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Chemical Composition of the Essential Oils of the Flowers *Asphodelus aestivus Brot.* Grown Wild in Jordan

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ABSTRACT

Recent technological developments and methodological advances in GC-MS have become a common tool for investigating the quantity, quality, and chemical diversity of plant secondary metabolites. The flower parts of Asphodelus aestivus Brot. were studied, leading to the isolation and identification of various secondary metabolites, primarily essential oils: alcohol (26.9%); aldehyde (23%); alkanes, acetate derivatives, and aliphatic derivatives (19.2%); ketones (7.7%); and epoxides (3.8%). The principal oil components were 18.79% vetocitral C (trans), 17.27% hexadecyl acetate, 14.5% hexanal (2E), and 9.6% sabinene hydrate (trans). The identification of oil components was performed by matching their spectra with the mass spectra data bank.

Keywords: Medicinal plants, *Asphodelus aestivus* Brot, Jordan flora.

INTRODUCTION

Asphodelus L. is a genus comprising about 20 species in the Asphodelaceae family. They are native to Europe, North Africa, and Asia, primarily the Mediterranean. Many have a small rhizomatous crown and thick, fleshy roots that were first described by Carl Linnaeus in 1753 [1]. *Asphodelus aestivus* Brot, syn. (*Asphodelus microcarpus* Salzm. et Vivi) is a stout, robust herb with roots consisting of several spindle-shaped tubers, widely distributed across the coastal Mediterranean region [2].

Medicinal plants and their respective phytochemicals, mainly secondary metabolites, are utilized to address specific nutrient deficiencies and sustain secure food and primary healthcare medicines [3]. Natural materials derived from plants have played a significant role in drug discovery and in improving the healthcare system [4-8]. The World Health Organization (WHO) estimates that approximately 80 percent of the world's population relies on natural resources for their basic healthcare needs, while the remaining 20 percent uses integrated natural resources [8]. In the 21st century, 11 percent of the 252 essential medicines considered by the WHO as crucial came exclusively from flowering plants [9]. The species *Asphodelus* L. (Asphodelaceae) is consumed in large quantities in the cuisines (e.g., soups, pastries, etc.) of several countries and cultures [4]. *Asphodelus aestivus* is found on agricultural lands, around roads, and on calcareous soils in pastures in the Mediterranean basin [3, 7]. *Asphodelus aestivus* is used for food and as a folk medicine for eczema, stomach diseases, and hemorrhoids [6, 7]. Currently, there is a growing interest in phytochemicals as potential new sources of natural antioxidants, with the goal of using them in foods and pharmaceutical preparations to replace synthetic antioxidants [10].

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EXPERIMENTAL

Plant Material

The flowers of *A. aestivus* were collected from the Rujm Al-Shoof area (15 km northeast of Amman) in February 2022. The taxonomic identities of the collected plant material were confirmed with the assistance of a plant taxonomist (Dr. Mohammad Gharaibeh, Faculty of Agriculture, Jordan University of Science and Technology) and by comparing a collected voucher specimen with those of known identity in the herbarium of the Faculty of Agriculture, Jordan University of Science and Technology. A voucher specimen (ID No.: Phar 09-4) of the collected plant was deposited in the research laboratory of the Department of Medicinal Chemistry and Pharmacognosy, Faculty of Pharmacy, Jordan University of Science and Technology.

Oil distillation

The flowers of the collected plant material of *A. aestivus* were air-dried and ground to about a 0.5 mm particle size (30-35 mesh). Essential oils were obtained by subjecting 395 g of the ground materials to hydro distillation using the Clevenger-type apparatus (JSGW, India) for 4 h. The obtained oils (n = 2) were dried over anhydrous sodium sulfate, Na2SO4, and stored in dry, dark glass bottles at 4°C for later analysis.

Analysis of the Essential Oils

A quantitative analysis using gas chromatography with a flame ionization detector (GC-FID) was conducted using a Hewlett Packard HP-8590 gas chromatograph equipped with a split-splitless injector (split ratio 1:50) and an FID detector. The column (OPTIMA5 (5% diphenyl 95% dimethyl polysiloxane)) was a fused silica capillary column (30 m x 0.25 mm; 0.25 μm film thickness). The oils were separated under a linear temperature program set at a 3°C/min heating rate from 60-250°C and then held at 250°C for 5 min. The temperature of the injector and detector were maintained at 250°C and 300°C, respectively. The relative peak area for each component of the oil was measured. The concentrations of the oil components were calculated as a percentage content using their relative peak areas assuming a unity response by all components. Each sample was analyzed twice.

GC-MS Analysis

A GC-MS analysis was conducted on a Varian Chrompack CP-3800 GC/MS/MS-200, equipped with a split-splitless injector and a DB-5 GC column (5% diphenyl 95% dimethyl polysiloxane, 30 m x 0.25 mm ID, 0.25 μm film thickness). The injector temperature was set at 250°C with a split ratio of 1:10. Detector and transfer-line temperatures were 160°C and 230°C, respectively. A linear temperature program was used to separate the different oil components. Temperature programming was applied at a 3°C/min heating rate, starting from 60°C to 250°C, and then held at 250°C for 5 min. The mass detector was set to scan ions between 40-400 m/z using full scan mode and electron impact (EI, 70 eV). Each sample was analyzed twice. A hydrocarbon mixture of n-alkanes (C8-C20) was separately applied on a GC-MS using the same chromatographic conditions. The Linear retention index (arithmetic Kovat's index) was calculated for each component separated by GC-MS using the values of its retention time and the retention times of the reference n-alkanes applying the Van Den Dool equation [11-14 ,16,17].

The identification of oil components was performed by matching their spectra with the mass spectra data bank (Wiley, Nist, and Adams 2007 libraries), and also by comparing their calculated arithmetic indices with the reported values in the literature [11, 13, 15 -17].

RESULTS AND DISCUSSION

The simultaneous use of mass spectral and retention (Kovat's) index matching enabled the unequivocal identification of more than 98% of the components in the collected oil obtained from the flowers of the plant under study, as determined by GC and GC-MS. The oil yield (expressed as % v/w of dried material) was 0.35%. The analyses permitted the identification of 26 compounds in

the oils of A. aestivus. The identified components and their corresponding contents are presented in Table 1: Octane (1), hexanal (2), hexanal (2E) (3), N-heptanal (4), acetonyl acetone (5), ethyl hexanoate (6), octanol (N-) (7), sabinene hydrate (trans) (8), vetrocitral C (trans) (9), isopulegol (neoiso-) (10), terpinol (gamma-) (11), octenol acetate (2E) (12), geraniol (13), undecanal (14), decadienol (2E,4E) (15), docdecanol (n-) (16), pentadecane (17), liguloxide (18), hepatadecane (n-) (19), heptadecane (n-) (20), longifolol (21), acoron (22), farnesyl acetate (2E, 6E) (23), nonadecane (n-) (24), methyl hexadecanoate (25), and hexadecyl acetate (26).

The essential oil was characterized by high percentage levels of aldehydic volatile oil (hexanal, hexanal (2E), N-heptanal, vetrocitral C (trans), undecanal) (40.94%); followed by alkane and acetate derivatives (octane, ethyl hexanoate, pentadecane, hexadecane (n-), heptadecane (n-), farnecylacetate (2E,6E), nanodecane (n-), methylhexadecanoate, hexadecyl acetate) (31.15%); alcoholic volatile oil (octanol (N-) isopulegol (neoiso-), terpinol (gamma-), octenol acetate (2E), geraniol, decadienol (2E,4E), dodecanol (n-), longifolol)) (14.38%); bicyclic monoterpene (sabinene hydrate (trans) (9.6%); ketanes volatile oil (acetonyl acetone, acorone) (2.9%) and epoxides (liguloxide) (1.68%).

Vetrocitral C (trans) 18.79% was the principal oil component, with 17.27% hexadecyl acetate, 14.5% hexanal (2E), and 9.6% sabinene hydrate (trans) (shown in bold in Table 1) as the major oil constituents (8).

The identification of oil components was performed by matching their spectra with the data bank mass spectra (Wiley, Nist, and Adams 2007 libraries).

Table1: Chemical composition the essential oil hydro-distilled from the flowers parts of Jordanian *A. aestivus*

No	RI exp	RI lit	Content %	Compound
1	801	800	2.75	Octane
2	802	801	2.15	Hexanal
3	851	855	14.5	**Hexanal (2E)**
4	903	902	3.5	N- Heptanal
5	925	924	1.3	Acetonyl acetone
6	998	998	1.7	Ethyl hexanoate
7	1070	1068	3.6	Octanol (N-)
8	1100	1098	**9.6**	**Sabinene hydrate (trans)**
9	1107	1105	18.79	**Vetrocitral C (trans)**
10	1171	1171	1.3	Isopulegol (neoiso-)
11	1199	1199	2.2	Terpinol (gamma-)
12	1206	1209	1.1	Octenol acetate (2E).
13	1252	1249	1.9	Geraniol
14	1308	1306	2.0	Undecanal
15	1321	1321	1.1	Decadienol (2E,4E)
16	1469	1470	1.5	Dodecanol (n-)
17	1498	1500	1.3	Pentadecane
18	1537	1536	1.68	Liguloxide
19	1598	1600	1.17	Hexadecane (n-)

No	RI exp	RI lit	Content %	Compound
20	1700	1700	1.56	Heptadecane (n-)
21	1715	1714	1.68	Longifolol
22	1819	1820	1.6	Acorone
23	1844	1846	1.5	Farnesyl acetate (2E, 6E)
24	1897	1900	2.8	Nonadecane (n-)
25	1981	1921	1.1	Methyl hexadecanoate
26	2002	2003	17.27	**Hexadecyl acetate**
			40.94	Aldehide v. oils 31.15 Alkane and acetate derivatives v. oils 14.38 Alcohol v. oils
			9.6	Bicyclic monoterpene v. oils
			2.9	Ketanes v. oils
			1.68	Epoxides v. oils

RI exp: Linear (arithmetic) retention index calculated on DB-5 equivalent column
RI lit: reference retention index value from literature
*Average% content of 4 determinations (2 oil samples, 2 replicates each), for which the standard deviation (SD) values were within 2% (+2%) of the mean
Compounds in bold are the major components (≥1.0%)

CONCLUSION

The flower parts of Asphodelus aestivus Brot. were studied, leading to the isolation and identification of various secondary metabolites, mainly essential oil. The major essential oil components were vetrocitral C (trans), hexadecyl acetate, hexanal (2E), and sabinene hydrate (trans). The identification of oil components was performed using GC-MS.

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Conflicts of interest:

The authors have declared that there is no conflict of interest associated with the publication.

REFERENCES

1. Al-Eisawi D: Flora of Jordan Checklist, Revised. In *Book Flora of Jordan Checklist, Revised* (Editor ed.^eds.). City: The University of Jordan Press, Amman, Jordan. 2013.
2. Ghoneim MM, Ma G, El-Hela AA, Mohammad A-EI, Kottob S, El-Ghaly S, Cutler SJ, Ross SA: Biologically active secondary metabolites from Asphodelus microcarpus. *Natural product communications*. 2013; 8:1934578X1300800822.
3. Malmir M, Serrano R, Canica M, Silva-Lima B, Silva O: A comprehensive review on the medicinal plants from the genus Asphodelus. *Plants.* 2018; 7:20.
4. Alavi M, Hamblin MR, Martinez F, Kennedy JF, Khan H: Synergistic combinations of metal, metal oxide, or metalloid nanoparticles plus antibiotics against resistant and non-resistant bacteria. *Micro Nano Bio Aspects.* 2022; 1:1-9.
5. Alavi M, Rai M: Antisense RNA, the modified CRISPR-Cas9, and metal/metal oxide nanoparticles to inactivate pathogenic bacteria. *Cellular, Molecular and Biomedical Reports.* 2021; 1:52-59.
6. Alavi M, Rai M, Martinez F, Kahrizi D, Khan H, Rose Alencar De Menezes I, Douglas Melo Coutinho H, Costa JGM: The efficiency of metal, metal oxide, and metalloid nanoparticles against cancer cells and bacterial pathogens: different mechanisms of action. *Cellular, Molecular and Biomedical Reports.* 2022; 2:10-21.
7. Anand U, Jacobo-Herrera N, Altemimi A, Lakhssassi N: A comprehensive review on medicinal plants as antimicrobial therapeutics: potential avenues of biocompatible drug discovery. *Metabolites.* 2019; 9:258.
8. Kheyrodin H, Jami R, Rehman FU: Cellular structure and molecular functions of plants, animals, bacteria, and viruses. *Cellular, Molecular and Biomedical Reports*. 2022; 2:33-41.
9. Ahmadi S, Ahmadi G, Ahmadi H: A review on antifungal and antibacterial activities of some medicinal plants. *Micro Nano Bio Aspects.* 2022; 1:10-17.
10. Tawaha K, Alali FQ, Gharaibeh M, Mohammad M, El-Elimat T: Antioxidant activity and total phenolic content of selected Jordanian plant species. *Food chemistry.* 2007; 104:1372-1378.
11. Adams RP: *Identification of essential oil components by gas chromatography/mass spectrometry. 5 online ed.* Texensis Publishing; 2007.
12. Barakat SA, Hudaib M, Burns D: Composition of volatile oil and methanolic extract of jordanian Melissa officinals L. And actions againsthuman cancer cell lines. *Oriental Journal of Chemistry*. 2016; 32:2355.
13. Hudaib MM, Tawaha KA, Hudaib HS, Battah AH: Chemical composition of volatile oil from the aerial parts of Rosmarinus officinalis L. grown in Jordan. *Journal of Essential Oil Bearing Plants.* 2015; 18:1282-1286.
14. Van Den Dool H, Kratz PD: A generalization of the retention index system including linear temperature programmed gas-liquid partition chromatography. In *Book A generalization of the retention index system including linear temperature programmed gas-liquid partition chromatography* (Editor ed.^eds.). City; 1963.
15. Olimat S: Essential oils of the flowers Urginea maritima (L.) Baker (Liliaceae) grown in Jordan. *Asian J Pharmacogn.* 2021; 4:5-9.
16. Abbas M, Disi A, Al-Khalil S. Isolation and Identification of anti-ulcer components from *Anchusa strigosa* root. *Jordan Journal of Pharmaceutical Sciences*. 2009; 2(2).
17. Turk W, Olimat S. Determination of Colchicine Contents in Different Jordanian *Colchicum* spp. *Jordan Journal of Pharmaceutical Sciences*. 2015;8(2):119-22.

الزيوت الأساسية للزهور نبتة أسفوديلاس *ايستيفاس* .(*Asphodelus aestivus* Brot) والتي تنمو بريا في الأردن

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ملخص

أصبحت التطورات التكنولوجية الحديثة والتطورات المنهجية في كروماتوغرافيا الغاز ومقياس الكتلة أداة شائعة لدراسة كمية ونوعية والتنوع الكيميائي للمستقلبات الثانوية النباتية. تمت دراسة أزهار نبتة أسفوديلاس ايستيفاس، والتي تنمو بريا في الأردن، مما أدى إلى عزل وتحديد مختلف مستقلبات ثانوية مختلفة، ولا سيما الزيوت الأساسية: الكحول (26.9 %) ؛ الألدهيد (23 %)؛ والألكانات ومشتقات الأسيتات والمشتقات الأليفاتية (19.2 %) ؛ الكيتانات (7.7%) والأكاسيد الخارجية (3.8%). كان المكون النفطي الرئيسي 18.79٪ فيتروسيترال (Cترانس)، 17.27٪ أسيتات سداسي أسيل، 14.5٪ سداسي (2E)، 9.6٪ هيدرات سابينين (ترانس). تم تحديد مكونات الزيت عن طريق مطابقة أطيافها مع أطياف كتلة بنك البيانات.

الكلمات الدالة: النباتات الطبية، غوصلان، نباتات الاردن.

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