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RESEARCH ARTICLE

Chemical constituents and antimicrobial activities of the essential oils from *Sedum pallidum* var. *bithynicum* and *S. spurium* grown in Turkey

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Abstract

Chemical compositions of the essential oil of *Sedum pallidum* Bieb. var. *bithynicum* (Boiss.) and *S. spurium* Bieb. (Crassulaceae) from Turkey were investigated by GC-MS, and antimicrobial activity of the oil samples were assessed against Gram-positive/negative bacteria and yeast-like fungi. Thirty-eight and thirty-five components were identified in the essential oils and the main components of these species were found to be caryophyllene oxide from *S. pallidum* var. *bithynicum* and hexahydrofarnesyl acetone from *S. spurium* in the ratios of 12.8% and 15.7%, respectively. The isolated essential oils of the plants showed low antimicrobial activity against Gram-negative/positive bacteria and yeast-like fungi, having the MIC values of 500-2000 µg/mL. Antibacterial activity was not observed against *Bacillus cereus*.

Keywords: Sedum pallidum *var.* Bithynicum*;* Sedum spurium*; Crassulaceae; essential oil; antimicrobial activity; GC-MS*

Introduction

The genus *Sedum* L. (Crassulaceae) is represented by 43 species in Turkey (Chamberlain, 1972; Hart & Alpınar, 2000). Several species such as *S. acre* L., *S. album* L., *S. telephium* L., and *S. pallidum* are used in Anatolian folk medicine for wounds, hemorrhoids, constipation, food fungi, and as laxative and diuretic (Baytop, 1999; Zeybek & Zeybek, 1994; Karahan et al., 2006). *Sedum pallidum* Bieb. var. *bithynicum* (Boiss.) is distributed mainly in north-west and rarely in central Anatolia and *Sedum spurium* Bieb. grows in north-east Anatolia (Chamberlain, 1972; Hart & Alpınar, 2000).

Previous phytochemical studies in the genus *Sedum* (*S. spurium*, *S. pallidum*) have shown the presence of different natural compounds including arbutin, hydroquinone (Stanislaw et al., 1984), flavonoids (Bandyukova & Shinkarenko, 1965; Stevens et al., 1996), alkaloids (Franck, 1958; Gill et al., 1979; Franck & Hartmann, 1963), free sugars (Nordal & Klevstrand, 1951), and sedoheptose (Nordal, 1940). Volatile constituent studies on some *Sedum* species have been mentioned in the literature (Stevens et al., 1994a, 1994b; Mesicek & Perpar, 1973). However, the volatile composition and antimicrobial activity of the essential oils of *S. pallidum* var. *bithynicum* and *S. spurium* have not been studied. Thus, current study deals with GC-MS analysis and antibacterial and antifungal activity determinations of the essential oils from *S. pallidum* var. *bithynicum* and *S. spurium*.

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Materials and methods

Plant material

S. pallidum var. *bithynicum* and *S. spurium* were collected in Cimil Başköy, Rize-Turkey (A8), alpine meadows at heights of ~2000 m and 1900 m in the north-eastern part of Turkey, in July 2004, respectively. The plants were authenticated by K. Coşkunçelebi. Voucher specimens (Coşkunçelebi 488-2004 and 487-2004, KTUB) were deposited in the herbarium of the Department of Biology, Karadeniz Technical University, Turkey. Plant materials were air-dried at room temperature for later analysis.

Isolation of the essential oils

The air-dried whole plants (~40 g, each) of *S. pallidum* and *S. spurium* were hydrodistilled in a Clevenger-type apparatus using a cooling bath (-15°C) system (3 h). The obtained oils were extracted in HPLC grade *n*-hexane (0.5 mL) and kept at 4°C in a sealed brown vial. The percentage yields of the oils from *S. pallidum* var. *bithynicum* and *S. spurium* calculated on a moisture-free basis were 0.15% and 0.12% (v/w), respectively.

Identification of components by GC-MS

GC-MS analyses were described previously (Güleç et al., 2007; Küçük et al., 2006; Yaylı et al., 2005). Retention indices of all the components were determined by Kovats method (Miller & Bruno, 2003) using *n*-alkanes (C_6-C_{32}) as standards. Identification of individual components was made by comparison of their retention times with those of available analytical standards (α-pinene, camphene, linalool, geraniol, *n*-tetradecane, *n*-pentadecane, *n*-heptadecane, and *n*-octadecane), and by computer searching, matching mass spectral data with those held in the National Institute of Standards and Technology (NIST) and Wiley libraries of mass spectra and literature comparison (Adams, 2004; Javidnia et al., 2005; Jovanovic et al., 2004; Güleç et al., 2007; Küçük et al., 2006; Skaltsa et al., 2001, 2003; Yaylı et al., 2005; Couladis et al., 2002).

Antimicrobial activity assessment

All test microorganisms were obtained from the Refik Saydam Hifzissihha Institute (Ankara, Turkey) and were as follows: Gram-negative bacteria (*Escherichia coli* ATCC 25922, *Yersinia pseudotuberculosis* ATCC 911, *Klebsiella pneumoniae* ATCC 13883, *Pseudomonas aeruginosa* ATCC 10145), Gram-positive bacteria (*Enterococcus faecalis* ATCC 29212, *Staphylococcus aureus* ATCC 25923, *Bacillus cereus* 709 ROMA), and yeast-like fungus (*Candida albicans* ATCC 60193, *Candida tropicalis* ATCC 13803).

Agar dilution MIC assay

Using a modification of the assay described in the literature [National Committee for Clinical Laboratory Standards (NCCLS), 2008; Mann & Markham, 1998; Southwell et al., 1993], essential oil solution was added to molten Mueller Hinton Agar (MHA) and Potato Dextrose Agar (PDA)/Tween 20 medium at 48°C, to give concentrations ranging from 250 to 6000 µg/mL. The antibacterial and antifungal assays were performed in Mueller-Hinton buffer (MHB) (Difco, Detroit, MI) at pH.7.3 containing 1% agar and buffered yeast nitrogen base (Difco) at pH 7.0, with 1% agar, respectively. Plates prepared in triplicate, were spot-inoculated with 3 µL aliquots of culture in MHB adjusted to yield a density within McFarland 0.5 turbidity. Plates were incubated at 37°C for 18 h and the minimal inhibition concentration (MIC) was determined as the lowest concentration of oil to result in no growth of the inoculum on two of three plates. The samples were dissolved in chloroform to prepare stock solutions. Chloroform and Tween 20 were used as control and the results are shown in Table 1. Ampicillin and fluconazole were used as standard antibacterial and antifungal drugs, respectively. All test microorganism showed activity with MIC values between < 32, 1-32 µg/mL against standard drug.

Table 1. Screening results of antimicrobial activities of the essential oils from *S. pallidum* var. *bithynicum* and *S. spurium*.

	MIC (µg/mL)			
Microorganisms	*S. pallidum* var. *bithynicum*	*S. spurium*	Ampicillin	Fluconazole
Escherichia coli	1000	500	8	
Yersinia pseudotuberculosis	2000	1000	32	
Klebsiella pneumoniae	2000	1000	32	
Pseudomonas aeruginosa	2000	1000	32	
Enterococcus faecalis	1000	500	2	
Staphylococcus aureus	1000	500	2	
Bacillus cereus	> 6000	> 3000	< 1	
Candida albicans	2000	1000		< 1
Candida tropicalis	2000	1000		8

Results and discussion

In total, 38 and 35 components, representing about 94.3% and 96.9% of the oil samples of *S. pallidum* var. *bithynicum* and *S. spurium*, respectively, were identified. The general chemical profile of the essential oils, the percentage content, and retention indices of the constituents are summarized in Table 2. The major compounds of the essential oil of *S. pallidum* var. *bithynicum* were caryophyllene oxide (12.8%), *n*-nonanal (9.4%), α-bisabolol (6.8%), β-sesquiphellandrene (4.5%), and β-bisabolene (3.7%). On the other hand, the major constituents of the essential oil of *S. spurium* were hexahydrofarnesyl acetone (15.7%), (*Z*)-phytol (10.2%), δ-cadinene (4.7%), *allo*-aromadendrene (3.8%), and geranyl acetone (3.6%).

The chemical class distributions of the essential oil components of the plants are listed at the bottom of Table 2. The compounds were separated into six classes, which were aliphatic monoterpenes, oxygenated monoterpenoids, sesquiterpenes, sesquiterpenoids, diterpenoids, and others. Sesquiterpenoid components were the main constituents of both oils in the ratios of 30.7% and 27.7%, and 19 compounds were common with the ratio of 39.8% and 55.3% in *S. pallidum* var. *bithynicum* and *S. spurium,* respectively. However, the general chemical profile of the essential oils of *S. pallidum* var. *bithynicum* and *S. spurium* showed some differences, which can be explained by the environmental factors, the subspecies, and the parts of the plant used.

Previous GC and GC-MS studies on the waxes of *Sedum* species showed the presence of alkanes, alcohols, aldehydes, fatty acids, fatty acid methyl esters, wax esters, triterpenes, and triterpenoids (Stevens et al., 1994a, 1994b). However, there has been no report regarding direct GC-MS analysis of essential oils of *Sedum* species, except the oils obtained by hydrolysis at pH 4 from *S. maximum* (Mesicek & Perpar, 1973). In our case we similarly identified alkanes, alcohols, and aldehydes in the essential oil of *S. pallidum* var. *bithynicum* and *S. spurium*.

The antimicrobial activities of the essential oils of *S. pallidum* var. *bithynicum* and *S. spurium* were tested by the agar dilution method (NCCLS, 2008; Mann & Markham, 1998; Southwell et al., 1993) against seven bacteria and two yeast-like fungi**.** The species were chosen as representatives of three major groups of microorganisms: Gram-positive bacteria, Gram-negative bacteria, and yeast-like fungi. The results are presented in Table 1. Both samples showed low antimicrobial activity against Gram-positive and negative bacteria and yeast-like fungi, having the MIC values of 500-2000 µg/mL. None of the oils showed any activity against *B. cereus* 709 ROMA at 1000-6000 µg/mL. Previous antimicrobial and antioxidant studies of

Table 2. Identified components in the essential oils of *S. pallidum* var. *bithynicum* and *S. spurium*.

No.	Lit. RI	Exp. RI	Compound	A[a] % Area	B[a] % Area
1	939	940	α-Pinene[b]	1.4	1.8
2	954	952	Camphene[b]	1.8	2.7
3	998	998	2-Pentylfuran	0.5	-
4	1042	1044	Benzene acetaldehyde	-	0.5
5	1068	1066	*n*-Octanol	0.7	0.3
6	1097	1096	Linalool[b]	-	3.1
7	1101	1103	*n*-Nonanal	9.4	3.0
8	1134	1136	Terpineol	-	0.7
9	1169	1171	1-Nonanol	0.3	-
10	1189	1186	α-Terpinol	-	2.2
11	1196	1193	Mrytenal	1.4	1.2
12	1196	1197	Myrtenol	1.2	-
13	1202	1203	*n*-Decanal	1.3	1
14	1253	1254	Geraniol[b]	-	0.4
15	1270	1271	Decanol	1.4	-
16	1280	1281	Nopol	-	0.4
17	1290	1292	Thymol	0.4	-
18	1299	1299	Carvacrol	0.3	-
19	1307	1305	Undecanal	0.2	-
20	1317	1315	(2*E*,4*E*)-Decadienal	1.9	2.9
21	1375	1373	α-Ylangene	-	1.1
22	1377	1376	α-Copaene	1.9	-
23	1400	1400	*n*-Tetradecane[b]	-	0.7
24	1408	1406	Longifolene	0.4	0.8
25	1419	1418	β-Caryophyllene	1.9	-
26	1441	1442	Aromadendrene	3.5	0.7
27	1455	1453	Geranyl acetone	0.8	3.6
28	1457	1456	*trans*-β-Farnesene	-	2.3
29	1460	1460	*allo*-Aromadendrene	-	3.8
31	1481	1482	α-Curcumene	1.7	-
30	1485	1483	Germacrene D	0.9	1.9
32	1489	1487	*trans*-β-Ionone	1.2	-
33	1500	1500	*n*-Pentadecane[b]	-	0.6
34	1505	1504	Cuparene	0.4	
35	1507	1508	β-Bisabolene	3.7	-
36	1523	1523	β-Sesquiphellandrene	4.5	2.3
37	1523	1525	δ-Cadinene	-	4.7
38	1569	1567	Ledol	0.7	0.7
39	1583	1582	Caryophyllene oxide	12.8	-
40	1585	1585	Globulol	1.8	0.7
41	1613	1610	Tetradecanal	3.2	-
42	1647	1645	Cubenol	-	1.8
43	1651	1649	β-Eudesmol	-	0.9
44	1654	1655	α-Cadinol	-	1.1
45	1676	1675	Caryophyllenol-II	1.7	-
46	1686	1688	*a*-Bisabolol	6.8	-
47	1691	1690	*trans*-α-Bergamotol	0.9	1.6
48	1700	1700	*n*-Heptadecane[b]	1.2	1.3
49	1713	1711	Curcumen-15-al	1.7	-
50	1800	1800	*n*-Octadecane[b]	-	0.6
51	1845	1845	Hexahydrofarnesyl acetone	3.4	15.7

Table 2. continued on next page.

Table 2. Continued.

No.	Lit. RI	Exp. RI	Compound	A[a] % Area	B[a] % Area
52	1914	1916	Farnesyl acetone	0.9	2.9
53	2057	2056	Manool	2.3	-
54	2114	2113	(*Z*)-Phytol	2.9	10.2
			Total isolate	83.4	80.2
Aliphatic monoterpenes				3.2	4.5
Oxygenated monoterpenoids				5.3	11.6
Sesquiterpenes				18.9	15.3
Sesquiterpenoids				30.7	27.7
Diterpenoids				5.2	10.2
Others				20.1	10.9
The common compounds				39.8	55.3

RI, retention index; A, *Sedum pallidum* var. *bithynicum*; B, *Sedum spurium*.

[a]Compounds are listed in order of elution. RI values are calculated from retention times relative to those of *n*-alkanes (C_6-C_{32}) on the non-polar HP-5 column.

[b]Identified with authentic samples.

Sedum species have also showed low activity (Garcia et al., 2003; Mavi et al., 2004).

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