International Journal of NaturalandEngineeringSciences
E-ISSN: 2146-0086 16(2): 69-74, 2022
Research Article

FIRST REPORT ON THE CHEMICAL COMPOSITION OF ESSENTIAL OIL OF *HELIANTHEMUM RUFICOMUM* (CISTACEAE)

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(Received 27th March 2022; Accepted 14th July 2022)

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ABSTRACT. The essential oil of the aerial parts of *Helianthemum ruficomum* was obtained by steam distillation and analyzed by GC-FID and GC-MS. 59 components were identified corresponding to 98.3% of the total oil. Among the identified constituents, oxygenated compounds represented 56.3% and hydrocarbons (43%).The major components were palmitic acid (9.1%), heneicosane (8,0%), α-terpinolene (6.9%), 6,10,14-trimethylpentadeca-2-one (5.1%), tricosane (4.7%), dodecanoic acid (3.5%), eicosane (3.1%) and nonadecane (3.1%).This is the first report on the chemical composition of the essential oil of this species.

Keywords: *Helianthemum ruficomum, Cistaceae, essential oils, fatty acid, palmitic acid*

INTRODUCTION

Cistaceae family plants are distributed primarily in the northern temperate regions and the Mediterraneen area and are also found more rarely in North and South America. It is a large family which has striking flowers, and consists of annual or perennial herbaceous and shrub plants [1]. Cistaceae consists of 8genera and about 180 species [2] . *Helianthemum* genus (Cistaceae) contains approximately 110 species [3], some of them are important medicinal plants used in several countries for different purposes [4, 5, 6, 7, 8]. *Helianthemum* species are used in traditional medicines for gastrointestinal disorders, constipation, antiparasitic, analgesic, cytotoxic, blood-cutting, antiinflammatory, antiulcerogenic, and antiprotozoal properties [9, 10, 11, 12]. In Algeria this species was studied for antioxidant, antimicrobial and antibacterial activities [13, 14, 15]. Some species have been previously examined for bioactive components like flavonoids, phenolic acids, lignans and essential oils [14,16, 17, 18, 19, 20].

We have previously shown the high antioxidant potential of the extracts and their components, of *Helianthemum ruficomum* (Viv.) Spreng, an endemic species to the septentrional Sahara. Our study showed a remarkable richness of this species in phenolic compounds and flavonoids [15]. In continuation of this work, we studied the chemical composition of the essential oil of the aerial parts of this species. There are few studies on the essential oil of *Helianthemum* species [19, 21]. This is the first report on the chemical composition of essential oil of *Helianthemum ruficomum* by GC-FID and GC-MS.

MATERIALS AND METHODS

Plant material

The aerial parts of *Helianthemum ruficomum*, were collected from the area of Mougheul in the south-west of Algeria, and authenticated by M. Mohamed Benabdelhakem, director of the nature preservation agency, Bechar on the bases of [22]. A voucher specimen has been deposited in the Herbarium of the VARENBIOMOL research unit, University Frères Mentouri Constantine 1.

Extraction of the essential oil

The aerial parts of *Helianthemum ruficomum* were subjected to steam distillation in a Kaiser Lang apparatus for three hours. The obtained essential oil was collected and dried over anhydrous sodium sulphate and kept at 4°C until analysis. The yield of the oil was calculated in relation of the dry weight of the plant.

GC-FID Analysis

The essential oil was analyzed on an Agilent gas chromatograph (GC-FID) Model 6890, equipped with a HP-5MS fused silica capillary column (5%-diphenyl-95%-dimethylpolysiloxane) stationary phase (25 m x 0.25 mm, film thickness 0.25 µm), programmed from 50°C (5 min) to 250 °C at 3°/min and held for 10 min. Injector and flame ionization detector temperatures were 280 and 300 °C, respectively. The essential oil was diluted in acetone (3.5%, v/v) and injected in split mode (1/60), hydrogen was used as a carrier gas (1.0 mL/min). Solutions of standard alkanes (C_8-C_{20}) were analyzed under the same conditions to calculate retention indices (RI) with Van delDool and Kratz equation.

GC-MS Analysis

Mass spectrometry was performed on an Agilent gas chromatograph-mass spectrometer (GC-MS) Model 7890/5975, equipped with HP-5MS capillary column (25 m x 0.25 mm, film thickness 0.25 µm) programmed with the same conditions as for GC-FID. The mass spectrometer (MS) ionization was set in positive electron impact mode at 70 eV and electron multiplier was set at 2200 V. Ion source and MS quadrupole temperatures were 230°C and 180°C, respectively. Mass spectral data were acquired in the scan mode in the *m/z* range 33-450. The essential oil constituents were identified by matching their mass spectra and retention indices (RI) with those of reference compounds from libraries such as Adams and McLafferty& Stauffer [23, 24]. The proportions of the identified compounds were calculated by internal normalization.

RESULTS AND DISCUSSION

The analysis and identification of the compounds of the essential oil was performed using the GC-FID and GC-MS. The general chemical profile of the essential oil, the percentage content and retention indices of the constituents are summarized in (**Table 1**) and (Fig. 1). This investigation allowed the identification of 59 constituents corresponding to 99.3% of the total oil. Among the identified constituents, oxygenated compounds represented 56.3% and hydrocarbons 43%. The major components were Palmitic acid (9.1%), heneicosane (8%), α-terpinolene (6.9%), 6,10,14-trimethyl penta deca-2-one (5.1%), tricosane (4.7%), dodecanoic acid (3.5%), eicosane (3.1 %) and nonadecane (3.1%).

Table 1. *Composition of the essential oil of Helianthemum ruficomum with retention times, retention indices and percentages*

[a]Components	[b]RI	%	[a]Components	[b]RI	%
4-Hydroxy-4-methyl-2-pentanone	838	0.3	Tetradecanoic acid	1758	2.1
Caprylic acid	1178	1.2	Cyclocolorenone	1764	1
Decanal	1207	0.2	Myristoleic acid	1786	1.1
Nonanoic acid	1267	2.6	Ethyltetradecanoate	1793	1.6
α-Cubebene	1340	2.6	Octadecane	1800	2.1
Undecanol	1363	2.9	14-Hydroxy-α-muurolene	1806	0.7
α-Copaene	1379	0.4	Isopropyltetradecanoate	1817	0.3
Tetradecane	1400	0.3	6,10,14-Trimethylpentadeca-2-one	1841	5.1
Geranylacetone	1448	0.3	Phthalate	1859	5.6
α- Humulene	1460	0.4	Ethylpentadecanoate	1894	2.8
β-Ionone	1480	0.6	Nonadecane	1900	3.1
trans-5,6-Epoxy- β-ionone	1484	0.6	Ethyllinoleate	1906	0.2
Pentadecane	1500	0.6	α-Terpinolene	1954	6.9
γ-Cadinene	1512	0.4	Palmitic acid	1959	9.1
δ-Cadinene	1518	0.6	Ethylhexadecanoate	1993	2.0
α-Cadinene	1532	0.7	Eicosane	2000	3.1
Elemol	1551	0.2	Phytol	2094	2.0
Dodecanoic acid	1560	3.5	Heneicosane	2100	8.0
Spathulenol	1581	0.4	Osthole	2138	0.4
Caryophylleneoxide	1588	0.5	Ethyloctadecanoate	2194	1.3
Methylmorpholine	1593	0.5	Docosane	2199	1.8
Hexadecane	1600	0.8	(Z)-9-Tricosene	2294	1.2
epi-α-Muurolol	1647	1.2	Tricosane	2299	4.7
ß-Eudesmol	1660	1.4	Tricosan-2-one	2307	0.9
α-Eudesmol	1672	0.7	Tetracosane	2399	0.3
Isoeugenol	1678	1.0	Pentacosane	2499	1.4
β -Sinensal	1693	0.9	*n*-Pentacosane	2527	0.4
Heptadecane	1700	1.9	Heptacosane	2699	0.6
Pentadecanal	1716	0.7	Nonacosane	2899	0.7
Tetradecanal	1728	0.4			

Total identified: 99.3%
Oxygenated compounds: 56.3%
Non oxygenated compounds: 43%
RT (retention time)
[a] Compounds are listed in order of their RI.
[b] RI (retention index) measured relative to *n*-alkanes (C_8-C_{20}) using HP-5MS column.

These results are getting closer of those of *Helianthemum Kahiricum* [19] which reported that the main components of the oils were fatty acids : hexadecanoic acid (36.2%), tetradecanoic acid (7.3%), linoleic acid (6.5%), and dodecanoic acid (4.7%). However, heneicosane, 6,10,14-Trimethylpentadeca-2-one and nonadecane which are reported as minor compounds, were found in our species in more high content. This result is important because the major component palmitic acid have some biological activities, such as antitumor [25]. To the best of our knowledge this is the first report on essential oil chemical composition of *Helianthemum ruficomum.*

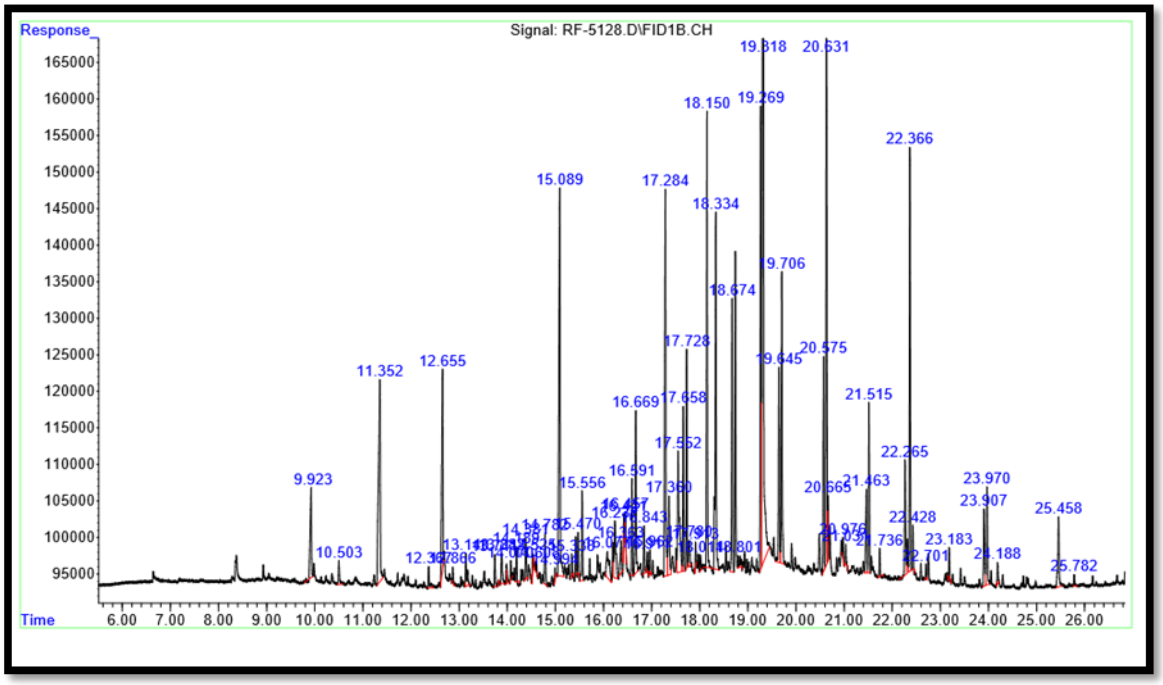

Fig. 1. *GC-FID Chromatogram of Helianthemum ruficomum essential oil.*

CONCLUSION

We report for the first time the essential oil composition of *Helianthemum ruficomum* collected from the area of Mougheul in the south-west of Algeria. Analysis by GC-FID and GC-MS allowed the identification of 59 components from which palmitic acid (9.1%), heneicosane (8%), α-terpinolene (6.9%), 6,10,14-trimethylpentadeca-2-one (5.1%), tricosane (4.7%), dodecanoic acid (3.5%),eicosane (3.1%), nonadecane (3.1%), undecanol (2.9%), ethylpentadecanoate (2.8%), nonanoic acid (2.6%), *α*-cubebene (2.6%),tetradecanoic acid (2.1%), octadecane (2.1%), ethylhexadecanoate (2%) and phytol (2%) were the main components. It should be noted that fatty acids accounted for a significant percentage (24.9%).

Acknowledgement. We are grateful to Dr. Mohamed Benabdelhakem, director of the nature preservation agency, Bechar, for the identification of the plant material and MESRS (DGRSDT) for financial support

REFERENCES

[1] Heywood, VH., Brummitt, R., Culham, A., Seberg, O. (2007): Flowering plant families of the world. Firefly Books Ontario. Royal Botanic Gardens, Kew. 424pp. ISBN 1 84246 165 5.

[2] Guzmán, B., Vargas, P. (2009): Historical biogeography and character evolution of Cistaceae (Malvales) based on analysis of plastid rbcL and trnL-trnF sequences. Organism Diversity Evolution 9(2): 83-99.

[3] Mabberley, D. J. (1997): The plant-book: a portable dictionary of the vascular plants. Cambridge University Press.

[4] Rubio-Moraga, Á., Argandoña, J., Mota, B., Pérez, J., Verde, A., Fajardo, J., Gómez-Navarro, J., Castillo-López, R., Ahrazem, O., Gómez-Gómez, L. (2013): Screening for polyphenols, antioxidant and antimicrobial activitiesof extracts from eleven *Helianthemum* taxa (Cistaceae) used in folk medicine in south-eastern Spain. Journal of Ethnopharmacology 148(1): 287-296.

[5] Meckes, M., Villarreal, M. L., Tortoriello, J., Berlin, B., Berlin, E. A. (1995): A microbiological evaluation of medicinal plants used by the Maya people of Southern Mexico. Phytotherapy Research 9(4): 244-250.

[6] Meckes, M., Torres, J., Calzada, F., Rivera, J., Camorlinga, M., Lemus, H., Rodríguez, G. (1997): Antibacterial properties of *Helianthemum glomeratum*, a plant used in Maya traditional medicine to treat diarrhoea. Phytotherapy Research 11(2): 128-131.

[7] Calzada, F., Alanis, A. D., Meckes, M., Tapia-Contreras, A., Cedillo-Rivera, R. (1998): In vitro susceptibility of *Entamoeba histolytica* and *Giardia lamblia* to some medicinal plants used by the people of Southern Mexico. Phytotherapy Research 12(1): 70-72.

[8] Rigat, M., Bonet, M. À., Garcia, S., Garnatje, T., Valles, J. (2007): Studies on pharmaceutical ethnobotany in the high river Ter valley (Pyrenees, Catalonia, Iberian Peninsula). Journal of Ethnopharmacology 113(2): 267-277.

[9] Meckes, M., Calzada, F., Tapia-Contreras, A., Cedillo-Rivera, R. (1999): Antiprotozoal properties of *Helianthemum glomeratum*. Phytotherapy Research 13(2): 102-105.

[10] Barbosa, E., Calzada, F., Campos, R. (2006): Antigiardial activity of methanolic extracts from *Helianthemum glomeratum* Lag. and *Rubus coriifolius* Focke in suckling mice CD-1. Journal of Ethnopharmacology 108(3): 395-397.

[11] Tawaha, K., Alali, F. Q., Gharaibeh, M., Mohammad, M., El-Elimat, T. (2007): Antioxidant activity and total phenolic content of selected Jordanian plant species. Food Chemistry 104(4): 1372-1378.

[12] Alsabri, S. G., Rmeli, N. B., Zetrini, A. A., Mohamed, S. B., Meshri, M. I., Aburas, K. M., Bensaber, S. M., Mrema, I. A., Mosbah, A. A., Allahresh, K. A. (2013): Phytochemical, anti-oxidant, anti-microbial, anti-inflammatory and anti-ulcer properties of *Helianthemum lippii*. Journal of Pharmacognosy and Phytochemistry 2(2): 86-96.

[13] Bouzergoune, F., Bitam, F., Aberkane, M., Mosset, P., Fetha, M., Boudjar, H., Aberkane, A. (2013): Preliminary phytochemical and antimicrobial activity investigations on the aerial parts of *Helianthemum kahiricum*. Chemistry of Natural Compounds 49(4): 751-752.

[14] Benabdelaziz, I., Marcourt, L., Benkhaled, M., Wolfender, J. L., Haba, H. (2017): Antioxidant and antibacterial activities and polyphenolic constituents of *Helianthemum sessiliflorum* Pers. Natural Product Research 31(6):686-690.

[15] Chemam, Y., Benayache, S., Marchioni, E., Zhao, M., Mosset, P., Benayache, F. (2017): On-line screening, isolation and identification of antioxidant compounds of *Helianthemum ruficomum*. Molecules 22(2): 239.

[16] Calzada, F., Lopéz, R., Meckes, M., Cedillo-Rivera, R. (1995): Flavonoids of the aerial parts of *Helianthemum glomeratum*. International Journal of Pharmacognosy 33(4): 351-352.

[17] Barbosa, E., Calzada, F., Campos, R. (2007): In vivo antigiardial activity of three flavonoids isolated of some medicinal plants used in Mexican traditional medicine for the treatment of diarrhea. Journal of Ethnopharmacology 109(3): 552-554.
[18] Calzada, F., Alanís, A. D. (2007): Additional antiprotozoal flavonol glycosides of the aerial parts of *Helianthemum glomeratum*. Phytotherapy Research 21(1): 78-80.
[19] Javidnia, K., Nasiri, A., Miri, R., Jamalian, A. (2007): Composition of the essential oil of *Helianthemum kahiricum* Del. from Iran. Journal of Essential Oil Research 19 (1): 52-53.
[20] Benabdelaziz, I., Haba, H., Lavaud, C., Harakat, D., Benkhaled, M. (2015): Lignans and Other Constituents from *Helianthemum sessiliflorum* Pers. Rec. Natural Products 9(3): 342.
[21] Gökşen, N., Demirci, B., Baldemir, A., Koşar, M. (2017): Essential oil composition of *Helianthemum canum* (L.) Baumg. (Cistaceae) growing in Turkey. Asian Society of Pharmacognosy 1(1): 5-10.
[22] Quezel, P., Santa S. (1963): Nouvelle flore de l'Algérie et des régions désertiques méridionales. CNRS, Paris. 710.
[23] Adams, R. P. (2007): Identification of essential oil components by gas chromatography/mass spectrometry. Vol. 456. Allured, Carol Stream (IL).
[24] Mc Lafferty, F.W., Stauffer, D. B. (1991): The Wiley/NBS registry of mass spectral data. 5th Edition, J. Wiley and Sons, New York.
[25] Harada, H., Yamashita, U., Kurihara, H., Fukushi, E., Kawabata, J., Kamei, Y. (2002): Antitumor activity of palmitic acid found as a selective cytotoxic substance in a marine red alga. Anticancer Research 22(5): 2587-2590.