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Original article

# Isolation of New Phytoconstituents From *Actiniopteris radiata*

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**ABSTRACT:**

Two new phytoconstituents have been isolated from petroleum ether extract of the whole plant of *Actiniopteris radiata*. The petroleum ether extract was subjected to column chromatography and isolated the phytoconstituents. Two new phytoconstituents were characterised by TLC, IR, UV spectral analysis, NMR and Mass spectra. Compound 1 is Dec-3-enyl, 2-(heptyloxy)-1-mercaptopyethyl terephthalate and compound 2 is 1-(Phenylethyl butanoate ether), 2-(3-ene heptanoate) ethane.

**KEYWORDS:** *Actiniopteris radiata*; Column chromatography; Polypodiaceae; Terephthalate.

## INTRODUCTION

*Actiniopteris radiata* is a desert fern belongs to the Polypodiaceae family and is distributed throughout India. A herbaceous miniature palm like fern up to 25 cm high with densely tufted stipe. Fronds fan like with numerous dichotomous segments which are rush like in texture, veins few, subparallel with distinct midrib, segments of fertile frond longer than those of the barren one, sori linear, elongate and submarginal. The plant is bitter, astringent, sweet, cooling, acrid, constipating, anthelmintic, haemostatic, antileprotic and febrifuge. It is useful in vitiated conditions of kapha and pitta, diarrhea, dysentery, helminthiasis, haemoptysis, haematemesis, leprosy, skin diseases, diabetes and fever<sup>1-3</sup>. Two new compounds have been isolated from the selected plant. The purity of the compounds were tested by thin layer chromatography on precoated silica gel GF<sub>254</sub> plates. The mobile phase used for TLC was petroleum ether:ethyl acetate:9:1. The structures of the isolated compounds were established by UV spectrum, IR, NMR and Mass spectrum. The structures of these compounds are

given in Fig. 1. The spectral data of compound 2 has been given in Table 1.

## MATERIALS AND METHODS

### PLANT MATERIAL

The whole plant of *Actiniopteris radiata* was collected from Nilgiri district, Tamil Nadu, India, in June 2012. The plant was identified by Dr. S. Rajan, Field Botanist, Survey of Medicinal Plants and Collection Unit, Emerald, Nilgiri. A voucher specimen has been deposited at Survey of Medicinal Plants and Collection Unit, Emerald, Nilgiri.

### INSTRUMENTS

Melting points were determined using a Lab India melting point apparatus. UV-Visible spectrums were recorded using a Shimadzu UV-1700. IR spectrums were recorded on a Shimadzu FTIR-8400s. <sup>1</sup>H (500 MHz) and <sup>13</sup>C (100 MHz) spectrums were recorded on a BRUKER AV-400. ESIMS spectrums were recorded on a HCT-Ultra PTM discovery, BRUKER.

## EXTRACTION AND ISOLATION

The coarsely powdered plant material (500 g) was packed in soxhlet apparatus. The packed plant material extracted successively with petroleum ether, chloroform, ethyl acetate and ethanol for 18-20 hrs. These extracts were filtered and dried under vacuum. The column chromatography was used as a purification technique for isolation of compounds from extracts<sup>4-6</sup>. The petroleum ether extract was selected for column chromatography. Fractionations were carried out using silica gel column. The column was prepared by wet packing method using petroleum ether as solvent. The extract dried under vacuum was found to be 10.0 g. It was packed in a column chromatography with a silica gel 60-120 mesh size as adsorbent (300.0 g). The mobile phase was allowed to flow through the column in the increasing order of polarity and fractions were collected. Thin layer chromatography was performed for all collected fractions and the fractions showing similar chromatograms were combined. The eluents used were Petroleum ether 100, Petroleum ether: Chloroform (95:5, 90:10, 85:15, 80:20), Chloroform 100, Chloroform: Ethyl acetate (95:5, 90:10, 85:15, 80:20), Ethyl acetate 100, Ethyl acetate: methanol (95:5, 90:10, 85:15, 80:20). Eighty five fractions were collected. The purification was done for major fractions by re-column<sup>7</sup>. The fractions 35 to 56 were evaporated to yield 350 mg of yellow residue. The re-column of this fraction yielded 14 mg of compound 1 and 18 mg of compound 2.

**Compound 1:** A white amorphous powder; UV (CHCl<sub>3</sub>) λ<sub>max</sub>: 222 (0.47), 258 (0.73) nm; IR ν<sub>max</sub>: 1735, 1647, 1635, 1375 cm<sup>-1</sup>; Melting point 104°C; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 7.53 (dd, 2H, H-3, H-5), 7.70 (dd, 2H, H-2, H-6), 5.35 (d, 2H, H-11), 5.28 (d, 2H, H-2<sup>1</sup>), 1.29 (br, s, <sup>1</sup>H), 0.90 (d, 2H, H-2); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 173.32 and 167.77 (2-COOH), 130 (C-1), 131 (C-2, C-6), 128 (C-3, C-5), 132.42 (C-4), 28.91, 29.11, 29.27, 29.36, 29.48, 29.70, 30.33 and 31.92 (-CH<sub>2</sub>), 62.00, 64.00, 68.00 and 69.00 (C-O); - ESI-MS: m/z 478 [M<sup>+</sup>] (calcd for C<sub>27</sub>H<sub>42</sub>O<sub>5</sub>S, 478.00)

**Compound 2:** A white amorphous powder; UV (CHCl<sub>3</sub>) λ<sub>max</sub>: 232 (0.32), 274 (0.86) nm; IR ν<sub>max</sub>: 1746, 1627 cm<sup>-1</sup>; Melting point 118°C; <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 8.10 (dd, 2H, H-2, H-6), 7.45 (dd, 2H, H-3, H-5), 7.60 (t, 1H, H-4), 5.35 (m, 2H, CH=CH), 2.00 (d, 2H, CH<sub>2</sub>); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ 178.50 and 170.68 (2-C=O), 129.69 (C-1), 130.08 (C-2, C-6), 128.40 (C-3, C-5), 133.53 (C-4), 22.64, 24.86, 25.61, 27.18, 29.00, 31.49, 31.88

and 33.86 (-CH<sub>2</sub>); (+)- ESI-MS: m/z 362 [M<sup>+</sup>] (calcd for C<sub>21</sub>H<sub>30</sub>O<sub>5</sub>, 362.00)

## RESULTS AND DISCUSSION

The petroleum ether extract was chromatographed over silica gel column to yield compound 1 and 2.

**Compound 1**, a white amorphous powder with absorption maximum 258 nm indicating that carbonyl group is in conjugation with an aromatic ring system. The IR spectrum shows the presence of ester carbonyl group at 1735 and 1647 cm<sup>-1</sup>, 1635 cm<sup>-1</sup> due to C=C. The ESIMS shows M<sup>+</sup> ion at m/z 478 suggests a molecular formula of compound 1 as C<sub>27</sub>H<sub>42</sub>O<sub>5</sub>S. Melting point 104 °C. The <sup>1</sup>H-NMR has signals at δ 7.53 (dd, 2H, H-3, H-5) and 7.70 (dd, 2H, H-2, H-6) indicating the presence of aromatic ring. The <sup>13</sup>C-NMR has six signals at δ 130.00 (C-1), 131.00 (C-2, C-6), 128.00 (C-3, C-5) and 132.42 (C-4) indicating the presence of six aromatic carbon atoms. The two signals in the downfield at δ 173.32 and 167.77 suggest the presence of two -COO groups. The protons were assigned to respective carbon atoms using HSQC spectral data. The signal at δ 5.35 is H-11 has cross Peak with proton at δ 2.00, which has cross peak with protons at δ 1.29 (br, s) indicating the presence of a long alkyl chain connecting to the double bond. Further absence of any cross peaks with signals in the same region suggested the presence of an alkyl substituent at the other carbon (C-10) of the double bond. The corresponding signal present at δ 130.00 in <sup>13</sup>C-NMR. One more signal is observed in the <sup>1</sup>H-NMR at δ 5.28 (H-21) for one proton with a corresponding signal at δ 69.00 in the <sup>13</sup>C-NMR (based on HSQC spectral data) suggesting the presence of a proton which is connected to hetero atoms. The downfield nature of this signal may probably due to the presence of two hetero atoms on either side of the proton. The proton at δ 5.28 has cross peak with signals at δ 4.14 and 4.30 indicating that the adjacent methylene group (H-31) was in turn connected to the hetero atom oxygen. The <sup>13</sup>C-NMR has two signals at δ 173.32 and 167.77 indicates the presence of two carbonyl group.

The presence of signals at δ 14.05 and 14.12 in the <sup>13</sup>C-NMR indicates the presence of two methyl groups. The <sup>1</sup>H-NMR has signals at δ 0.90 as a multiplet suggests that the methyl groups are connected to -CH<sub>2</sub> groups. The broad singlet at δ 1.29 indicates the presence of long chain

methylene groups and was supported by the signals at  $\delta$  28.91, 29.11, 29.27, 29.36, 29.48, 29.70, 30.33 and 31.92 in  $^{13}\text{C}$ -NMR. The signal at  $\delta$  2.30 and 2.00 in  $^1\text{H}$ -NMR and at  $\delta$  34.33 and 34.05 in  $^{13}\text{C}$ -NMR suggests the presence of protons adjacent to the double bond. The signals at  $\delta$  62.00, 64.00, 68.00 and 69.00 in  $^{13}\text{C}$ -NMR supports the fact that they are carbon atoms attached to the oxygen atoms and they are two ester carbonyl groups. Therefore the structure of compound 1 was elucidated as Dec-3-enyl, 2-(heptyloxy)-1-mercaptptoethyl terephthalate.

**Compound 2**, a white amorphous powder. The UV spectrum shows the absorption maximum at 274 nm. The IR spectrum exhibits C=O group at  $1746\text{ cm}^{-1}$  and C=C at  $1627\text{ cm}^{-1}$ . The positive mode ESI-MS exhibited molecular ion peak at  $m/z$  362. It shows molecular formula of  $\text{C}_{21}\text{H}_{30}\text{O}_5$  and molecular weight 362.00. Melting point  $118\text{ }^\circ\text{C}$ . The signals at  $\delta$  8.1 (2H, dd,  $J=7.0\text{ Hz}$ , 2 Hz, H-2, 6), 7.6 (1H, t,  $J=7.0\text{ Hz}$ , H-4) and at  $\delta$  7.45 (2H, t, d,  $J=7.0\text{ Hz}$ , 2 Hz, H-3, 5) in  $^1\text{H}$ -NMR indicates the presence of a monosubstituted aromatic ring. This was supported by the  $^{13}\text{C}$ -NMR Signals at  $\delta$  130.08 (2C, C-2, C-6), 128.40 (2C, C-3, C-5), 133.53 (C-4) and at 129.69 (C-1). The  $^1\text{H}$ -NMR has Signals at  $\delta$  5.35 (m, 2H) for two unsaturated protons. These two signals have cross peaks with signals at  $\delta$  2.0 and 2.8 indicating the presence of two methylene groups on either side. The signals at  $\delta$  135.83 and 129.00 in  $^{13}\text{C}$ -NMR were due to unsaturated carbon atoms.

The signal at  $\delta$  55.96, 63.32, 65.11 and 70.26 in the  $^{13}\text{C}$ -NMR indicates the presence of methylene groups attached to oxygen function. The corresponding signals at  $\delta$  3.60, 3.70, 3.80, 3.90 and 4.15 in proton NMR indicates the presence of five methylene groups attached to oxygen. The methylene groups attached to carbonyl groups are indicated by the presence of signals at  $\delta$  2.30 in  $^1\text{H}$ -NMR and at  $\delta$  34.12 in  $^{13}\text{C}$ -NMR. The presence of a long hydrocarbon chain in the molecule was proved by the presence of a methyl group at  $\delta$  0.90 in  $^1\text{H}$ -NMR and at  $\delta$  14.06 in  $^{13}\text{C}$ -NMR and a chain of methylene groups showed signals at  $\delta$  1.29 in  $^1\text{H}$ -NMR and couple of signals between  $\delta$  22.64 to 33.86 in  $^{13}\text{C}$ -NMR. The cross peaks were observed between the peaks at  $\delta$  8.1 and 7.45, 7.45 and 7.60, confirming the presence of only one aromatic ring. The cross peaks of the signals at  $\delta$  5.35 with signals at  $\delta$  2.00 and 2.30 indicates the presence of methylene groups on either side of the double bond ( $-\text{CH}_2-\text{CH}=\text{CH}-\text{CH}_2-$ ). The cross

peaks between the signals at  $\delta$  4.15 and 3.90, 3.90 and 3.70, 3.80 and 3.60, 3.70 and 3.60 indicates that the methylene protons under oxygen function couple among themselves. The presence of two carbonyl groups is confirmed by the presence of two signals at  $\delta$  178.50 and 170.68 in the  $^{13}\text{C}$ -NMR spectrum. These signals were assigned based on the HSQC data. Thus structure of compound 2 was elucidated as 1-(Phenylethyl butanoate ether), 2-(3-ene heptanoate) ethane.

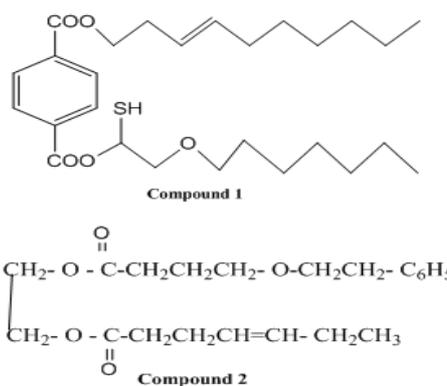


Figure 1. Structure of compound 1 and 2.

Table 1. NMR Spectral data of Compound 2.

| Carbon                                   | Signal( $\delta$ )   | Proton                              | Signal( $\delta$ )                 |
|--|--|-------------------------------------|------------------------------------|
| C=O                                      | 178.50<br>170.68   |                                     |                                    |
| <b>Aromatic ring</b>                     |  |                                     |                                    |
| C-1                                      | 129.69   |                                     |                                    |
| 2, 6                                     | 130.08   | H-2, 6                              | 8.10, dd, 2H                       |
| 3, 5                                     | 128.40   | H-3, 5                              | 7.45, dd, 2H                       |
| 4  | 133.53   | H-4                                 | 7.60, t, 1H                        |
| <b>Unsaturation</b>                      |  |                                     |                                    |
| 1  | 128.00   | CH=CH                               | 5.35, m, 2H                        |
| 2  | 135.83   |                                     |                                    |
| <b>Carbon under Oxygen function</b>      |  |                                     |                                    |
| C-O-                                     | 70.26<br>65.11<br>63.32<br>55.96                                     | CH <sub>2</sub> -O                  | 3.90<br>4.15<br>3.70, 3.60<br>3.80 |
| <b>Carbon attached to carbonyl group</b> |  |                                     |                                    |
| $-\text{CH}_2-\text{C}-$<br>  <br>O      | 37.26<br>34.12   | $-\text{CH}_2-\text{C}-$<br>  <br>O | 2.30                               |
| <b>Long chain methylene groups</b>       |  |                                     |                                    |
|  | 33.86<br>31.88<br>31.49<br>29.00<br>27.18<br>25.61<br>24.86<br>22.64 | $-(\text{CH}_2)-$                   | 2.00<br>1.56<br>1.29               |
| <b>Methyl group</b>                      | 14.06  | CH <sub>3</sub>                     | 0.90                               |

## CONCLUSION

Two new compounds have been isolated from *Actiniopteris radiata*. The chemical name of these compounds were found as Compound 1 is Dec-3-enyl, 2-(heptyloxy)-1-mercaptopyethyl terephthalate and compound 2 is 1-(Phenylethyl butanoate ether), 2-(3-ene heptanoate) ethane.

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