SHORT REPORT

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Volatiles and Antimicrobial Activity of the Essential Oils of the Mosses *Pseudoscleropodium purum*, *Eurhynchium striatum*, and *Eurhynchium angustirete* Grown in Turkey

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Abstract: The chemical composition of the essential oils from all parts of *Pseudoscleropodium purum*, *Eurhynchium striatum* and *Eurhynchium angustirete* were analysed by GC-FID-MS. Sixty-five, thirty-four and seven compounds, accounting for 99.7%, 97.3% and 99.9% of the oils, were identified and the main components were α-pinene (16.1%), 3-octanone (48.1%), and eicosane (28.6%), respectively. The essential oils were also tested against nine strains using a broth microdilution method and showed moderate antimicrobial activity with minimum inhibitory concentrations (MIC) ranging from 278.2 to 2225 µg/mL. All the mosses essential oils showed good antituberculosis activity against *Mycobacterium smegmatis* with MIC of 278.2-312.0 µg/mL.

Keywords: *Pseudoscleropodium purum*; *Eurhynchium striatum*; *Eurhynchium angustirete;* essential oil; antimicrobial and antituberculosis activity; GC-FID-MS.

1. Plant Source

P. purum, *E. striatum* and *E. angustirete* were collected from Artvin- Turkey (at heights of 1044 m, 1900 m and 456 m, respectively) in August and June, 2011, respectively. The mosses were authenticated immediately after collection. Voucher specimens were deposited in the Herbarium of the Department of Biology (Özdemir & Batan 1510, 1515 and 1514, respectively), Faculty of Science, Karadeniz Technical University, Turkey.

2. Previous Studies

Bryophytes are known to be oldest land plants, which includes hornworts, liverworts, and mosses. More than 22,000 members of the mosses were present in the world [1-2]. The mosses of the genera *Pseudoscleropodium purum* (Hedw.) M. Fleisch., *Eurhynchium striatum* (Hedw.) Schimp., and *Eurhynchium angustirete* (Broth.) T. J. Kop. belong to the *Brachytheciaceae* family (Bryophytes). The

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genus of *Brachytheciaceae* is a member of Bryophytes family consisting of 570 species, making it one of the largest and youngest moss families [3-4]. *P. purum, E. striatum*, and *E. angustirete* were represented by 1, 2 and 2 taxa in Turkey, respectively. Bryophytes have been used as a traditional medicine in Europe, Chinese, and Indian medicine for the treatment of bronchitis, tonsillitis, in skin diseases and burns. They also possess interesting biological activities such as antibacterial, antitumor, antiseptic, anticoagulant, insect antifeedant, nerve protecting and cytotoxic [5-6]. Most of the phytochemical mosses articles mentioned terpenoids, phenolics, glycosides, fatty acids, as well as some rare aromatic compounds [7-11]. But, volatile investigations showed that mosses contain huge amounts of aldehydes, terpenes, and aliphatic and aromatic compounds [12-13]. Very few investigations on this genus have been reported that describe the biologically active constituents of bryophytes [14]. This work provides new information reported about chemotaxonomic aspects of bryophytes. In the present work, different bryophyte mosses were studied for the volatile constituents and antimicrobial activity of the essential oils of *P. purum, E. striatum*, and *E. angustirete*.

3. Present Study

In the present work, the mosses (*P. purum, E. striatum*, and *E. angustirete)* were collected at different locations in Artvin-Turkey. Before hydrodistillation, the plant materials were carefully inspected for contaminations. The chemical constituents of the essential oil of these three mosses species were identified by using GC-FID-MS analyses on the basis of their mass spectra, retention indices, literature data and the mass spectral libraries NIST98, Wiley7n and HPCH1607 [15-18]. The percentage of each component within the essential oils was determined on HP-5-MS column and is presented in Table 1.

A total of 65, 34, and 7 volatile compounds were identified in the essential oils of *P. purum, E. striatum*, and *E. angustirete*, respectively. The volatile components identified accounted for 97.3-99.9% of the oils composition. The analyzed oils can be classified in nine groups according to their chemical class distributions (Table 1).

P. purum oil contained high amounts of mono and sequiterpenes (28.2% and 26.9%), with *α*-pinene (16.1%) and *β*-longipinene (8.7%) being the most abundant and were dominant components in the terpene hydrocarbon fractions. The essential oil of *E. striatum* had a high content of the ketone (48.1%) and aldehydes (23.3%) with 3-octanone (48.1%) and nonanal (13.7%) being the major constituents. *E. angustirete* oil was characterized by high amounts of hydrocarbones (81.6%). The remaining components in essential oil of *E. angustirete* were monoterpenes (18.3%), and the main constituent was *α*-pinene (11.6%). The monoterpene hydrocarbons *α*-pinene, *β*-pinene and limonene are very common volatile compounds for the mosses studied.

Previous research indicated that volatiles of most mosses have been abundant in aliphatic and aromatic aldehydes (n-heptanal, n-octanal, n-nonanal, 2E,4E-decadienal, 2E,4Z-decadienal, n-teradecanal, benzaldehyde, and benzene acetaldehyde) and hydrocarbons (C_{12}-C_{18}, saturated, mono- and di-unsaturated) [19]. Very common constituents of the essential oils of moss were the monoterpene hydrocarbons (*α*- and *β*-pinene, camphene, *p*-cymene, myrcene, limonene, *α*-terpinene and camphor) [17, 19]. Based on the oil composition analysis of all three investigated mosses, we also observed the similar terpenes, aliphatic-aldehydes and hydrocarbons in the oils of mosses (Table 1). It is apparent from the data shown that the chemical components of the three mosses were different as in cases reported in the literature. The chemical composition differences of the mosses might be caused by the climatic conditions, ecological niches, and other biotic factors.

The present data could be helpful in selecting the future targets for phytochemical study as well as enriching our current chemical knowldege about *Bryophytes* species. Such kind of data could also be useful elucidation of the chemotaxonomical relationships among the sections of *Bryophytes* genus.
The work demonstrated that *α*-pinene, *β*-pinene and limonene of monoterpene class are characteristic and represent excellent chemotaxonomical markers for this family.

Table 1. Identified components in the essential oils of *P. purum* (A), *E. striatum* (B) and *E. angustirete*(C)[a,b].

Compounds	A Area[a] (%)	B Area[a] (%)	C Area[a] (%)	Ex. RI[b]	Lit. RI
Heptanal	8.1	1.1	-	900	902
Tricyclene	0.6	-	-	924	927
α-Thujene	0.1	-	-	929	930
α-Pinene[d]	16.1	1.4	11.6	937	939
Camphene[d]	1.4	0.2	-	951	954
Thuja-2,4(10)-diene	0.3	-	-	957	960
Benzaldehyde	-	0.4	-	958	960
β-Pinene[d]	3.5	2.0	3.1	977	979
3-Octanone	2.2	48.1	-	983	984
2-Amyl furan	0.8	2.8	-	988	991
Octanal	1.1	2.2	-	997	999
α-Phellandrene	2.3	1.0	-	1003	1003
α-Terpinene[d]	0.2	-	-	1015	1017
o-Cymene	0.4	-	-	1024	1025
Limonene[d]	1.0	0.7	3.6	1027	1029
Benzene acetaldehyde	0.6	1.4	-	1042	1042
γ-Terpinene[d]	1.4	0.5	-	1058	1060
p-Cymenene	0.8	-	-	1089	1091
Nonanal	4.7	13.7	-	1101	1101
α-Campholenal	0.4	-	-	1124	1126
E-Pinocarveol	0.6	-	-	1137	1139
2*E*-Nonenal	-	0.9	-	1160	1162
Pinocarvone	1.0	-	-	1163	1165
Terpinen-4-ol	0.6	-	-	1176	1177
Myrtenal	0.8	-	-	1194	1196
Decanal	0.7	0.8	-	1200	1202
2*E*-Decanal	0.4	0.3	-	1263	1264
2*E*,4*Z*-Decadienal	0.3	-	-	1291	1293
Undecanal	0.2	0.8	-	1305	1307
2*E*,4*E*-Decadienal	0.8	0.6	-	1315	1317
α-Cubebene	0.4	-	-	1349	1351
α-Copaene	0.4	-	-	1375	1377
β-Bourbonene	0.2	-	-	1387	1388
β-Elemene	0.2	-	-	1389	1391
Tetradecane	-	0.2	-	1397	1400
β-Longipinene	8.7	-	-	1399	1401
E-Caryophyllene	-	0.3	-	1417	1419
β-Copaene	0.2	-	-	1430	1432
Aromadendrene	0.8	-	-	1440	1441
α-Humulene	1.8	-	-	1453	1455
Aromadendrane-dehydro	0.9	-	-	1460	1463
γ-Muurolene	2.1	-	-	1479	1480
α-Amorphene	-	0.2	-	1483	1485
β–Selinene	1.2	-	-	1488	1490
E-Muurola-4(14)-5-diene	-	0.4	-	1492	1494
Viridiflorene	1.1	-	-	1495	1497
α-Muurolene	1.2	-	-	1498	1500
δ-Amorphene	-	0.4	-	1509	1512
γ-Cadinene	2.8	-	-	1512	1514
δ-Cadinene	3.9	-	-	1521	1523
E-Cadina-1(2),4-diene	0.1	-	-	1532	1535
α-Cadinene	0.2	-	-	1538	1539
α-Calacorene	0.5	-	-	1544	1546

Caryophyllene oxide	0.3	0.4	-	1581	1583	
Tetradecanal	-	1.1	-	1612	1613	
Alloaromadendrene-epoxy allo-	5.4	-	-	1640	1641	
α-Selina-3,11-dien-6-ol	0.7	-	-	1642	1644	
Cubenol	0.7	-	-	1646	1647	
E-Calamenen-10-ol	1.2	-	-	1667	1669	
Tetradecanol	-	5.7	-	1670	1673	
Cadalene	0.2	-	-	1675	1677	
Pentadecanol	0.7	-	-	1772	1774	
Hexahydrofarnesyl acetone	2.4	1.8	-	1845	1847	
Hexadecanol	-	0.8	-	1875	1876	
Cyclohexadecanolide	1.1	-	-	1932	1935	
Eicosane[d]	1.9	2.8	28.6	2000	2000	
Abietatrene	0.8	-	-	2050	2057	
Abietadiene	0.6	-	-	2085	2088	
Heneicosane[d]	-	1.1	-	2100	2100	
Docosane[d]	0.4	0.4	-	2200	2200	
Sandaracopimarinol	0.2	-	-	2267	2270	
Tricosane[d]	1.0	1.5	17.2	2300	2300	
Tetracosane[d]	0.8	0.6	19.8	2400	2400	
Pentacosane[d]	1.7	1.5	16.0	2500	2500	
				N.C		
				A	B	C
Monoterpenes	28.1	5.8	18.3	12	6	3
Monoterpenoids	5.2	-	-	5	-	-
Sesquiterpenes	26.9	1.3	-	19	4	-
Sesquiterpenoids	8.3	0.4	-	5	1	-
Diterpene	0.6	-	-	1	-	-
Diterpenoids	1.0	-	-	2	-	-
Terpenoids related	3.7	1.8	-	3	1	-
Hydrocarbons	5.8	8.1	81.6	5	7	4
Aldehydes/ketones	17.8	23.3/48.1	-	9	12	-
Others	2.3	8.5	-	4	6	-
Total identifications	99.7	97.3	99.9	65	37	7

[a]RI calculated from retention times relative to that of *n*-alkanes(C_6-C_{32}) on the non-polar HP-5 column. [b]Percentages obtained by FID peak-area normalization. [c]Identified by authentic samples. [d]NC: Number of compounds.

The antimicrobial activities of the isolated essential oils of *P. purum, E. striatum*, and *E. angustirete* were also tested *in vitro* against Gram-positive, Gram-negative, and acido-resistant bacteria and fungal microorganisms. The antimicrobial activity with the essential oil of *P. purum, E. striatum*, and *E. angustirete* were observed against the bacteria *Y. pseudotuberculosis*, *P. aeruginosa*, *S. aureus, E. faecalis, B. cereus, M. smegmatis,* and the fungi *C. albicans* and *S. cerevisiae*. The minumum inhibitory concentrations (MIC) of *P. purum, E. striatum* and *E. angustirete* essential oils for microorganisms tested are shown in Table 2. Only the Gram-positive bacteria, acido-resistant mycobacterium and fungi were sensitive to the oils, while no activity was observed against Gram-negative bacteria *E. coli*, *Y. pseudotuberculosis*, and *P. aeruginosa*. The minimal inhibition concentration (MIC) values for bacterial strains for the essential oils *P. purum*, *E. striatum* and *E. angustirete* were in the range of 278.2-2225 μg/mL.

The secondary metabolites from mosses identified so far are: terpenoids, flavonoids, and bibenzyls, and derivatives of fatty acids, acetophenols, arylbenzofurans [20]. Mosses are a rich source of secondary metabolites with antimicrobial activity [21]. Analyzing the volatile oil results, this essential oils included of terpenoids. Maybe, the essential oils antimicrobial activity against Gram-positive bacteria, acido-resistant mycobacterium and fungi caused by this compounds.

Finally, analyses of the volatile constituents from essential oils of *P. purum*, *E. striatum*, and *E. angustirete* collected from different locations in Artvin-Turkey indicate α-pinene (16.1%), 3-octanone (48.1%), and eicosane (28.6%) to be the predominant components, and the qualitative and quantitative determination of the essential oils showed that major constituents were monoterpenes (28.1%), a ketone

(48.1%) and hydrocarbons (81.6%), respectively. These results indicate that chemical constituents in the volatile oils showed variation in their chemical composition. The antimicrobial test results showed moderate antimicrobial activity against Gram-positive bacteria and *M. smegmatis*. This is the first report on the analysis of volatile constituents and antimicrobial activities of the essential oils from *P. purum, E. striatum,* and *E. angustirete* in Turkey.

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Supporting Information

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

References

[1] M. D. Russell (2010). Antibiotic Activity of Extracts from some Bryhophytes in South Western British Columbia, Research papers.

[2] R. G. Bodade, P. S. Borkar, A. Saiful and C. N. Khobragade (2008). *In vitro* Screening of Bryophytes for Amtimicrobial Activity, *J. Med. Plants.* **7,** 23-28.

[3] M. R. Crosby, R. E. Magill, B. Allen and S. He (1999). A Checklist of the Mosses. Missouri Botanical Garden, St. Louse.

[4] S. Huttunen, M. S. Ignatov, K. Muller and D. Quandt (2004). Phylogeny and evolution of epiphytism in the three moss families *Meteoriaceae, Brachytheciaceae* and *Lembophyllaceae*. Monogr. Syst. Bot.

[5] L. Li and J. Zhao (2009). Determination of the Volatile Composition of *Rhodobryum giganteum* (Schwaegr.) Par. (Bryaceae) using Solid-Phase Microextraction and Gas Chromatography/ Mass Spectrometry (GC/ MS), *Molecules.* **14**, 2195-2201.

[6] F. F. Boyom, V. Ngouana, P. H. Amvam Zollo, C. Menut, J. M. Bessiere, J. Gut and P. J. Rosenthal (2003). Composition and anti-plasmodial activities of essential oils from some Cameroonian medicinal plants, *Phytochem.* **64**, 1269-1275.

[7] N. Jockovic, P. B. Andrade, P. Valentão and M. Sabovljevic (2008). HPLC-DAD of phenolics in bryophytes *Lunularia cruciata*, *Brachytheciastrum velutinum* and *Kindbergia praelonga*, *J. Serb. Chem. Soc*. **73**, 1161-1167.

[8] A. Sabovljevic, M. Sabovljevic and N. Jockovic (2009). *In vitro* culture and secondary metabolite ısolation in Bryophytes. In: Mohan Jain S and Saxena PK (eds) Methods in Molecular Biology: Protocols for *in vitro* cultures and secondary metabolite analysis of aromatic and medicinal plants, Humana Press. **547**, 117-128.

[9] A. Sabovljević, M. Sokovic, J. Glamočlija, A. Ćirić, M. Vujičić, B. Pejin and M. Sabovljević (2010). Comparison of extract bio-activities of *in-situ* and *in-vitro* grown selected bryophyte species, *Afr. J. Microbiol. Res*. **4**, 808-812.

[10] H. D. Zinsmeister and R. Mues (1987). Moose as reservoir remarkable sekundäerer. Ingredients GIT *Mag. Lab.* **31**, 499-512.

[11] H. D. Zinsmeister, H. Becker and T. Eicher (1991). Bryophytes, a source of biologically active, naturally occurring material, *Angew. Chem. Int. Ed. Engl*. **30**, 130-147.

[12] O. Üçüncü, T. B. Cansu, T. Özdemir, Ş. Alpay Karaoğlu and N. Yaylı (2010). Chemical composition and antimicrobial activity of the essential oils of mosses (*Tortula muralis* Hedw., *Homalothecium lutescens* (Hedw.) H. Rob., *Hypnum cupressiforme* Hedw., and *Pohlia nutans* (Hedw.) Lindb.) from Turkey, *Turk. J. Chem*. **34**, 825-834.

[13] Y. Sarıtaş, M. M. Sonwa, H. Iznaguen, W. A. König, H. Muhle and R. Mues (2001). Volatile constituents in mosses (Musci), *Phytochem.* **57**, 443-457.

[14] F. Savaroğlu, S. İlhan and C. Filik-Iscen (2011). An evaluation of the antimicrobial activity of some Turkish mosses, *J. Med. Plants Res*. **5**, 3286-3292.

[15] T. Özdemir, N. Yaylı, T. B. Cansu, C. Volga and N. Yaylı (2009). Essential Oils in mosses (*Brachythecium salebrosum, Eurhynchium pulchellum*, and *Plagiomnium undulatum*) Grown in Turkey, *Asian J. of Chem.* **21**, 5505-5509.

[16] A. C. Goren, F. Piozzi, E. Akcicek, T. Kılıç, S. Çarıkçı, E. Mozioğlu and W. N. Setzer (2011). Essential oil composition of twenty-two *Stachys* species (mountain tea) and their biological activities, *Phytochem. Lett.* **4**, 448-453.

[17] O. Üçüncü, T. B. Cansu, T. Özdemir, Ş. Alpay Karaoğlu and N. Yaylı (2010). Chemical composition and antimicrobial activity of the essential oils of mosses (*Tortula muralis* Hedw., *Homalothecium lutescens* (Hedw.) H. Rob., *Hypnum cupressiforme* Hedw., and *Pohlia nutans* (Hedw.) Lindb.) from Turkey, *Turk. J. Chem*. **34**, 825-834.

[18] G. Tosun, N. Kahriman, C. Güleç Albay, Ş. Alpay Karaoğlu and N. Yaylı (2011). Antimicrobial activity and volatile constituents of the flower, leaf, and steam of *Paeonia daurica* grown in Turkey, *Turk. J. Chem.* **35**, 145-153.

[19] T. B. Cansu, O. Üçüncü, N. Kahriman, T. Özdemir and N. Yaylı (2010). Essential oil composition of *Grimmia trichophylla Grew.* and *G. decipiens (Shultz) Lindb*. grown in Turkey, *Asian J. of Chem*. **22**, 7280-7284.

[20] M. Veljic, A. Duric, M. Sokovic, A. Ciric, J. Glamoclija and P. D. Marin (2009). Antimicrobial activity of methanol extracts of fantinolis antipyretica, *Hypnum cupressiforme*, and *Ctenidium molluscum*, *Arch. Biol. Sci., Belgrade*. **61**, 225-229.

[21] P. O. Tedela, A. O. Adebiyi and A. Aremu (2014). *In vitro* antibacterial activity of two mosses: *Calymperes erosum* C. Mull and *Bryum coronatum* schwaegr from south-western Nigeria, *J. Biol. Li. Sci*. **5**, 65-76.