ejpmr, 2024, 11(9), 50-53

SJIF Impact Factor 7.065

EUROPEAN JOURNAL OF PHARMACEUTICAL AND MEDICAL RESEARCH

www.ejpmr.com

Research Article

ISSN 2394-3211

EJPMR

PHYTOCHEMICAL SCREENING, ISOLATION AND CHARACTERIZATION OF ALKALOID FROM HAPLOPHYLLUM TUBERCULATUM (FORSK.) A JUSS

Zohour Mahgoub Hadrah*[1] and Eiz Aldeen Alfatih Barakat[2]

[1]University of Hafr Al Batin Collage of Science Dep. of Chemistry, Saudi Arabia.
[2]Sudan University of Science and Technology Dep. of Chemistry, Saudi Arabia.

***Corresponding Author: Zohour Mahgoub Hadrah**

University of Hafr Al Batin Collage of Science Dep. of Chemistry, Saudi Arabia.

Article Received on 09/07/2024 **Article Revised on 30/07/2024** **Article Accepted on 20/08/2024**

ABSTRACT

The present study was designed to screen the phytochemicals present in the ethanolic extract of the leaves of ***Haplophyllum tuberculatum*** and to isolate the alkaloids present. Phytochemical screening revealed the presence of alkaloids, flavonoids, glycosides and tannins. An alkaloid was isolated using TLC, solvent system (petroleum ether: ethyl acetate 2:3). Spectral analysis UV, IR, H1NMR and MS were carried to the isolated alkaloid.

KEYWORDS: Phytochemical screening, *Haplophyllum tuberculatum, alkaloids.*

INTRODUCTION

Alkaloids are basic nitrogenous compounds usually of complex chemical structure of plant origin and generally possessing physiological activities.[1] Alkaloids have important clinical use such as analgesics, anti malarial, antispasmodic, for pupil dilation and treatment of hypertension, mental disorder and tumors. They were all nitrogen hetrocycles which occur mainly in plants as their salts of common carboxylic acids such as citric, lactic, oxalic, acetic, malic and tartaric acids as well as fumaric, benzoic, acontic and verartic acids.[2]

H- tuberculatum has been used in the traditional medicine in the Mediterranean region for the treatment of various ailments such as vomiting, nausea, constipation, malaria, difficult child birth, anemia, rheumatism, gastric pain, and as aphrodisiac.[3] It has been reported that it used as antidote for scorpion stings.[4] for these and other reasons this plant attracted attention of researchers and still attract them to know more about it's medicinal and economic importance. *H-tuberculatum* is found in Saudi Arabia, Egypt, Palestine, Irag and Northern and central Sudan.[5] Today a number of phytochemical reports on *H- tuberculatum* have been published. Compounds isolated comprise diverse chemical structures typical of the genus *Haplophyllum*. However, *H-tuberculatum* showed a considerable biosynthetic trends in the production of typical rutaceous alkaloids such as angular pyranoquinolones, linear furoquinoline and the unique tyramine derived alkaloid.

Apart from the alkaloids, the arylnaphtalene and the aryltetrahydronaphthalene lignans represent one of the most characteristic secondary metabolite of this species. The Arial parts of Haplophyllum tuberculatum from Palestine have yielded two quinoline alkaloids, a known angular pyrano -2- quinoline, filindesine and a novel related compound designated as 3-(3,3dimethyl allyl) – 4 - 3,3 –dimethylallyloxy) – quinolone.[6]

Examination of a material from Iraq has given the more typical furoquinoline alkaloids flindersine, γ fagarine, skimmianine and evoxine.[1] On the basis of these studies it has been suggested that there at least two chemical racese of *H- tuberculatum.*[1] The 3 – (3,3 – dimethylallyl) -4 – 3,3 – dimethylallyloxy) – 2-quinolone has also been isolated from Lybian *H-tuberculatum* by Sheriha et al (1987).[7] *H-tuberculatum* a very variable taxon and according to Townsend (1966), its limitation is one of the most difficult problem presented by the genus. Compounds isolated comprise diverse chemical structures typical of the genus Haplophyllum. However, Haplophyllum tuberculatum showed a considerable biosynthetic trends in the production of typical rutaceous alkaloids such as angular pyranoquinolones, linear furoquinoline and the unique tyramine derived alkaloid Apart from the alkaloids, the arylnaphtalene and the aryltetrahydronaphthalene lignans represent one of the most characteristic secondary metabolite of this species.[5]

MATERIALS AND METHODS

Plant materials leaves *of Haplophyllum tuberculatum* (Forsk.)A Juss. Werecollected in the flowering stage during March from Khartoum, Sudan.

Documentation of the plant material
Herbarium materials were retained in the Department of Botany University of Khartoum and the samples were authenticated by the staff of the herbarium.

Phytochemical screening methods
The phytochemical screening was carried according to the following methods.

100g of powdered air dried leaves of *H-tuberculatum* were extracted with (200 ml) 95% ethanol using soxhlet extractor for 5 hours .The cool solution was filtered and the volume was adjusted to (200ml) by addition of enough ethanol this prepared extract was use for the following tests.

Alkaloids
(30ml) of the prepared extract was evaporated to dryness on a water bath (5ml) of 2N hydrochloric acid was added and the solution was heated with stirring in a water bath for 10 min. the cooled solution was filtered . To portion (5ml) of this solution, few drops of Dragendorff's reagent were added. Brown red precipitate was formed indicating the presence of alkaloids.

Flavonoids
(75ml) of the prepared extract was evaporated to dryness on a water bath; the cooled residue was defatted usig hexane. To (3ml) of filtrates few drops of methanolic aluminum chloride were added. Formation of a dark yellow color was taken as a positive test for flavonoids.

To 3ml of the prepared extract few drops of ferric chloride solution were added, development of a blue coloration was taken as a positive test for flavonoids.

Tannins
(25 ml) aliquot solution of the prepared extract was evaporated to dryness on a water bath and the residue was extracted with n-hexane and filtered, the hexane insoluble portion was filtered with (10ml) of hot saline solution (0.9%W/V of Nacl). The mixture was cooled and filtered and the volume adjusted to (100ml) with more saline solution; a blue color was formed indicating the presence of tannins.

Glycosides
(20ml) of the prepared extract was vigorously shaken in a test tube. The presence of a froth that could persist for one hour indicated the presence of glycosides.

Extraction of alkaloids
1Kg of the leaves of the plant were extracted with 70% methanol using sohxlet extractor, the extraction was carried for 72 hours. Methanol extract was subjected to rotatory evaporator. The reduced methanol extract was treated with hexane to remove fats, waxes and gums. The defatted methanol extract was treated with chloroform to extract alkaloids and other contents. The concentrated chloroform extract was shaken with 5% hydrochloric acid until no further alkaloids could be extracted (checked By Mayer's and Dragendorrf's reagents) .The combined acid extracts were made alkaline with concentrated ammonium hydroxide then extracted repeatedly with chloroform.

Isolation of alkaloids using TLC
Thin layer chromatography TLC was carried using solvent system petroleum ether : ethyl acetate (2:3). One compound isolated..

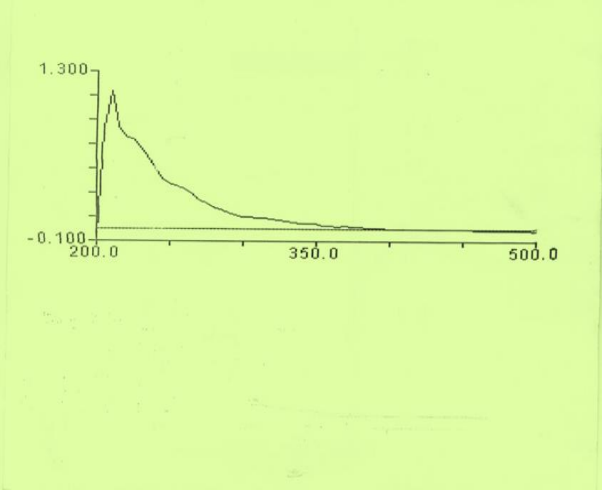
Physicochemical methods
UV spectrum were recorded in methanol on Ajanaway 6505 UV/VIS spectrometer. IR spectrum were recorded as thin film on Nacl disc on Thermonicolet 300 IR spectrophotometer. H1NMR spectrum were recorded on Gemini 300 (330MHz) in the appropriate deuterated solvent, using tetra methyl silane (TMS) as internal standard. Mass spectrum MS was recorded on JEOL AX 505W.

RESULT AND DISCUSSION
The preliminary phytochemical screening results .

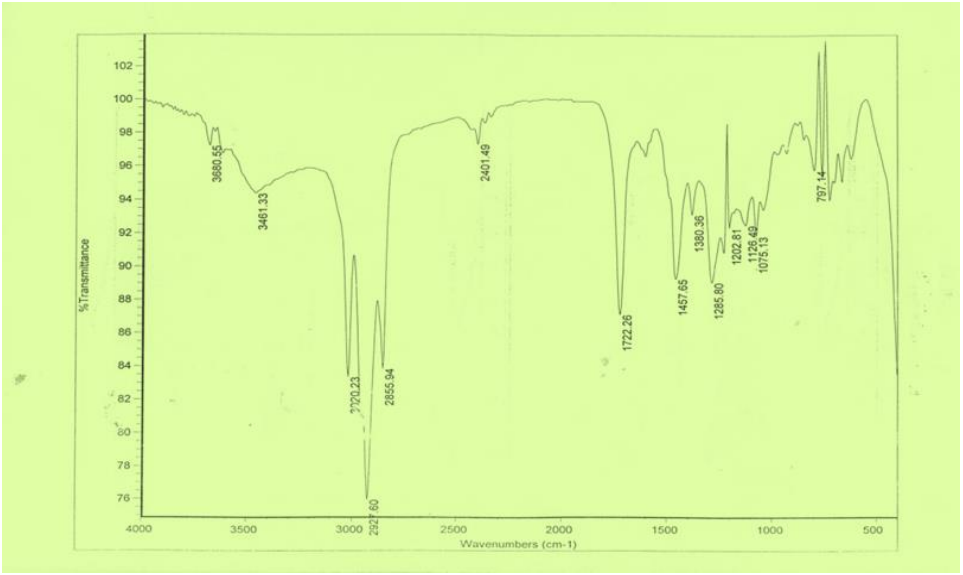
Alkaloids	+ + +
Flavonoids	+ + +
Tannins	+ +
Glycosides	+ +

+= presence of phytochemical

UV Result
The UV spectrum in methanol gave ג max. 210nm

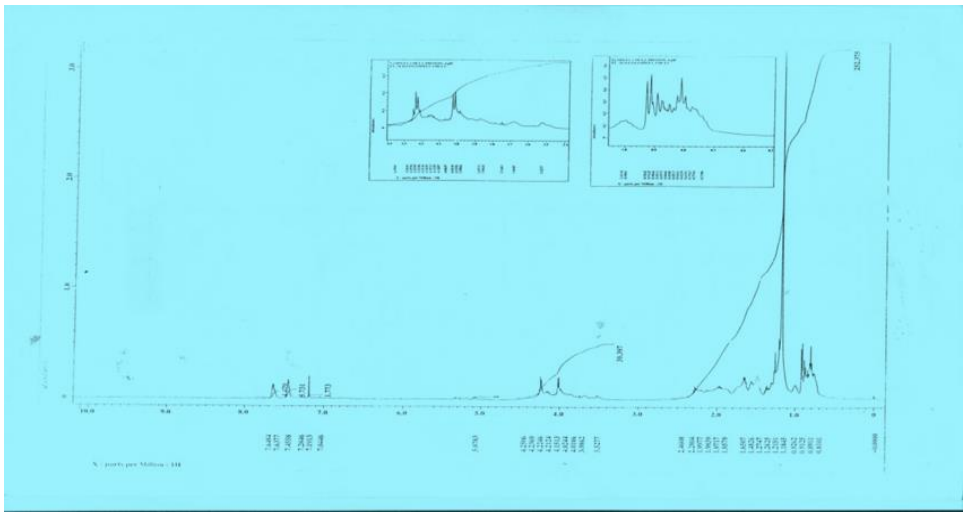


Infra red result

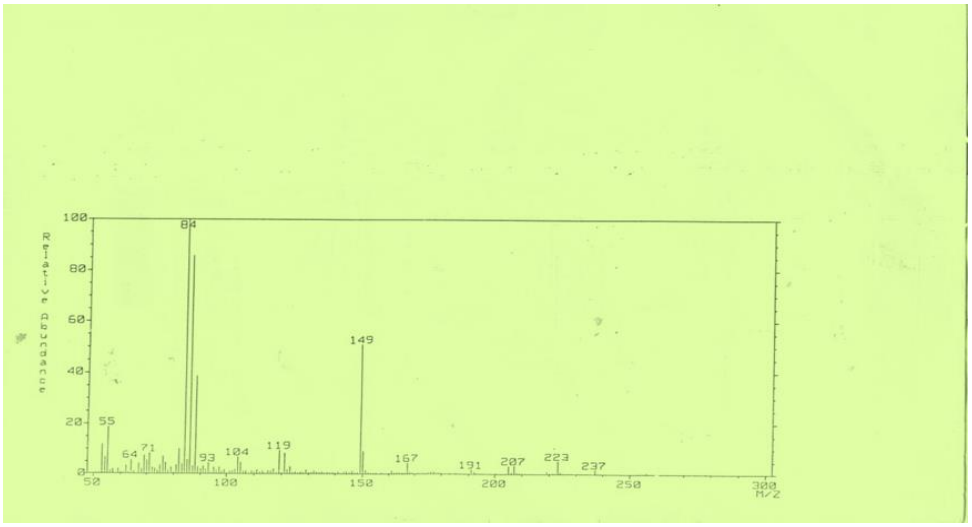


[1]HNMR result

Chemical shift ppm.: 0.8101, 0.8911, 0.9125, 0.9262, 1.1845, 1.2151, 1.2625, 1.2747, 1.4826, 1.6507, 1.9579, 1.9717, 1.9839, 1.9977, 2.2804, 2.4608, 3.5277, 3.9862, 4.0106, 4.0244,4.1513, 4.2124, 4.2246, 42369, 4.2506, 7.0446, 7.1913, 7.2846, 7.4558, 7.6377, 7.6464



Mass spectrum result



DISCUSSION OF THE RESULT

Characterization of the isolated alkaloidal compound

Isolated alkaloidal compound was analyzed by means of spectroscopic techniques IR, UV, ^{1}HNMR and Ms.

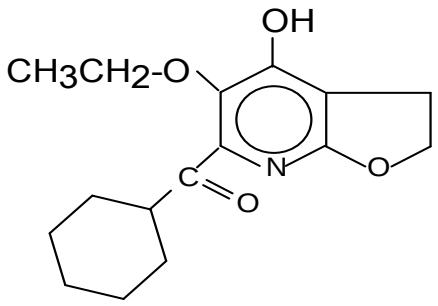
The mass spectrum result is characterized by a base peak (100% relative abundance) at m/z 84 and this attributed to a fragment of cyclohexane C_6H_{12}. The molecular ion decomposed into fragments did not appear at m/z 291.

Also, the spectrum is characterized by m/z 207 and this fragment is due to the loss of the hexane C_6H_{12} from the molecular ion m/z 291 (M-84).

The fragment m/z 191 appeared at the spectrum with relatively low abundance and this attributed to the loss of oxygen from the fragment m/z 207.

Also, the spectrum is characterized by small peaks with relatively low abundances at m/z 119 and m/z 104 and these two fragments can be attributed to the loss of (M-72) and a loss of methyl group (M-15) from m/z119 to give m/z 104 respectively.

considering the spectroscopic results of IR, UV, ^{1}HNMR and the mass spectrum result the following chemical structure of the alkaloidal constituent was suggested.



REFERENCES

1. Al – Shamma, A, AL-Douri, N.A. and Phillipson, J.D. phytochemistry, 1979; 18: 1417. alkaloids of haplophyllum tuberculatum
2. Hazim A.Kadry et al. chemistry of natural products, 1989; 2.
3. Bessonova-IA; yunusoy-S-Yu, Alkaloids of Haplophyllum roots. Institute of Khimii Rastitel, Tashkent Usbek, 1986.
4. Al – Yahiya, M.A., A1 L –Rehaily, A.J, Ahmed, M.S., AL – Said, M.M. and AL – Feraly, F.S. (+) Dihydroperfamine, and alkaloid from haplophylum tuberculatum.International journal of pharmacognacy, 1991; 29: 268-272.
5. Townsend, C, C. Towards a revision of H – tuberculatum A. Juss., 1949.
6. Lavie, D., Danieli, N., Weitman, R. and Glotter a new quinoline tybe alkaloid from H-tuberculatum (Rutaceae), 1968.
7. Sheriha, G.M and Abouamer, K.M. Lignan of H. tuberculatum. Phytochemistry, 1984; 23: 151-153.