Phytochemical Studies on the Leaves of Xylia dolabriformis

Fakir Shahidullah Tareq^{1,2}, Md. Hossain Sohrab¹, A. M. Sarwar Uddin Chowhdury², Farhana Afroz¹, M. Al-Mansur¹ and Choudhury M. Hasan³

¹Bangladesh Council of Scientific and Industrial Research, Dhaka-1205, Bangladesh ²Department of Applied Chemistry & Chemical Engineering, University of Dhaka, Dhaka-1000, Bangladesh ³Department of Pharmaceutical Chemistry, Faculty of Pharmacy, University of Dhaka, Dhaka-1000, Bangladesh

Three compounds were isolated from the petroleum ether extract of the dried leaves of Xylia dolabriformis. Based on the spectral evidence, their structures were determined to be taraxerol (1), taraxerone (2) and stigmasterol. This is the first report of occurrence of these compounds from X. dolabriformis.

Xylia dolabriformis Benth locally known as Lohakath is a big tree belonging to the family Leguminosae. The plant is widely distributed in the coastal forest and hill tracts of Bangladesh, India and Burma^{1, 2} and has been used in folk medicine to treat gonorrhea, diarrhea, vomiting and ulcers³. Previous investigations resulted in the isolation of tannins, oil and triterpene³. As part of our chemical investigation on Bangladeshi medicinal plants, we report, herein, the isolation and structure elucidation of two pentacyclic triterpenes taraxerol (1), taraxerone (2) and a steroid, stigmasterol from the petroleum ether extract. The structures of these compounds were determined by spectral and chemical analyses. The presence of triterpenoid skeleton in both of compounds 1 & 2 was suggested by the violet vanillin sulfuric acid test⁴.

Tel: +88-02-9661920-73; Fax: +88-02-8615583

Correspondence to: Choudhury Mahmood Hasan E-mail: cmhasan@gmail.com

The ¹H-NMR spectra were recorded using a Bruker AMX-400 (400 MHz in deuterated chloroform and the chemical shifts were referenced to the residual nondeuterated solvent signal. Column chromatography (CC) was performed on Kieselgel 60, (70-230 mesh, Merck). Precoated silica gel (60 PF₂₅) plates were used for analytical purposes. The leaves of X. dolabriformis were collected from Savar, Dhaka during March 2008 and identified by Ms. Bushra Khan, Senior Scientific Officer, Bangladesh National Herbarium. A voucher specimen has been deposited in Bangladesh National Herbarium (DACB accession no. - 32761) Dhaka, Bangladesh. The dried and powdered leaf (725 gm) of X. dolabriformis was extracted with petroleum ether, dicholomethane and methanol sequentially using a Soxhlet apparatus. The concentrated crude petroleum ether extract (4.05 gm) was subjected to column chromatography by using gradients of petroleum ether/ethyl acetate, then ethyl acetate, followed by a gradient of ethyl acetate/methanol and finally with methanol to afford a total of 35 fractions (each 200 mL). Fractions 21, 15 and 29 upon repeated washing with petroleum ether gave compounds 1 (10 mg), 2 (15 mg) and stigmasterol (15 mg), respectively as amorphous solid.

Taraxerol (1): Colorless crystal; ¹H-NMR (400 MHz, CDCl₃): δ 5.52 (1H, dd, J= 8.0, 3.2 Hz, H-15), δ 3.19 (1H, dd, J= 9.2 Hz, H-3), 1.08 (3H, s, Me-8), 0.97 (3H, s, Me-10), 0.94 (3H, s, Me-20α), 0.92 (3H, 172 Tareq et al.

s, Me-4 β), 0.90 (6H, s, Me-4 α , Me-20 β), 0.82 (3H, s, Me-13), 0.80 (3H, s, Me-17). Taraxerone (2): White mass; 1 H-NMR (400 MHz, CDCl₃): δ 5.55 (1H dd, J= 8.0, 3.2 Hz, H-15), 2.56 (m, H-2a), 2.32 (m, H-2b), 1.13 (3H, s, CH₃), 1.08 (3H, s, CH₃), 1.07 (3H, s, CH₃), 1.06 (3H, s, CH₃), 0.95 (3H, s, CH₃), 0.91 (3H, s, CH₃), 0.90 (3H, s, CH₃), 0.82 (3H, s, CH₃). Stigmasterol: Colorless needles; 1 H-NMR (400 MHz, CDCl₃) spectral data was identical to published values. 5

The ¹H-NMR spectrum of compound **1** showed eight three proton singlets at δ 0.80, 0.82, 0.90, 0.90, 0.92, 0.94, 0.97, and 1.08. These were attributed to the methyl group protons at C-17, C-13, C-4α, Me-4β, C-20α, C-10, C-8 respectively. The double doublet (J=3.2 Hz, 8.0 Hz) centered at δ 5.52 attributable to the olefinic proton at C-15. The broad doublet (J= 9.2 Hz) centered at δ 3.19 could be assigned to the oxymethine proton at C-3. The large coupling of this proton (H-3) with the vicinal methylene protons suggested a β (beta) orientation of the hydroxyl group at C-3. The above ¹H-NMR signals suggested the presence of a typical pentacyclic triterpene skeleton. This was identified as B-taraxerol by comparison these data with those reported previously for taraxerol.^{4, 6}

The structure of compound 2 was elucidated by direct comparison of its spectral data with that of compound 1. Although, the ¹H-NMR spectral data of

2 was in close agreement to that of **1**, except the resonance at δ 3.19 for the oxymethine proton at C-3 in the spectrum of **1**, was absent in **2**. This suggested that the hydroxyl group at C-3 in compound 1 was replaced by a carbonyl function. On the basis of the above spectral features, compound **2** was characterized as taraxerone, the identity of which was further substantiated by comparison of its spectroscopic data with published values. The third compound was characterized as stigmasterol by comparison of its ¹H-NMR spectral data with published values ⁵ as well as by co-TLC with an authentic sample.

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