Available online at www.joac.info

ISSN: 2278-1862

Journal of Applicable Chemistry

2013, 2 (2):254-256

(International Peer Reviewed Journal)

Chemical constituents from fruits of *Sapindus mukorossi*

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Received on 18th February and finalized on 22nd February 2013.

ABSTRACT

A novel glycoside identified as 6, 7-dihydroxy-2-methylnapthalene-9,10-dimethylethane-10-O-[3'-acetyl-α-L-rhamnopyranosyl] have been isolated from the fruits of Sapindus mukorossi. The structures of these saponins were characterized by means of chemical and spectral methods including advanced 2D NMR studies.

Keywords: Novel glycoside, Isolation from the fruits of Sapindus mukorossi, Characterization of saponins.

INTRODUCTION

Sapindus mukorossi Gaertn, of family sapindaceae is well known as soap nut tree, which distributed in tropical and subtropical regions of Asia. Fruits of sapindus mukorossi are popular ingredient in ayurvedic shampoos and cleansers. The plant is an important remedy for relieving cough, detoxification, emetic, contraceptive, treatment of excessive salivation, epilepsy and chlorosis [1,2,3]. Previous studies on the plants of this genus led to isolation and identification of triterpenoids, saponins [4,5,6,7], fatty acids [8] and flavonoids [9], from the pericarp, stem and fruit of the plant. This article deals with isolation and structure elucidation of a novel acetylated triterpene bisdesmoside saponin compound 1 (Figure 1) with the help of modern spectroscopic methods.

MATERIALS AND METHODS

General Experimental Procedure: Melting point was recorded on the Perfit melting point apparatus. UV spectrum was measured on Perkin-Elmer Lamba-25 spectrometer in methanol. IR spectra were recorded on Perkin-Elmer, spectrum RX I FT-IR spectrometer (KBr discs). NMR spectra were obtained on JEOL NMR spectrometer (300 MHz for ^{1}H and 125 MHz for ^{13}C, DMSO-d^{6} as solvent, TMS as internal standard). LCMS spectra were recorded on Finnigan MAT spectrophotometer (CA, USA, Excalibur ver-2 software). Column chromatography was performed on silica gel (Merck 60-120 mesh, 15x100 cm). Thin layer chromatography was carried out on silica gel (Merck 10-40 μ) percolated plates were visualized by spraying with 7% H_2SO_4 as a universal spray reagent along with other specific reagents for particular class of natural products.

Plant material: Pericarps of *S. mukorossi* were collected from Durgadhar District Chamoli and identified by Prof. R. D. Gaur, Department of Botany, H.N.B. Garhwal University Srinagar.

Extraction and Isolation: Shade dried and powdered fruit of *S.mukorossi* was extracted three times with 95% EtOH at 35^0 C on a heating mantle. The extraction mixture was filtered off and concentrated under reduced pressure to yield black brown residue. This residue was fractionated with EtOAc (repeatedly 3-4 times) yielded EtOAc soluble and insoluble fractions. EtOAc soluble layer after evaporation of solvent under reduced pressure afforded of crude extract. This crude extract (preadsorbed with silica gel) were column chromatographed using silica gel (Merck 60-120 mesh, 500g) using solvent gradient system in order of increasing polarity e.g. chloroform : methanol (95: 5 → 70 : 30). The fractions were obtained were collected every 50 ml. The $CHCl_3$: MeOH (88: 12) eluent afforded compound 1.

Compound 1: It was crystallized as white amorphous powder from $CHCl_3$:MeOH, mp: 288-290°C. The LCMS of compound furnished a peak at m/z438 M^+, which correspond the molecular formula $C_{23}H_{34}O_8$ other fragments ion peak were observed at 380, 234, 214, 202 and the most abundant ion peak observed at m/z 234. Its IR spectrum exhibited characteristic absorption at 3450 (C=O) and 1731 (OH) cm^{-1}. ^{1}H NMR signal of compound displayed ortho, meta-substituted benzene ring B (δ6.84s) and two hydroxyl overlapped peak at (δ6.24s). ^{1}H spectrum showed signals characteristic for three singlet methyls at δ1.03, 1.04, 1.21 and 1.16. ^{1}H-NMR spectrum indicated the presence of one aromatic proton signal and one glycoside anomeric proton signals which suggested that the compound was similar to sesquiterpene glycoside. ^{13}C NMR spectrum showed 23 carbons which were identifies as five methyl, two methylene, ten methine and three quaternary carbon atoms. [Zhang et al., 1998; Stipanovic et al., 2006]. This fact was further confirmed by acidic hydrolysis of the compound which afforded an aglycone together with a mixture of sugars, identified as L-rhamnose by compared with authentic samples. The ^{1}H- and ^{13}C-NMR spectrum also confirmed the presence of L-rhamnose. The position of L-rhamnose was determined to be at C-10, based on the HMBC correlation. The configuration the sugar moiety was determined to be α by the presence of an anomeric proton at *δ* 5.17s (H-1') and an anomeric carbon at *δ* 93.34 (C-1') in the ^{1}H- and ^{13}C-NMR spectra respectively. The ^{1}H NMR value at δ 2.45 and ^{13}C NMR signals at δ 169.28 (-COO) and 21.05 (Me) indicated to the presence of acetoxyl group attached to rhamnose ring was corroborated by HMBC correlation. The long range ^{1}H- ^{13}C HMBC experiment suggested that acetoxyl group from the correlation between δ_H 4.37 (Rha-3')/δc-169.28 and rhamnose group attached at C-10. Hence on the basis of spectral evidences and comparison with (IR, NMR and MS) it has been identified as 6, 7-dihydroxy-2-methylnapthalene-9,10-dimethylethane-10-*O*-[3'-acetyl-α-L-rhamnopyranosyl] (fig.1)

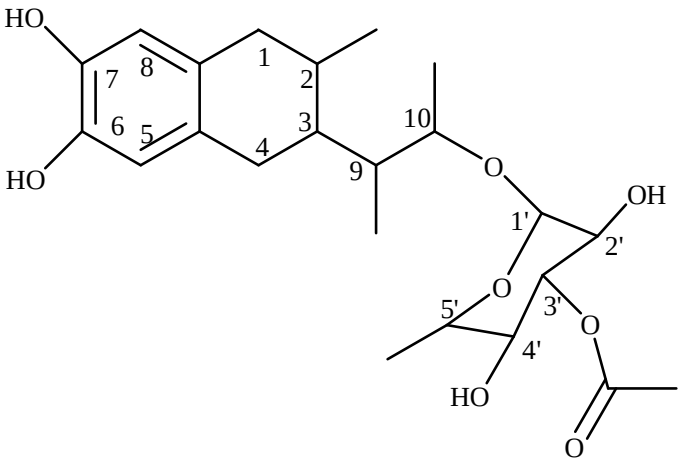
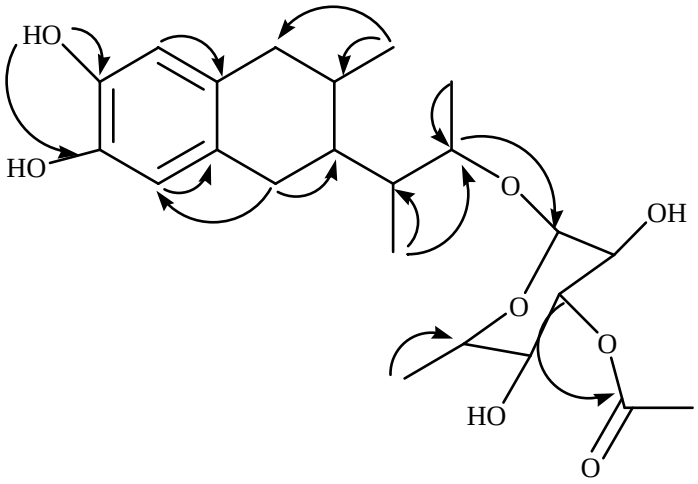


Fig. 1



^{1}H-^{13}C HMBC correlation of Compound 1

APPLICATIONS

Isolated compound have marked antibacterial activity against gram positive and negative fungal strains.

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