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PENTACYCLIC TRITERPENES FROM THE STEM BARK OF *MIMUSOPS ELENGI* L.

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Abstract: A new farnane-type pentacyclic triterpene, farnan-2-one-3β-ol (mimusopfarnanol), was isolated from the stem bark of *Mimusops elengi* L. along with the known triterpenoids, farnan-3-one, olean-18-en-2-one-3-ol and lup-20 (29)-en-3β-ol and their structures have been characterized on the basis of spectral data analyses and chemical reactions.

Keywords: *Mimusops elengi*, stem bark, pentacyclic triterpene, Sapotaceae

Mimusops elengi L., family Sapotaceae, is a glabrous tree found in western Peninsula of India (1). Its bark is acrid, sweet, cardiotonic, alexipharmic, astringent and used to cure biliousness, diseases of the gums and teeth (2). It exhibited antimicrobial, anti-ulcer, hypotensive, anti-HIV and spasmolytic activities (3-7). Ursolic acid, betulinic acid, fatty acid ester of α-spinasterol (8), 3β,6β,19α, 23-tetrahydroxy-urs-12-ene, 1β-hydroxy-3β-hexanoyl-lup-20(29)-ene-23,28-dioic acid and bassic acid have been isolated from the bark (9). This paper describes isolation and characterization of a new farnane-type triterpenoid along with three known pentacyclic triterpenoids from the stem bark of *M. elengi*.

EXPERIMENTAL

General procedure

Melting points were determined on a Perfit melting point apparatus and are uncorrected. FTIR: Jasco FT/IR-5000; UV: Lambda Bio 20 Spectrophotometer, MeOH; ^{1}H NMR (400 MHz): Advance DRY 400, Bruker Spectrospin, $CDCl_3$; ^{13}C NMR (75 MHz): Advance DRY 100, Bruker Spectrospin, $CDCl_3$ with TMS as an internal standard; MS: FAB ionization on JEOL-JMS-DX 303; CC: Silica gel (Qualigens), 60-120 mesh; TLC: Silica gel G (Qualigens). Spots were visualized by exposure to iodine vapors, UV radiation and by spraying reagents.

Plant material

The bark of *M. elengi* was procured from the Khari Baoli local market of Delhi and identified by Dr H.B. Singh Taxonomist, NISCAIR, CSIR, New Delhi. A voucher specimen is deposited in the herbarium of the Phytochemical Research Laboratory, Faculty of Pharmacy, Jamia Hamdard, New Delhi.

Extraction

The bark of *M. elengi* (3 kg) was dried in an oven at 45°C for 2-3 days and coarsely powdered. The ground bark was extracted with ethanol in a Soxhlet apparatus. The ethanol extract was concentrated under reduced pressure to yield a dark brown viscous mass (120 g, 4.0%) which was dissolved in a minimum amount of methanol and adsorbed on silica gel (60-120 mesh) for preparation of slurry. The air-dried slurry was chromatographed over the silica gel column packed in petroleum ether (60-80°C). The column was eluted with petroleum ether (60-80°C), chloroform and methanol in their various combinations in the order of increasing polarity.

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Isolation and characterization of 1

Elution of the column with petroleum ether-chloroform (1:1, v/v) mixture furnished colorless crystals of **1**, recrystallized from MeOH, yield 500 mg (0.016%); R_f 0.81 (petroleum ether : toluene : ethyl acetate; 10:5:3, v/v/v), m.p. 238-240°C. UV λ_{max} (MeOH): 208 nm (log ε 5.2). IR ν_{max} (KBr, cm^{-1}): 3450, 2929, 2850, 1716, 1470, 1388, 1225, 1130, 1080, 1020. ^{1}H NMR: Table 1. ^{13}C NMR: Table 1. +ve FAB-MS [m/z] (rel. int.): 442 $[M]^+$ ($C_{30}H_{50}O_2$) (1.7), 427 (27.6), 424 (5.3), 329 (5.7), 306 (4.8), 291 (4.8), 288 (24.7), 274 (8.3), 272 (4.1), 260 (3.9), 258 (4.3), 220 (5.1), 218 (4.9), 204 (9.1), 191 (4.7), 168 (4.8), 154 (100), 153 (12.3), 139 (17.8), 138 (26.8), 137 (48.3), 136 (55.7), 124 (10.3), 123 (10.5), 122 (10.4), 114 (6.1), 109 (10.8), 108 (21.6), 95 (20.1).

Compound 2

Elution of the column with petroleum ether : chloroform (1:1, v/v) afforded light yellow crystals of **2**, recrystallization from methanol, yield 400 mg (0.013%), R_f 0.76 (petroleum ether : toluene : ethyl acetate; 10:5:3, v/v/v), m.p. 218-220°C. UV λ_{max} (MeOH): 208 nm (log ε 5.6). IR λ_{max} (KBr, cm^{-1}): 2930, 2855, 1461, 1389, 1262, 1187, 1079, 970, 800. ^{1}H NMR: Table 1. ^{13}C NMR: Table 1. +ve FAB-MS [m/z] (rel. int.): 426 $[M]^+$ ($C_{30}H_{50}O$) (80.1), 411 (11.3), 274 (12.6), 272 (11.8), 245 (10.1), 206 (45.3), 166 (13.7), 152 (21.8), 138 (37.2), 124 (41.9), 111 (13.6).

Compound 3

Elution of the column with chloroform : methanol (99:1, v/v) gave colorless amorphous powder of **3**, recrystallized from methanol, yield 240 mg (0.008%), R_f 0.57 (toluene : ethyl formate : formic acid; 10:10:3, v/v/v), m.p. 118-120°C.
UV λ_{max} (MeOH): 203 nm (log ε 5.1). IR λ_{max} (KBr, cm^{-1}): 3410, 2931, 2845, 1716, 1635, 1463, 1383, 1063, 958, 799. ^{1}H NMR: Table 1. ^{13}C NMR: Table 1. +ve FAB-MS [m/z] (rel. int.): 442 $[M]^+$ ($C_{30}H_{50}O_2$) (67.6), 413 (67.4), 411(51.2), 396 (59.2), 393 (15.6), 381 (11.2), 341 (8.1), 327 (4.2), 308 (6.3), 290 (5.3), 272 (14.8), 271 (15.1), 256 (13.6), 218 (19.8), 204 (27.6), 176 (24.3), 190 (18.1), 182 (11.6), 168 (22.8), 164 (23.1), 155 (26.8), 154 (47.1), 150 (21.6), 146 (26.7), 141 (13.7), 137 (42.3), 136 (42.1), 132 (41.8), 131 (24.8), 123 (37.8), 121 (43.6), 118 (43.1), 117 (24.8), 112 (14.8), 95 (100), 94 (59.8).

Compound 4

Elution of the column with chloroform : methanol (19:1, v/v) yielded colorless amorphous powder of **4**, recrystallized from acetone to form needle shaped crystals, yield 130 mg (0.004%), R_f 0.54 (toluene : ethyl formate : formic acid; 10:10:3, v/v/v); m.p. 213-215°C, $[\alpha]_D^{20}$: 27.1° ($CHCl_3$). UV λ_{max} (MeOH): 218 nm (log ε 5.1). IR ν_{max} (KBr, cm^{-1}): 3451, 2931, 2851, 1460, 1376, 1225, 1044. ^{1}H NMR ($CDCl_3$, δ ppm): 4.73 (brs, 1H, H2-29 a), 4.60 (brs, 1H, H2-29 b), 3.18 (dd, *J* = 5.5, 9.3 Hz, 1H, H-3 α), 2.99 (brs, 1H, H-19), 1.68 (brs, 3H, Me-30), 1.36 (brs, 3H, Me-23), 1.25 (brs, 3H, Me-25), 0.92 (brs, 3H, Me-24), 0.89 (brs, 3H, Me-26), 0.84 (brs, 3H, Me-28), 0.75 (brs, 3H, Me-27). ^{13}C NMR ($CDCl_3$, ä ppm): 38.70 (C-1), 50.49 (C-2), 79.01 (C-3), 46.88 (C-4), 55.32 (C-5), 18.27 (C-6), 29.66 (C-7), 38.37 (C-8), 49.24 (C-9), 37.04 (C-10), 23.64 (C-11), 24.10 (C-12), 32.13 (C-13), 42.90 (C-14), 25.47 (C-15), 30.55 (C-16), 40.69 (C-17), 49.68 (C-18), 48.36 (C-19), 144.84 (C-20), 27.34 (C-21), 34.30 (C-22), 27.96 (C-23), 19.34 (C-24), 16.08(C-25), 15.32 (C-26), 14.67 (C-27), 20.82 (C-28), 109.66 (C-29), 22.66 (C-30); +ve FAB-MS [m/z] (rel. int.): 426 $[M]^+$ ($C_{30}H_{50}O$) (22,6), 411(18,4), 408 (34.2), 396 (27.8), 393 (21.0), 381 (25.7), 363 (8.9), 286 (33.4), 272 (21.7), 258 (15.3). 236(76.2), 222 (23.8), 219(100), 207(35.6), 205 (21.1), 191 (41.0), 189 (18.1, 177 (15.8), 174 (48.2), 168 (36.6), 159 (35.1), 154 (67.8), 140 (36.5), 138 (30.1), 124 (57.0), 110 (89.5).

RESULTS AND DISCUSSION

Compound **1**, named mimusopfarnanol, was obtained as colorless crystals from petroleum ether : chloroform (1:1, v/v) eluants. It responded positive to Liebermann-Burchard test for triterpenes. Its IR spectrum exhibited absorption bands for hydroxyl group (3450 cm^{-1}) and carbonyl group (1716 cm^{-1}). On the basis of mass and ^{13}C NMR spectrum its molecular weight was established as 442 consistent

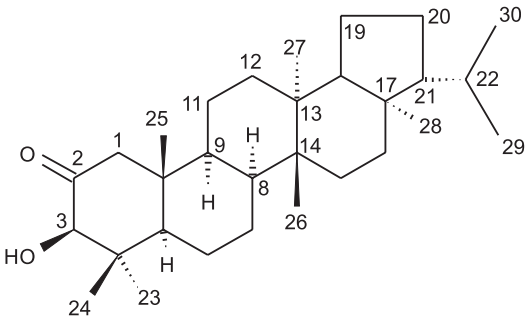

Figure 1. Chemical structure of 1

Table 1. ^{1}H and ^{13}C NMR values of compounds **1, 2** and **3**

Position	^{1}H NMR			^{13}C NMR		
	1	**2**	**3**	**1**	**2**	**3**
1	2.40 brs, 2.23 brs	2.37 m, 2.25 m	2.36 brs, 2.27 brs	41.28	39.26	39.75
2	-	2.17 brs	-	202.13	22.71	201.33
3	3.73 brs	-	3.52 brs	72.71	203.16	71.77
4	-	-	-	32.77	32.78	37.22
5	2.36 dd (3.6, 6.9)	2.34 m	2.03 brs	53.10	53.11	56.75
6	2.27 m, 2.25 m	2.30 m,	2.23 m	18.64	18.68	19.01
7	1.39 m	1.38 m, 1.28 m	1.33 m, 1.09 m	28.16	28.19	32.67
8	-	1.73 m	-	36.01	36.02	42.29
9	1.95 m	1.95 m	1.99 brs	41.56	41.19	55.17
10	-	-	-	35.60	35.35	38.89
11	1.55 m, 1.45 m	1.52 m, 1.44 m	1.55 brs, 1.18 m	18.23	18.25	20.18
12	1.76 m, 1.65 m	1.77 m, 1.67 m	1.68 m, 1.57 m	35.60	35.63	24.27
13	-	-	2.17 brs	41.28	41.30	41.50
14	-	-	-	42.79	42.80	42.58
15	1.57 m, 1.51 m	1.55 brs	1.55 brs, 1.83 m	30.50	29.71	28.22
16	1.48 m, 1.33 m	1.48 m, 1.33 m	1.48 m, 1.18 m	35.01	35.04	36.11
17	-	-	-	41.69	41.54	33.93
18	1.85 m	1.88 m	-	58.21	58.23	150.91
19	1.26 m, 1.41 m	1.41 m	5.36 brs	22.27	22.29	121.69
20	1.57 m, 1.55 m	1.55 brs	-	31.77	30.51	32.31
21	1.73 m	1.73 m	1.83 m, 1.79 m	59.47	59.48	33.53
22	1.39 m	1.38 m	1.86 m, 1.83 m	32.07	31.79	28.87
23	1.18 brs	1.25 brs	1.25 brs	32.41	32.42	31.90
24	0.72 brs	0.75 brs	0.87 brs	15.78	14.67	11.85
25	1.00 brs	1.04 brs	0.84 brs	14.64	18.25	19.78
26	0.95 brs	1.00 brs	0.83 brs	11.61	14.65	19.78
27	1.04 brs	1.10 brs	0.72 brs	17.93	17.96	22.75
28	0.92 brs	0.95 brs	0.68 brs	28.16	29.71	23.05
29	0.88 d (6.0)	0.88 d (6.1)	1.00 brs	6.81	6.83	31.96
30	0.86 d (6.0)	0.86 d (6.3)	0.92 brs	20.25	20.27	29.66

Coupling constants in Hertz are given in parentheses.

to a molecular formula $C_{30}H_{50}O_2$. It indicated six double bond equivalents, five of them were adjusted to the pentacyclic triterpenic skeleton and one in carbonyl group. The mass spectrum of **1** showed important ion peaks at m/z 329 $[C_{1,2}\text{-}C_{5,10}\text{-}C_{6,7}$ fission$]^+$, 328, 114 $[C_{1,10}\text{-}C_{4,5}$ fission$]^+$, 315 $[C_{1,2}\text{–}C_{5,10}\text{–}C_{7,8}$ fission$]^+$, 272 $[315\text{–}C_3H_7]^+$, 301 $[C_{1,10}\text{-}C_{5,10}\text{-}C_{7,8}$ fission$]^+$, 258 $[301\text{-}C_3H_7]^+$, 287 $[C_{1,10}\text{-}C_{5,10}\text{-}C_{7,8}$ fission$]^+$ suggesting the location of carbonyl and hydroxyl groups in ring A. The ion fragments arising at m/z 288, 154 $[C_{6,7}\text{–}C_{5,10}$ fission$]^+$, 274, 168 $[C_{6,7}\text{-}C_{9,10}$ fission$]^+$, 260 $[C_{6,7}\text{-}C_{8,9}$ fission$]^+$, 220 $[C_{11,12}\text{-}C_{8,14}$ fission$]^+$, 306 $[C_{12,13}\text{-}C_{8,14}$ fission$]^+$, 291 $[306\text{-}H_2O]^+$, 177 $[220\text{-}COCH_3]^+$, 152 $[C_{13,18}\text{-}C_{14,15}$ fission$]^+$, 109 $[152\text{–}COCH_3]^+$, 138 $[C_{13,18}\text{-}C_{15,16}$ fission$]^+$, 95 $[138\text{-}COCH_3]^+$,124 $[C_{13,18}\text{-}C_{15,16}$ fission$]^+$ and 109 $[124\text{-}COCH_3]^+$ indicated saturated nature of rings B, C and D. The ^{1}H NMR spectrum of **1** showed one-proton broad signals at 3.73 ppm assigned to carbinol H-3. Six three-proton broad signals at 1.18, 0.72, 1.00, 0.95, 1.04 and 0.92 ppm were ascribed to tertiary C-23, C-24, C-25, C-26, C-27 and C-28 methyl protons, respectively. Two three-proton doublets at δ 0.88 (J = 6.0 Hz) and 0.86 (J = 6.0 Hz.) were attributed to C-29 and C-30 secondary methyl protons. The remaining methylene and methine protons resonated between 2.40 and 1.29 ppm. The presence of all the methyl signals in the range 1.18 to 0.86 ppm indicated their attachment on the saturated carbons. The ^{13}C NMR spectrum of **1** showed 30 carbon signals in the molecule. The C-2 carbonyl and C-3 carbinol carbons appeared at 202.13 and 72.71 ppm, respectively. The carbon signals at 32.41 (C-23), 15.78 (C-24), 14.64 (C-25), 11.61 (C-26), 17.93 (C-27), 28.16 (C-28), 6.81 (C-29) and 20.25 (C-30) ppm were assigned to methyl carbon. The absence of ^{1}H NMR signals beyond 3.73 ppm and ^{13}C NMR signals from 202.13 to 72.71 ppm supported saturated nature of the molecule. The DEPT spectrum of **1** showed the presence of eight methyl, nine methylene, seven methine and six quaternary carbons. The ^{1}H-^{1}H COSY spectrum of **1** exhibited correlation of H_2-1 with H_3-25 and H-3; H-3 with H_3-23 and H-5; and H-22 with H-21, H_3-29 and H_3-30. The ^{1}H-^{13}C HETCOR spectrum of **1** showed interactions of C-2 with H_2-1 and H-3; C-5 with H-3, H_2-6 and H_3-23; and C-21 with H_2-20, H-22 and H_3-29. The ^{13}C NMR spectral values of **1** were compared with the fernane-type triterpenoids (10). On the basis of these evidences, the structure of **1** has been established as farnan-2-one-3β-ol. Compounds **2**, **3** and **4** have been identified as farnan-3-one, olean-18-en-2-one-3-ol and lup-20(29)-en-3β-ol, respectively.

CONCLUSION

Compound **1,** farnan-2-one-3β-ol is a new pentacyclic triterpene isolated for the first time from this plant.

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