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CHEMICAL INVESTIGATION OF *CYPERUS ARTICULATES*

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ABSTRACT

Two compounds 4, 9 –dihydroxy aromadendrene and 4-α-hydroxy aromadendrene were isolated from the n-hexane soluble partitionate of methanol extract of rhizome of *Cyperus articulatus.* At first the powdered rhizome of *C. articulatus* was extracted with 95% methanol. An aliquot of the extract was chromatographed over silica gel (Kiesel gel 60H) and vacuum liquid chromatography (VLC) column was eluted with n-hexane and ethyl acetate mixtures of increasing polarities to give a total of 18 fractions, each 100 ml. VLC fraction 6 was subjected to preparative thin layer chromatography (PTLC) using toluene-ethyl acetate (80:20) as solvent system. From the developed plates the bands were scrapped off and eluted with same solvent system- Toluene-Ethyl acetate (80:20), a 50:50 mixture of ethyl acetate and chloroform followed by 100 % ethyl acetate. The structures of the compounds were elucidated by high field NMR studies as well as comparison with spectral data of related compounds.

KEYWORDS: *Cyperus articulatus*, aromadendrene, Cyperaceae, VLC, PTLC, NMR.

INTRODUCTION

Nature represents an extraordinary reservoir of novel molecules and there is currently a resurgence of interest in natural products as a possible source of new lead compounds. Recent estimates suggest that several thousands of plants have been known with medicinal applications in various cultures. Bangladesh is a good source of the medicinal plants belonging to various families, including Cyperaceae. The Cyperaceae plants contain wide range of pharmacologically active compounds, including anticonvulsant, anti-rheumatic, anti-

diarrhoeal and anti-emetic activities. In Bangladesh there are about 40 plants belonging to the family Cyperaceae. Here, an attempt has been taken to study the chemical constituents of *Cyperus articulatus*, a member of the family Cyperaceae, growing in Bangladesh.

Cyperus articulatus is a tall marsh grass that grows near the edges of lakes, ponds, rivers, streams and wetlands. Various parts of plants were also reported to have antioxidant,[1] antimicrobial,[2-3] antibacterial,[4] sedative,[5] cytotoxic,[6] hepatoprotective,[7] antiepileptic[8] and antimalarial[9] properties. So, the main objective is to explore the possibility of developing new drug candidates from *Cyperus articulatus* for the treatment of various diseases.

MATERIALS AND METHODS

Experimental

NMR spectrum was acquired using the Ultra shield Bruker DPX 400 NMR instrument in $CDCl_3$ and the chemical shifts were reported in ppm with respect to residual non deuterated solvent signal.

Collection and preparation of plant materials

The plants were collected from their natural habitat around Dhaka University campus under the supervision of expert taxonomist on November 2013. Rhizomes were washed for several times with clean water. After that Rhizomes were dried under sun light for few days. Then these are cut into small pieces and dried in an oven for 24 hours. The dried plants were then pulverized into coarse powder using high capacity grinding machine.[10-11]

Extraction and isolation

The powdered rhizome (200 g) of *C. articulatus* was extracted with 95% methanol. An aliquot of the extract was chromatographed over silica gel (Kiesel gel 60H) and vacuum liquid chromatography (VLC) column was eluted with n-hexane and ethyl acetate mixtures of increasing polarities to give a total of 18 fractions, each 100 ml.[12-13] VLC fraction 6 was subjected to preparative thin layer chromatography (PTLC) using toluene-ethyl acetate (80:20) as solvent system.[14-16]

RESULT & DISCUSSION

Characterization of isolated compounds

Two aromadendrene derivatives were isolated from the n-hexane soluble partitionate of methanol extract of rhizome of *Cyperus articulatus.* The structures of the compounds were elucidated by high field NMR studies as well as comparison with spectral data of related compounds.[17-18]

Characterization of compound-1 as 4, 9 -dihydroxy aromadendrene derivative

VLC fraction 6 was subjected to preparative TLC (Stationary phase is Silica gel PF_{254} and Mobile phase is Toluene- Ethyl acetate (80:20). From the developed plates light sky blue colored band was visualized under UV at 366 nm. After spraying with vanillin-sulfuric acid reagent followed by heating in 110º C for 5 minutes it developed deep violet color. The band was scrapped on to aluminum foil and eluted initially using same solvent system- Toluene-Ethyl acetate (80:20), a 50:50 mixture of ethyl acetate and chloroform followed by 100 % ethyl acetate. The R_f value of the compound was 0.45. It was white amorphous powder. The compound was found to be soluble in n-hexane, ethyl acetate and chloroform.

The ^{1}H NMR spectrum contained signals for the hydrogens of the cyclopropane group at 0.29 (dd, *J* 11.5, 9.0 Hz, H-6) and 0.58 (ddd, *J* 12.2, 9.0, 4.8 Hz, H-7), and for three methyl groups at 1.19 (d, *J* 6.7 Hz, CH_3-15), 0.97 and 1.00 (s, CH_3-12 and 13, respectively). It also contained signals at 4.96 (1H, s) and at 4.85 (1H, s) which indicated exo-methylene group (-CH_2) at position C-10. Signal at 4.72 (1H, s) indicated hydroxyl proton at position C-9 because it was highly deshielded (table: 1) [19]. From ^{1}H NMR data, it can be proposed that structure of compound-1 is as following (Figure 1).

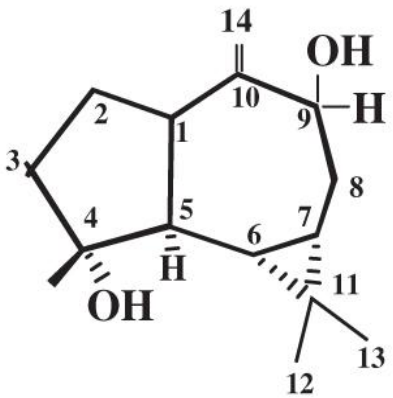

Figure. 1: 4, 9 –dihydroxy aromadendrene

Table 1: ^{1}H NMR spectral data of compound-1

Position	CA-65 (400 MHz, $CDCl_3$)
	δ_H, multi, *J* in Hz
1.	2.36
2	4.72,d,7.5 H
10	-
11	-
12	0.97,s
13	1.00,s
14	4.96,s &4.85,s
15	1.19,s

Characterization of compound-2 as 4-α-hydroxy aromadendrene

VLC fraction 6 was subjected to preparative TLC (Stationary phase is Silica gel PF_{254}, Mobile phase is Toluene- Ethyl acetate (80:20). From the developed plates light sky blue colored band was visualized under UV at 366 nm. After spraying with vanillin-sulfuric acid reagent followed by heating in 110º C for 5 minutes it developed deep brown color. The band was scrapped on to aluminum foil and eluted initially using same solvent system Toluene-Ethyl acetate (80:20), a 50:50 mixture of ethyl acetate and chloroform followed by 100 % ethyl acetate. The R_f value of the compound was 0.35. It was white amorphous powder. The ^{1}H NMR spectrum contained signals for the hydrogens of the cyclopropane group at 0.29 (dd, *J* 11.5, 9.0 Hz, H-6) and 0.58 (ddd, *J* 12.2, 9.0, 4.8 Hz, H-7), and for three methyl groups at 1.19 (d, *J* 6.7 Hz, CH_3-15), 0.97 and 1.00 (s, CH_3-12 and 13, respectively).These values also indicated the presence of hydroxyl group at position C-4. It also contained signals at 4.80 (1H, s) and at 4.65 (1H, s) which indicated CH_2 group at position C-10 (table-2).[19] From ^{1}H NMR data, it can be proposed that structure of the compound is as following (Figure 2).

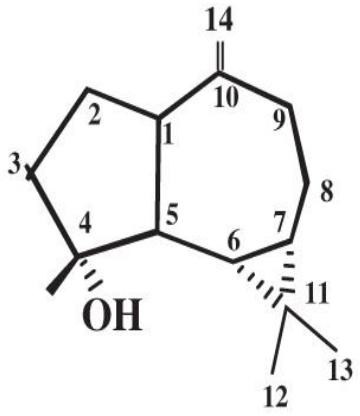

Figure 2: **4-α-hydroxy aromadendrene.**

Table 2: ^{1}H NMR spectral data of compound- 2.

Position	CA-71 (400MHz,$CDCl_3$)
	δ_H, multi, *J* in Hz
1.	2.36
10	-
11	-
12	0.97,s
13	1.00,s
14	4.80s & 4.65,s
15	1.19,s

It is important to mention that it was not possible to acquire ^{13}C NMR and mass spectral data due to insufficient sample and lack of instrumental facilities.

CONCLUSION

Phytochemial investigation of n-hexane soluble partitionate of methanol extract of rhizome of *Cyperus articulatus* led to the isolation of two compounds 4, 9 –dihydroxy aromadendrene and 4-α-hydroxy aromadendrene. Further biological studies are required to determine its specific therapeutic utilities.

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