	Tr.	-	0.8±0.2	
N.i.	2653	-	8.5±0.7	-	2.6±0.2	7
Heptacosane	2700	-	12.9±1.1	-	6.2±1.1	7
Tetracosanoic acid methyl ester	2737	-	31.±0.1	-	2.2±0.2	2
N.i.	2789	-	1.2±0.6	-	0.4±0.1	7
Octacosane	2800	-	0.9±0.0	-	0.6±0.2	7
Squalene	2838	-	2.3±0.0	-	0.6±0.3	2
Nonacosane	2900	-	4.3±0.3	-	2.3±1.0	7
N.i.	2909	-	0.9±0.4	-	0.7±0.3	5, 7
Hexacosanoic acid methyl ester	2940	-	0.5±0.2	-	0.9±0.1	7
Triacontane	3000	-	0.1±0.0	-	0.2±0.0	2
N.i.	3052	-	0.7±0.3	-	0.3±0.1	7
β-Tocopherol	3076	-	0.4±0.2	-	0.2±0.0	7
Hentriacontane	3100	-	0.7±0.3	-	0.3±0.1	2
Octacosanoic acid methyl ester	N.d.	-	Tr.	-	0.3±0.0	7
Vitamin E	N.d.	-	3.2±0.1	-	1.6±0.4	7
N.i.	N.d.	-	3.5±2.2	-	2.0±0.8	7
Stigmasterol	N.d.	-	3.9±0.3	-	2.1±0.3	
γ-Sitosterol	N.d.	-	0.9±0.2	-	1.5±0.3	2, 7
β-Amyrine	N.d.	-	4.4±0.2	-	5.4±1.3	2, 7
α-Amyrine	N.d.	-	6.5±0.2	-	6.8±1.6	2, 7
N.i.	N.d.	-	0.8±0.4	-	2.7±1.2	
N.i.	N.d.	-	0.9±0.5	-	2.3±1.3	

[a]N.i. - not identified; [b]Tr.: trace (<0.1%); [c]N.d. - not determined.

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