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CHEMICAL CONSTITUENTS FROM CENTIPEDA MINIMA

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INTRODUCTION

Centipeda minma (Family: Asteraceae) is a annual herbaceous plant found in moist places throughout the plains of India and Ceylon.[1] Centipeda is well known for its medicinal properties[2-3] Herein we report the isolation and characterization of β -sitosterol and β - sitosteryl- β -D-glucoside from the whole plant of Centipeda minima Experimental Dried powdered plant of *C.minima* (4.5 kg) was extracted with MeOH in a Soxhlet apparatus which on removal of solvent gave a brown mass (90 g). The MeOH extract was chromatographed over SiO_2 gel column eluting with solvents of increasing polarity. The fractions collected from C_6H_5-$CHCl_3$ (1:1), $CHCl_3$, $CHCl_3$-MeOH (50 : 1) and (9: 1) on concentration followed by crystallization from MeOH gave respectively β-sitosterol (**1**) as colourless needles (68 mg), 129-130 °C (lit. 133-135 °C); and β -sitosteryl- β D-glucoside (**2**)as crystalline solid (48 mg), 264–266 °C (lit. 257–258 °C).

Compound 1 [β -sitosterol] IR (KBr) νmax (cm^{-1}): 3300 (-OH), 2980 (-CH), 1640 (C=C); 'H NMR (300 MHz, CDC13) δ: 5.35 (1H, m, H-6), 3.53 (1H, m, H-3), 2.49(1H,m,H-1- β),2.48(1H,m,H-4- α),2.31(1H,m. H-4- β), 2.25 (1H, m, H-7- β), 1.91 (1H, m, H-7- α), 1.90 (1H,m,H-2- α),1.88(1H,m,H-2 β) 1.87 (1H, m, H 11- α), 1.87 (1H, m, H-12- β). 1.83 (1H, m, H-22- α), 1.81(1H, m, H-23- β), 1.75(1H, m, H-15- β), 1.73(1H, m, H-25), 1.72 (1H, m, H- 20), 1.66 (1H, m, H-16- α). 1.50 (1H, m, H-17), 1.45 (1H, m, H-8), 1.44 (1H, m, H 11- β), 1.42 (1H, m, H-28- β), 1.36 (1H, m, H-16- β). 1.35 (1H, m, H-1- α), 1.31 (1H, m, H-1 α), 1.30 (1H, m. H-24), 1.23 (1H, m, H-12- α), 1.21 (1H, m, H-9), 1.20 (1H,m,H-23- α),1,15(3H,s,CH3 -19),1.11(1H,m, H-28- α), 1.09 (1H, m, H-22- β), 1.06 (1H, m, H-15-α). 0.95 (3H, d, J 6.7 Hz, CH3-21), 0.92 (3H, t, J 7.3 Hz, CH3-29), 0.85 (3H, d, J 6.5 Hz, CH3- 27), 0.81 (3H, d. J 6.5 Hz, CH_2 -26), 0.67 (3H, s, CH3-18); 13C NMR (125 MHz, CDC13) δ: 141.0 (C-5), 122.2 (C-6), 71.9 (C-3),57.0(C-14),56.4(C-17),50.4(C-9),46.5(C- 24), 42.4 (C- 4), 42.1 (C-13), 39.5 (C-12), 37.5 (C-1). 36.9(C - 10), 36.5(C - 20), 34.2(C - 22), 32.1(C - 8), 32.0 (C-7), 31.9 (C-2), 29.5(C-25),28.5(C-16),26.5(C- 23), 24.3 (C-15), 23.5(C - 28), 21.2(C - 11), 20.0(C - 26) 19.6(C - 19), 19.5(C - 27), 19.0(C - 21), 12.3(C - 18), 12.1 (C-29); MS: m/z 414 ([M]+, C29H50O), 396, 381, 329, 303, 275, 255, 213, 159, 145, 107, 57, 55.

Compound 2 (β -sitosteryl- β -D-glucoside]: IR (KBr) νmax (cm^{-1}): 3380 (-OH), 2930 (-CH), 1635 (C=C); 'H NMR (300 MHz, CD3OD) δ: 5.34 (1H, m, H-6), 4.48 (1H, dd, J 11.7, 2.0 Hz, H-6'- β). 4.46 (1H, d, J7.6 Hz. H-1'), 4.22 (1H, dd, J 11.6, 5.0 Hz, H-6'- α), 4.21 (IH, m, H- 3'), 4.19 (1H, m, H-4'), 3.98 (1H, t, J 7.8 Hz. H 2'). 3.91 (1H, m, H-5'), 3.53 (1H, m, H-3), 2:52 (1H, m, H-1- β), 2.50(IH, m, H - 4 α), 2.35(IH, m, H - β) 2.10 (1H, m, H-7- β), 1.97 (1H, m, H-7- α), 1.92 (1H, m. H-2- α), 1.90 (1H,m,H-2- β),1,91(H,m,H-11- α), 1.82 (1H, m, H-12- β), 1.81 (1H, m, H-22- α), 1.80 (IH, m. H-23- β), 1.78 (1H, m, H-15- β). 1.77 (1H, m, H-25). 1.72 (1H,m,H-20),1.61(1H,m,H-16- α). 1.51 (IH, m, H-17), 1.50 (1H, m, H-8), 1.45 (1H, m, H-11- β). 1:42 (1H, m, H-28- β), 1.39 (1H, m, H-16- β), 1.31 (1H. m, H-1- α), 1.26 (IH, m, H-14), 1.24 (1H, m, H-24). 1.21 (1H, m, H-12-0), 1.20 (1H, m, H-9), 1.19 (1H, m. H-23- α), 1.17 (1H, m, H-28- α). 1.15 (3H, s, CH3-19). 1.12 (1H, m, H-22- β), 1.06 (1H, m, H-15- α), 0.89 (3H. d. J 6.6 Hz, CH3-21), 0.87 (3H. s. CH3-19), 0.81 (3H. t. J7.4 Hz, CH3-29), 0.78 (3H, d, J 6.7 Hz, CH3-26), 0.76 (3H, d, J 6.8 Hz, CH3-27), 0.66 (3H, s, CH3-18); 13C NMR (125 MHz, CD3OD) δ: 142.1 (C-5). 122.9 (C-6). 102.3 (C-1'), 79.8 (C-3'), 79.7 (C-3), 78.5 (C-5'). 76.1 (C-2'), 72.9 (C-4'), 63.1 (C-6'), 57.9 (C-14), 57.5 (C 17), 51.0 (C-9), 47.3 (C-24), 42.8 (C-13), 41.1 (C-4), 39.8(C-12),38.5(C-1),38.1(C-10),37.2(C-20),35.1 (C- 22), 33.0(C - 8), 32.3 3(C - 7), 33.1(C - 2), 30.4(C - 25) , 29.6(C - 16), 27.5(C - 23), 25. 4(C - 15), 24.8(C - 28), 22.9 (C-11),21.4(C-26),20.4(C-19). 20.3 (C-27). 20.1 (C-21), 13.7 (C-18), 13.5 (C-29); MS m/z 414 ([M-163+H]+), 399, 398, 397, 396, 382, 145 (Found: C. 72.7: H. 10.2: O. 16.7, C35H60O6requires C. 72.9;H- 10.4; O, 16.6%); MW 576.80. The glycoside (30 mg), was dissolved in MeOH and refluxed with 2 N HCI for 6 h. The reaction mixture was poured in H_2 O and extracted with CHCI3. The CHCI3, extract on evaporation furnished a white mass which was crystallised from MeOH. It was identified as

β -sitosterol by direct comparison with authentic sample (m.m.p. and co-TLC). The sugar in the hydrolysate was identified as D-glucose by co-PC with authentic sample.

The spectral data of all the compounds (**1**&**2**) matched well with the reported data.[5-9] Finally, the structures of all the above compounds were confirmed by direct comparison with authentic samples (m.m.p., co-TLC and superimposable IR).

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