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Short Communication

Contact Toxicity and Chemical Composition of Essential Oil of *Acantholimon scorpius*

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ABSTRACT

Background: Plumbaginaceae plants family is a valuable natural insecticidal compound. This research focused on contact toxicity and chemical composition of essential oil obtained from *Acantholimon scorpus*.
Methods: The essential oil from the aerial parts of *A. scorpius* was extracted by hydrodistillation method and tested for their toxicity against *Oryzeaphilus mercator* (Coleoptera: Silvanidae) and *Tribolium castaneum* (Coleoptera: Tenerbrionidae). Chemical compounds of the essential oil was analyzed by the gas chromatography-mass spectrometry (GC-MS) and gas chromatography-flame ionization detection (GC-FID).
Results: The essential oil showed toxic effect on tested insects. This oil showed 90.0% mortality of *O. mercator* and 85.2% mortality of *T. castaneum* at a dose of 12 µl /l air after 48h of exposure. The constituents of this oil were identified, representing more than 82.9% of the total essential oil composition. Hexadecanoic acid, tetrahydrogeranyl acetone and oleic acid were the main compounds of the essential oil.
Conclusion: According to the result the essential oil of *A. scorpius* showed a noticeable insecticidal activity in contact toxicity model.

Introduction

Plumbaginaceae plants family is a valuable natural remedy in the several nations such as Chinese, Mediterranean and Iranian traditional medicines. Plants of this family have been used as effective treatment of many diseases.[1-6] Recent studies have indicated that various biological activities such as antiplasmodial and insecticidal properties.[7-12] In different researches, various active constituents was reported from the palnts of this genus like anthocyanins, flavonoids, tannins and quinines. [13-19] Some of these secondary metabolites such as quinines act as a part of plants defense mechanism against insects.[20-22] Because of insect's resistance against to many synthetic insecticide, development of the safe natural insecticides or repellents is an important need for insect borne transmitted diseases control and reduction. Insecticide properties of plant and secondary metabolites lead researchers to find novel new insecticidal compounds form plants.[23-27] Among plants metabolites, the essential oils have appropriate insecticidal potencies for using against stored product insects.[28] *Acantholimon* genus belongs to Plumbaginaceae family, consists of 83 species of woody sub shrub flowering plants commonly have spikes with pink to purple flowers and needle leaves, widely distributed in many different climatic regions of Iran and known as 'Prickly thrift' in Europe.[29] *A. scorpius* is an endemic plant of northwest and Zagros region of Iran. Although this genus shows high biodiversity among Plumbaginaceae family, there are no reports on chemical profile and insecticidal activity of this genus till now. Hence, this is the first report on the essential oil compositions and insecticidal activities of *Acantholimon* genus.

Materials and methods

Plant Material

Aerial parts of *A. scorpius* were collected at the flowering period from wild population growing in East Azarbaijan province, Iran. Voucher specimens were authenticated by the Pharmacognosy Department and voucher specimens, 2558, deposited in the herbarium of Department of Pharmacognosy, Faculty of Pharmacy, Guilan University of Medical Sciences, Rasht, Iran.

Extraction of the Essential Oil

The air-dried ground aerial parts of *A. scorpius* (500 gr) were subjected to hydrodistillation for 3 h using a Clevenger-type apparatus, yielding 1.5% v/w of dry

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mass. The essential oil sample was dried over anhydrous sodium sulphate (Na_2SO_4) and stored at 4 ˚C in the dark until tested and analysis by gas chromatography (GC) and gas chromatography–mass spectrometry (GC–MS).

Analysis of the Essential Oil

The essential oil was analyzed by Shimadzu GC-MS-QP5050A fitted with a fused methyl silicon DB-5 column (60 m × 0.25 mm i.d., 0.25 µm film thickness). Helium was used as carrier gas at a flow rate of 1.3 mL/min. The column temperature was kept at 50 °C for 3 min, increased to 300°C at a rate of 5°C/min, and finally kept at 300° C for 5min. The injector temperature was 270ºC and split ratio was adjusted at 1:33. The injection volume was 1 µL. The mass spectral (MS) data were obtained at the following conditions: ionization potential 70 eV; ion source temperature 200ºC; quadrupole temperature 100ºC; solvent delay 2 min; resolution 2000 amu/s and scan range 30-600 amu; EM voltage 3000 volts. Identification of compounds was based on direct comparison of the Kovats indices and MS data with those for standard compounds, and computer matching with the NIST NBS54K Library, by comparison with references. For quantization (area %), the GC analyses were also performed on an Agilent 6890 series apparatus fitted with a FID detector. The FID detector temperature was 300 ºC. To obtain the same elution order as with GC-MS, simultaneous auto-injection was performed on a duplicate of the same column applying the same operational conditions. Relative percentage amounts of the separated compounds were calculated from FID chromatograms.

Insecticidal toxicity assay

Insect toxicity of the essential oil was determined according to Kordali et al method.[30] The insects were collected from a laboratory culture and reared on a mixture of whole wheat and maize flour at the ratio 1:1 (0.5 kg). All insect species were reared at 27±2°C, 12% moisture content and continuous darkness for about 3 weeks without exposing to insecticides. Adults used in the experiments were 1-3 week old and of mixed sex. The essential oil was applied with an automatic pipette on a paper strip (6cm×3cm). The amounts of essential oil applied were 12, 24, 36 and 48 µl, corresponding to 3, 6, 9 and 12 µl /l air. Each dose was applied with automatic pipette as 100 µl acetone solution also acetone used as control. After evaporation of the acetone, the strip was attached to the underside of the desiccators to avoid direct contact of test material. Twenty adults of *O.mercator* and *T. castaneum* were placed in petri dishes (9cm), maintained at 27±2°C, 12 % moisture content and 12 h photoperiod. The experimental design was completely randomized, with three replicates. Insect mortality was evaluated after 12, 24, and 48 h of exposure. Control treatments without the essential oil were treated in the same way. Responses to treated sample versus control were converted to "percentage of mortality".[31]

Data analysis

Statistical analysis of the data was done using SPSS 10.0 software package. The results were showed significant difference at p <0.05 levels.

Results

The composition of the essential oil and insecticidal activity of *A. scorpius* investigated for the first time. Hydrodistillation of air dried aerial parts of *A. scorpius* yielded yellowish oils (1.5% v/w on dry weight basis). The major components of the essential oil are hexadecanoic acid (34.3%), tetrahydrogeranyl acetone (18.1%), 9, 12-Octadecadienoic acid, methyl ester (6.2%) and oleic acid (4.7%). The major volatiles in our study showed in Table 1 where the compounds are listed in order of their elution on DB-5 column.

Table1. Major constituents of the essential oils of *A. scorpius.*

Compounds	K.I*	(%)
Nonanal (Pelargonaldehyde)	1098	0.5
Tetradecane	1399	3.7
Geranyl acetone	1436	0.4
Tetrahydrogeranyl acetone	1469	18.1
Tetradecanoic acid (Myristic acid)	1720	0.9
Nonadecane	1900	2.2
2-methyl-Nonadecane	1966	1.1
n-Hexadecanoic acid(Palmitic acid)	1984	34.3
Eicosane	2000	2.1
9,12,15-Octadecatrien-1-ol	2034	2.6
Oleic acid)(-Octadecenoic acid 9	2085	4.7
9,12-Octadecadienoic acid, methyl ester	2092	6.2
Docosane	2200	4.4
Palmitaldehyde diallyl acetone	2237	1.7
Grouped components		
Long chain hydrocarbons (%)	13.5	
Alcoholic hydrocarbons & derivatives (%)	2.6	
Aldehydes and ketones hydrocarbons (%)	20.7	
Fatty acid and derivatives (%)	46.1	
Others (%)		
Total identified (%)	82.9	

* Kovats Indice (K.I) was calculated according to the method described by Kovats.

The toxicity of the essential oil on *O. mercator* and *T. castaneum* were determined in 3, 6, 9 and 12 µl /l air doses of the essential oil. The mortality increased with increase in the exposure times and dose. The oil caused 90.0% mortality of *O. mercator* and 85.2% mortality of *T. castaneum*, respectively (in 12 µl /l air). In the present study the essential oil of *A. scorpius* that containing hexadecanoic acid (34.3%), tetrahydrogeranyl acetone (18.1%) and 9, 12-Octadecadienoic acid, methyl ester (6.2%) as major compounds showed potent insecticidal activity (Table 2).

Table 2. The toxicity of essential oil of *A. scorpius* against *O. mercator* and *T. castaneum.*

		Mean mortality [a] (%)		
Essential oil (*A. scorpius*)	Dose (µl/l air)	Exposure time (h)		
		12	24	48
O. mercator	3	16.7± 3.3*	26.7± 3.3*	46.3± 3.3*
	6	35.3± 3.3*	46.0± 3.3*	50.0± 0.0*
	9	48.3± 0.0*	52.7± 0.0*	63.0± 3.3*
	12	50.7± 0.0*	86.0± 0.0*	90.0± 0.0*
T. castaneum	3	0.0± 3.3*	0.0± 0.0*	3.0± 3.3*
	6	0.0± 3.3*	6.3± 3.3*	13.0± 3.3*
	9	28.0± 0.0*	30.0± 3.3*	45.0± 0.0*
	12	46.7± 3.3*	56.0± 0.0*	85.2± 0.0*

[a]Mean±S.E. of three replicates, each set-up with 20 adults.
*Values followed by different letters in the same column differ significantly at p <0.05.

Discussion

In this research the chemical composition and insecticidal activity of the essential oil of *Acantholimon* genus reported for first time. Based on this report and according to the previous studies toxicity of the essential oil is attributed to their main components such as 9, 12-Octadecadienoic acid, methyl ester, docosane and farnesyl derivatives.[32-35] The essential oil caused significant mortality (80-90% for two pests) at 12 µl /l air after 48h of exposure (Table. 2).

Conclusion

The results showed that the essential oil of *A. scorpius* has varying degree of insecticidal activity against *O. mercator* and *T. castaneum*. However, more investigation needs to investigate potential insecticidal activity of *Acantholimon* genus.

Conflict of interests

The authors claim that there is no conflict of interest.

References

1. Giday M, Teklehaymanot T, Animut A, Mekonnen Y. Medicinal plants of the shinasha, agew-awi and amhara peoples in northwest ethiopia. J Ethnopharmacol. 2007;110(3):516-25. doi:10.1016/j.jep.2006.10.011
2. Voeks RA, Leony A. Forgetting the forest: Assessing medicinal plant erosion in eastern brazil. Econ Bot. 2004;58(1):S294-S306. doi:10.1663/0013-0001(2004)58[s294:ftfamp]2.0.co;2
3. Tabuti JRS, Lye KA, Dhillion SS. Traditional herbal drugs of bulamogi, uganda: Plants, use and administration. J Ethnopharmacol. 2003;88(1):19-44. doi:10.1016/S0378-8741(03)00161-2
4. Markandeya D. Antimicrobial and phytochemical screening of *Plumbago zeylanica* linn.(plumbaginaceae) leaf. J Exp Sci. 2011;2(3).
5. Chiu N, Chang K. The illustrated medicinal plants of taiwan. Southern Materials Center. Inc: Taipei; 1986. p. 99.
6. Pio Corrêa M. Diccionário das plantas úteis do brasil e das exóticas cultivadas. Rio de Janeiro: Imprensa Nacional;1984.
7. Pullaiah T. Encyclopaedia of world medicinal plants: Daya books; 2006.
8. Sunil C, Duraipandiyan V, Agastian P, Ignacimuthu S. Antidiabetic effect of plumbagin isolated from *Plumbago zeylanica* l. Root and its effect on glut4 translocation in streptozotocin-induced diabetic rats. Food Chem Toxicol. 2012;50(12):4356-63. doi:10.1016/j.fct.2012.08.046
9. Simonsen HT, Nordskjold JB, Smitt UW, Nyman U, Palpu P, Joshi P, et al. In vitro screening of indian medicinal plants for antiplasmodial activity. J Ethnopharmacol. 2001;74(2):195-204. doi:10.1016/s0378-8741(00)00369-x
10. Olagunju J, Jobi A, Oyedapo O. An investigation into the biochemical basis of the observed hyperglycaemia in rats treated with ethanol root extract of *Plumbago zeylanica*. Phytother Res. 1999;13(4):346-8. doi:10.1002/(sici)1099-1573(199906)13:4<346::aid-ptr444>3.0.co;2-d
11. Patil C, Patil S, Salunke B, Salunkhe R. Bioefficacy of *Plumbago zeylanica* (plumbaginaceae) and Cestrum nocturnum (solanaceae) plant extracts against Aedes aegypti (diptera: Culicide) and nontarget fish poecilia reticulata. Parasitol Res. 2011;108(5):1253-63. doi:10.1007/s00436-010-2174-6
12. Chaieb M, Boukhris M. Flore succincte et illustrée des zones arides et sahariennes de tunisie. Sfax: Association pour la protection de la nature et de l'environnement; 1998. p. 67.
13. Gunaherath G, Gunatilaka A, Sultanbawa MUS, Balasubramaniam S. 1, 2 (3)-tetrahydro-3, 3′-biplumbagin: A naphthalenone and other constituents from *Plumbago zeylanica*. Phytochemistry. 1983;22(5):1245-7. doi:10.1016/0031-9422(83)80232-5
14. Trabelsi N, Oueslati S, Falleh H, Waffo-Téguo P, Papastamoulis Y, Mérillon J-M, et al. Isolation of powerful antioxidants from the medicinal halophyte limoniastrum guyonianum. Food Chem. 2012;135(3):1419-24.

doi:10.1016/j.foodchem.2012.05.120

15. Asen S, Plimmer J. 4, 6, 4′-trihydroxyaurone and other flavonoids from limonium. Phytochemistry. 1972;11(8):2601-3. doi:10.1016/s0031-9422(00)88547-7
16. Sreelatha T, Hymavathi A, Murthy JM, Rani P, Rao JM, Babu KS. Bioactivity-guided isolation of mosquitocidal constituents from the rhizomes of *Plumbago capensis* thunb. Bioorg Med Chem Lett. 2010;20(9):2974-7. doi:10.1016/j.bmcl.2010.02.107
17. Sankaram AV, Srinivasarao A, Sidhu GS. Chitranone—a new binaphthaquinone from *Plumbago zeylanica*. Phytochemistry. 1976;15(1):237-8. doi:10.1016/s0031-9422(00)89104-9
18. Kishore N, Mishra BB, Tiwari VK, Tripathi V. Difuranonaphthoquinones from *Plumbago zeylanica* roots. Phytochem Lett. 2010;3(2):62-5. doi:10.1016/j.phytol.2009.11.007
19. Trabelsi N, Oueslati S, Ksouri R, Nassra M, Marchal A, Krisa S, et al. The antioxidant properties of new dimer and two monomers of phenolic acid amides isolated from *Limoniastrum guyonianum*. Food Chem. 2014;146:466-71. doi:10.1016/j.foodchem.2013.09.077
20. Pavela R. Efficacy of naphthoquinones as insecticides against the house fly, musca domestica l. Ind Crops Prod. 2013;43:745-50. doi:10.1016/j.indcrop.2012.08.025
21. Lattanzio V, Lattanzio VM, Cardinali A. Role of phenolics in the resistance mechanisms of plants against fungal pathogens and insects. Phytochemistry: Advances in research 2006;661:23-67.
22. Duffey SS, Stout MJ. Antinutritive and toxic components of plant defense against insects. Arch Insect Biochem Physiol. 1996;32(1):3-37. doi:10.1002/(sici)1520-6327(1996)32:1<3::aid-arch2>3.0.co;2-1
23. Pasdaran A, Delazar A, Nazemiyeh H, Nahar L, Sarker SD. Chemical composition, and antibacterial (against staphylococcus aureus) and free-radical-scavenging activities of the essential oils of *Scrophularia amplexicaulis* benth. Rec Nat Prod. 2012;6(4):350-5.
24. Asnaashari S, Delazar A, Alipour SS, Nahar L, Williams AS, Pasdaran A, et al. Chemical composition, free-radical-scavenging and insecticidal activities of the aerial parts of *Stachys byzantina*. Arch Biol Sci. 2010;62(3):653-62. doi:10.2298/abs1003653a
25. Isman MB. Plant essential oils for pest and disease management. Crop Prot. 2000;19(8-10):603-8. doi:10.1016/s0261-2194(00)00079-x
26. Isman MB, Miresmailli S, Machial C. Commercial opportunities for pesticides based on plant essential oils in agriculture, industry and consumer products. Phytochem Rev. 2010;10(2):197-204. doi:10.1007/s11101-010-9170-4
27. Pasdaran A, Nahar L, Asnaashari S, Sarker SD, Delazar A. Gc-ms analysis, free-radical-scavenging and insecticidal activities of essential oil of *Scrophularia oxysepala* boiss. Pharm Sci. 2013;19(1):1-5.
28. Aslan I, Özbek H, Çalmaşur Ö, Şahin F. Toxicity of essential oil vapours to two greenhouse pests, tetranychus urticae koch and bemisia tabaci genn. Ind Crops Prod. 2004;19(2):167-73. doi:10.1016/j.indcrop.2003.09.003
29. Muzaffariyan VA. Dictionary of iranian plant names: Farhang Muasir; 1996.
30. Kordali S, Aslan I, Çalmaşur O, Cakir A. Toxicity of essential oils isolated from three Artemisia species and some of their major components to granary weevil, sitophilus granarius(l.)(coleoptera: Curculionidae). Ind Crops Prod. 2006;23(2):162-70. doi:10.1016/j.indcrop.2005.05.005
31. Isman MB, Proksch P, Yan JY. Insecticidal chromenes from the asteraceae: Structure-activity relations. Entomol Exp Appl. 1987;43(1):87-93. doi:10.1111/j.1570-7458.1987.tb02207.x
32. Chang CL, Cho IK, Li QX. Insecticidal activity of basil oil, trans-anethole, estragole, and linalool to adult fruit flies of ceratitis capitata, bactrocera dorsalis, and bactrocera cucurbitae. J Econ Entomol. 2009;102(1):203-9. doi:10.1603/029.102.0129
33. Prajapati V, Tripathi A, Khanuja S, Kumar S. Anti-insect screening of medicinal plants from kukrail forest, lucknow, india. Pharm Biol. 2003;41(3):166-70. doi:10.1076/phbi.41.3.166.15092
34. Papachristos DP, Karamanoli KI, Stamopoulos DC, Menkissoglu-Spiroudi U. The relationship between the chemical composition of three essential oils and their insecticidal activity against acanthoscelides obtectus (say). Pest Manag Sci. 2004;60(5):514-20. doi:10.1002/ps.798
35. Ayvaz A, Sagdic O, Karaborklu S, Ozturk I. Insecticidal activity of the essential oils from different plants against three stored-product insects. J Insect Sci. 2010;10(21):1-13. doi:10.1673/031.010.2101