Asian Journal of Chemistry Vol. 21, No. 2 (2009), 1639-1641

NOTE

Flavonoids of *Memecylon umbellatum* (Burm.)

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The flavonoids quercetin, rutin and apigenin has been isolated from the roots of *Memecylon umbellatum* and their structure were established by spectral analysis and direct comparison with authentic samples.

Key Words: *Memecylon umbellatum*, Iron wood tree, Flavonoids.

Memecylon umbellatum is a shrub or a small tree belonging to the family Melastomataceae. It is distributed in the coastal region of Deccan Peninsula, eastern part of India and Andaman Islands[1,2]. From the literature survey, it was revealed that many phytoconstitutents like triterpinoids, sterols and steroidal glycosides were isolated and characterized from the plant[3]. Various pharmacological activities like antidiabetic, antiviral, antibacterial activities were studied[4-7]. The present work deals with the isolation of further constituents from the roots of *Memecylon umbellatum*.

The roots of *Memecylon umbellatum* were collected from Mangalore, Dakshina Karnataka, India during October 2006. It was authenticated by Dr. Gopalakrishna Bhatt, Department of Botany, Poornaprajna College, Udupi. A voucher specimen no. 102a is deposited in the Herbarium of N.G.S.M. Institute of Pharmaceutical Sciences, Mangalore.

The roots were dried in shade. The dried roots were powdered (5 kg) and soaked in ethanol (95 %) and kept aside for 4 days, the ethanolic layer was decanted off. The process was repeated for 4 times. The solvent from the total extract was distilled off and concentrate was evaporated on water bath to a syrupy consistency and evaporated to dryness (660 g).

The ethanolic extract (600 g) was further fractionated with petroleum ether (60-80 °C), chloroform, butanol and ethyl acetate to give petroleum ether extract (60-80 °C) (45 g), chloroform extract (48 g), butanol extract (57 g) and ethyl acetate extract (40 g).

The ethyl acetate extract (35 g) was adsorbed on silica gel and chromatographed over silica gel column, eluting with solvents of increasing polarity. The elutes collected were monitored by TLC techniques and similar eluants were mixed together. The eluents collected from $CHCl_3$, $CHCl_3$:MeOH (90:10, 80:20) yielded compounds A (48 mg), B (42 mg) and C (38 mg) by crystallization from methanol.

Compound A (quercetin): Compound A, crystallized from methanol as light yellow needles, m.p. 312 ºC, showed positive Shinoda test for flavonoids. It exhibited UV: λ_{max}^{MeOH} 255, 269, sh, 301 sh, 370 nm, IR (KBr, ν_{max}, cm^{-1}): 3250 (OH), 1640 and 1610 (Conjugated Carbonyl), 2920 (C-H of CH_3 *str.*). Its molecular formula was found to be $C_{45}H_{10}O_7$ (M^+, 302) and other peaks were appeared at 301, 274, 273, 245, 229, 228, 153, 137, 128 and 69. ^{1}H NMR: (DMSO-d_6, δ): 6.16 (1H, d, J = 2 Hz), 6.40 (1H, dd, J = 2 Hz), 6.85 (1 H, dd, J = 9 H), 7.47 (1H, dd, J = 2 Hz), 7.62 (1 H dd, J = 2 Hz and 9 Hz), 9.60 (1H, s) and 12.48 (1 H s). ^{13}C NMR (DMSO-d_6): 145.05 (C-2), 135.72 (C-3), 175.83 (C-4), 160.70 (C-5) 98.34 (C-6)163.8 (C-7), 93.34 (C-8), 156.13(C-9), 103.01 (C-10), 121.96 (C-1'), 115.0 (C-2'), 145.05 (C-3'), 147.69 (C-4'), 115.6 (C-5'), 119.9 (C-6'). It was found to be identical with authentic quercetin (mixed m.p., Co-TLC and super imposable IR).

Compound B (rutin): Compound B, crystallized from methanol as yellow crystals (80 mg), m.p. 188-189 ºC showed positive Shinoda test for flavonoids. UV: λ_{max}^{MeOH} 259, 266, 299 sh, 363 nm, $\lambda_{max}^{(MeOH + MeOAc)}$ 272, 327, 415, $\lambda_{max}^{(MeOH + AlCl_3)}$ 275, 303, 433. $\lambda_{max}^{(MeOH + HCl)}$ 271, 325 393, $\lambda_{max}^{(boric\ acid)}$ 262, 298, 387. IR: (KBr, ν_{max}, cm^{-1}): 3300 (OH), 2920 (C-H *str.*), 1660 (C=0) conjugated carbonyl, 1620 (C=C), 1360 (C-O-C). ^{1}H NMR: (TMS, δ); 0.8 (d, 3H, ramnosyl methyl), 3.3 (m, 10H, ramnosyl glucosyl protons), 4.2 (s, 1H, ramnosyl H_1), 5.88 (d, 1H, glucosyl H_1), 6.2 (d, 1H, J = 2.5 Hz, H_6), 6.36 (d, 1H, J = 2.5 Hz, H_8), 6.9 (d, 1H, J = 9Hz, H_5), 7.4 (m, 2H, H_2', H_6' super imposable); ^{13}C NMR (DMSO-d_6): 156.37 (C-2), 133.26 (C-3), 177.327 (C-4), 161.18 (C-5), 98.61 (C-6), 164.00 (C-7), 93.52 (C-8), 156.55 (C-9), 103.92 (C-10), 121.53 (C-1'), 115.17 (C-2'), 144.69 (C-3'), 148.36 (C-4'), 116.22 (C-5), 121.13 (C-6'), 101.14 (C-1"), 74.03 (C-2"), 75.87 (C-3"), 69.96 (C-4"), 76.41 (C-5"), 66.643 (C-6"), 100.69 (C-1'"), 70.32 (C-2'"), 70.52 (C-3'"), 71.80 (C-4'"), 68.18 (C-5'"), 17.67 (C-6'"). It's molecular formula was found to be $C_{27}H_{30}O_{16}$ (M^+ 611) and other peaks appeared at 532, 465, 275, 194, 127. It was finally identified as rutin from direct comparison with authentic sample (mixed m.p., Co-TLC and super imposable IR).

Compound C (apigenin): Compound C, crystallized from methanol as yellow crystals, m.p. 345 ºC gave showed positive Shinoda test for flavonoids.UV: λ_{max}, λ_{max}^{MeOH} 268, 297 sh, 334 nm, $\lambda_{max}^{(MeOH + AlCl_3)}$ 278, 302, 344, 382 nm, $\lambda_{max}^{(MeOH + NaOCH_3)}$ 276, 326, 394 nm, $\lambda_{max}^{(MeOH + AlCl_3 + HCl)}$ 278, 300, 340, 378 nm. $\lambda_{max}^{(MeOH + NaOAc)}$ 276, 300, 380 nm, $\lambda_{max}^{(MeOH + NaOAc + boric\ acid}$ 273, 303 sh, 342 nm. IR: (KBr, ν_{max}, cm^{-1}): 3326 (br, OH *str.*) 3092 (Ar C-H *str.*, 1604 (C-C *str.*) 1653 (C=O str.) ^{1}H NMR (DMSO-d_6) δ: 10.84 (s, 1H, OH) 10.37 (s, 1H, OH) 12.93 (s,1H OH) 7.97 (d, 2H, H-2^1, H-6^1), 6.93 (d, 2H, H-3^1, H-5^1), 6.8 (d, 1H, H-8), 6.5 (d, 1H, H-6), 6.2 (s, 1H, H-3); ^{13}C NMR (DMSO-d_6): 182.110 (C-4), 164.88 (C-2), 164.101 (C-7), 161.818 (C-5), 161.530 (C-41), 157.671 (C-9), 128.835 (C-61,C-21), 121.541 (C-11), 116.318 (C-31, C-51), 103.208 (C-10), 99.195 (C-6), 94.321 (C-8), 103.510 (C-3). Its molecular formula was found to be $C_{15}H_{10}O_5$ (M^+, 270), The other peaks appeared at 242, 213, 153, 121, 96, 78 and 69. It was finally identified as apegenin from direct comparison with authentic sample. (Mixed m.p., Co-TLC and super imposable IR).

Chromatographic resolution of ethyl acetate extract furnished 3 compounds A, B and C. These were designated as quercetine, rutin and apigenin by detailed UV, IR, ^{1}H NMR, ^{13}C NMR and mass spectra. All the three compounds are being reported for the first time from the roots of *Memecylon umbellatum*.

ACKNOWLEDGEMENTS

The authors are thankful to Nitte Education Trust for providing necessary facilities and are also thankful to regional sophisticated instrumentation centre, CDRI, Lucknow, India and sophisticated instrumentation centre PTU Chandigarh, India for providing the spectras.

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(*Received*: 4 March 2008; *Accepted*: 20 October 2008) AJC-6963