

Four Compounds Isolated from *Polygonum lanatum* Roxb

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(Received : 25 January 2012; Accepted : 2 March 2013)

I. Introduction

Polygonum lanatum (Bengali name-Bishkatali; Family-*Polygonaceae*), a herb having anti-inflammatory, analgesic and diuretic activity¹, is distributed in Southeast Asia. Previous phytochemical investigations with *Polygonum* species led to the isolation of sterols², terpenoids³, flavonoid glycosides⁴, coumaryl glycoside², lignans⁵, quinones², 6-methoxyplumbagin⁶. Here, we report the isolation of *p*-anisaldehyde (**1**), 4-hydroxy benzaldehyde (**2**), 6-acetyl-2,3-dihydroxyanthracene-9,10-dione (**3**) and 2-hydroxy-3 methoxy-2,9-dimethyl-1,2 dihydrotetraphene-7,12-dione (**4**) from the methanol extract of the stem of *Polygonum lanatum* Roxb.

II. Materials and Methods

General experimental procedure: The ¹H NMR spectra were recorded by using a Bruker AMX-400 (400 MHz) instrument. For NMR studies deuterated chloroform was used and the δ values for ¹H spectra were referenced to the residual nondeuterated solvent signals.

Plant Material: The fresh stem of *Polygonum lanatum* was collected from Chittagong. It was identified by Bangladesh National Herbarium, Dhaka (DACB-31286). All the stems were cut into small pieces and then air dried for several days. The pieces were then oven dried for 24 hours at considerably low temperature to effect grinding. The plant was then ground into a coarse powder using a grinding machine.

Extraction and Isolation: The air dried stem bark powder (503 g) of *Polygonum lanatum* was successively extracted with *n*-hexane, dichloromethane and methanol through hot extraction process with the help of soxhlet apparatus. Afterwards the extracts were filtered by suction. The filtrates were then concentrated by a Rotary evaporator, which afforded *n*-hexane (4.25g), dichloromethane (2.0 g) and methanol (4.0 g) soluble materials. The methanol soluble fraction (3.5 g) was fractionated by column chromatography over silica gel (70-230 mesh) eluted with *n*-hexane followed by mixture of *n*-hexane, dichloromethane and finally dichloromethane and methanol mixtures of increasing polarity to give 46 fractions collected in each 200ml. In addition, the fractions from 5th to 7th taken from 46 fractions were combined and

subjected to column chromatography. As a result, 44 fractions were obtained and the fractions 13-15 taken from 44 fractions were combined and further subjected to column chromatography. Furthermore, the fractions 32-38 taken from the last column chromatography were mixture and also subjected to preparative thin layer chromatography (stationary phase- silica gel F₂₅₄, mobile phase – 70% toluene in ethyl acetate with few drops of acetic acid, thickness of plates-0.5 mm). Finally from the developed plate, four sets of identical bands were scrapped and initially eluted with a mixture of chloroform and ethyl acetate followed by 100% ethyl acetate. Consequently evaporation of solvent afforded the four compounds.

***p*-Anisaldehyde (1):** colored crystal; ¹H NMR (400 MHz, CDCl₃): δ 9.88 (1H, s, 1-CHO), 7.84 (2H, d, *J* = 9 Hz, H-2 and H-6), 7.00 (2H, d, *J* = 9 Hz, H-3 and H-5), 3.89 (3H, s, 4-OCH₃)

Hydroxy benzaldehyde(2): white gum; ¹H NMR (400 MHz, CDCl₃): δ 9.97 (1H,s, 1-CHO), 7.82 (2H, d, *J* =8.4, 2-H and H-6), 7.35 (2H, d, *J* =8.4, 3-H and 5-H), 7.51 (1H, s, 4-OH)

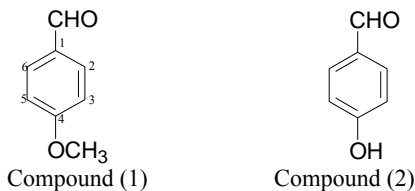
6-Acetyl-2,3-dihydroxyanthracene-9,10-dione (3): white gum; ¹H NMR (400 MHz, CDCl₃): δ 7.48 (1H, s, H-1, H-2, 2-OH, 3-H, 3-OH), 7.50 (1H, s, H-4), 8.54 (1H, d, H-5, H-6), 2.62 (3H, s, 6-COCH₃), 8.07 (1H, d, *J* =6.6, H-7), 8.15 (1H, d, *J* =6.6, H-8, H-9, H-10)

2-hydroxy-3 methoxy-2,9-dimethyl-1,2-dihydrotetraphene-7,12-dione (4): white gum; ¹H NMR (400 MHz, CDCl₃): δ 2.72 (2H, s, 1-H, 2-H, 2-OH), 2.15 (3H, s, H-3, 2-CH₃), 3.62 (3-OCH₃), 5.40 (1H, bs, H-4), 7.32 (1H, d, *J* =8.0, H-5), 8.0 (1H, d, *J* =8.0, H-6, H-7), 8.08 (1H, d, H-8), 2.34 (3H, s, 9-CH₃), 7.65 (1H, dd, *J* =8.0, H-10), 8 (1H, d, *J* =8.0, H-11, H-12)

III. Results and Discussion

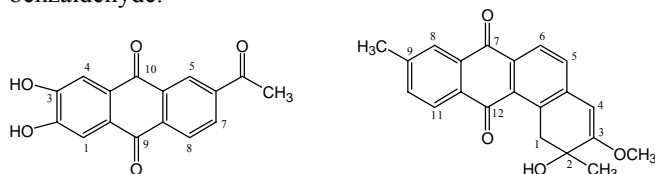
Compound (1) was obtained as colored crystal. It appeared as a dark quenching spot on the TLC plate under UV light at 254 nm and was soluble in dichloromethane and chloroform. The ¹H NMR spectra of compound (1) was given in the experimental part. On this basis of the NMR spectroscopic data, the compound (1) was identified as *p*-anisaldehyde.

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Compound (2) was obtained as colorless gummy material. It appeared as dark unclenching spots on TLC plate under UV light at 254 nm and was soluble in CH_2Cl_2 and CHCl_3 . The ^1H NMR spectrum of compound (2) showed signals for four methines, one aldehydic group and one hydroxyl group. The structure of compound (2) was elucidated by direct comparison of each spectral data with compound (1). Although the ^1H NMR spectrum of compound (2) was in close correspondence to that of compound (1), some differences could be observed, such as the resonance at $\delta=3.89$ in the ^1H NMR could not be observed in compound (2). On the other hand one-proton broad singlet at $\delta=7.51$ was appeared in the ^1H NMR spectrum of compound (2) corresponding to the 4-OH group. The ^1H NMR spectrum of compound (2) showed two symmetric pairs of coupled of two doublets at δ 7.82 (H-2 and H-6) and at δ -7.35 (H-3 and H-5) assigning to a 1,4-disubstituted benzene ring present in compound (2). The presence of sharp one proton singlet at $\delta=9.97$ could be attributed to one aldehydic group.

So it is clear from all of these ^1H NMR signals that the aldehydic and hydroxyl groups are the substituents at C-1 and C-4, respectively, of the 1,4-disubstituted benzene ring. On this basis compound (2) was identified as 4-hydroxy benzaldehyde.



Compound (3)

Compound (4)

Compound (3) was obtained as pink gum. It appeared as a dark quenching spot on the TLC plate under UV light at 254 nm and was soluble in CH_2Cl_2 and CHCl_3 . The ^1H NMR (400 MHz, CDCl_3) spectrum of compound (3) has shown general features of substituted anthraquinone system. The ^1H NMR spectrum has shown two singlets integrating for one proton each at $\delta=7.48$ and $\delta=7.50$ indicated a typical 1,2,4,5-tetra substituted aromatic ring and the position of the two singlet at $\delta=7.48$ and $\delta=7.50$ could be attributed to the two protons of C-1 and C-4 respectively. The two doublets ($J=6.6$ Hz), each integrating for one proton, centered at $\delta=8.07$ and $\delta=8.15$ could be assigned to the two *o*-coupled aromatic protons, located at C-7 and C-8 of the anthraquinone system. A one proton

singlet at $\delta=8.54$ was attributed for another aromatic proton at C-5. A singlet integrating for three protons at $\delta=2.62$ indicated the presence of a methyl group. The relatively deshielded nature of this methyl group indicated that it might be the part of an acetyl group attached to the aromatic ring at C-6. On these NMR data compound (3) could be proposed as 6-acetyl-2,3-dihydroxy anthracene-9,10-dione.

Compound (4) was obtained as gummy material. It appeared as a dark quenching spot on the TLC plate under UV light at 254 nm and was soluble in CH_2Cl_2 and CHCl_3 . The ^1H NMR spectra of compound (4) was given in the experimental part. On this basis of the NMR spectroscopic data, the compound (4) was identified as 2-hydroxy-3-methoxy-2,9-dimethyl-1,2-dihydrotetraphene-7,12-dione.

Acknowledgement

The authors wish to thank the Bangladesh Council of Scientific and Industrial Research under Ministry of Science and Information & Communication Technology, Government of the Peoples' Republic of Bangladesh.

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