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Compounds Isolated from *Tannacetum polycephalum*

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Several known secondary metabolites, namely lupeol (**1**), β-sitosterol (**2**), stigmasterol (**3**), and 2,6-dimethoxy-4-hydroxy acetophenone (**4**) were isolated from a folkloric medicinal plant, *Tannacetum polycephalum*. The structures of these compounds were elucidated on the basis of various spectroscopic methods.

Key Words: Tannacetum polycephalum, Asteraceae, lupeol, β-sitosterol, stigmasterol, 2,6 dimethoxy-4-hydroxy acetophenone.

Introduction

The genus *Tannacetum* belongs to the family Asteraceae, comprising 152 species distributed mainly in Europe and temperate Asia, North Africa, North America, and the southern hemisphere. There are about 12 species found in Pakistan.[1] *Tannacetum polycephalum* is a sub-shrub and is distributed in the north and north-west regions of Pakistan. In traditional medicine, *Tannacetum* species are used as a tonic and anthelmintic, spasmodic, and emmenagogue, and possess anticancer and antimicrobial activities.[2]

According to the literature, no phytochemical investigations have been carried out on *Tannacetum polycephalum*. We report here the isolation and structure elucidation of 4 known compounds, **1**-**4**, which were isolated for the first time from this plant. The structures of these compounds were deduced by comparison of their spectral data with those reported in the literature.

Experimental

General Experimental Procedures: The ^{1}H-NMR spectra were recorded in $CDCl_3$ on Bruker AM-400 and AMX-500 NMR spectrometers with TMS as an internal standard using the UNIX operating system at 400 and 500 MHz, respectively. The ^{13}C-NMR spectra were recorded in $CDCl_3$ at 125 MHz on a Bruker

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AMX-500 NMR spectrometer. HREI-MS were recorded on Jeol JMS 600 and HX 110 mass spectrometers with the data system DA 5000. The IR spectra were recorded on a Jasco A-302 spectrophotometer. The UV spectra were recorded on a Hitachi U-3200 spectrophotometer. The optical rotations were measured on a JASCO DIP-360 digital polarimeter. The melting point was determined on a Buchi 510 apparatus. Column chromatography (CC) was carried on a silica gel column (70-230 mesh). Purity of the samples was checked by TLC on pre-coated silica gel GF-254 preparative plates (20 × 20 cm, 0.25 mm thick, Merck) and were detected under UV light (254 and 366 nm), while ceric sulphate was used as the spraying reagent.

Plant Material: The aerial parts of *Tannacetum polycephalum* (4.5 kg) were collected in July 2002 from Chitral (Pakistan) and were dried in the air. The plant was identified by Mr. Tahir Ali, Plant Taxonomist, Department of Botany, University of Karachi, Karachi, Pakistan. A herbarium specimen of this plant (KUH # 84326) was deposited as a reference at the Department of Botany, University of Karachi, Karachi, Pakistan.

Extraction and Isolation of Compounds: The air-dried plant of *Tannacetum polycephalum* (2.0 kg) was extracted with methanol (5 L) at room temperature (30 °C) for 15 days. After evaporation of the solvent, a crude extract (168.2 g) was obtained, which was dissolved in distilled water (500 mL) and defatted with petroleum ether (2.5 L). The defatted aqueous layer was extracted with $CHCl_3$ (3 L) to afford a chloroform fraction (30.4 g). Lastly, the remaining aqueous portion was extracted with ethyl acetate (3 L) to obtain an ethyl acetate fraction (26.8 g).

The resulting chloroform extract (30.4 g) was subjected to CC on a silica gel column (70-230 mesh, 300 g) and the column was eluted with about 5 L of petroleum ether:$CHCl_3$ to $CHCl_3$:MeOH mixtures with increasing polarity (from 9:1 to 8:2, respectively) to afford 6 major fractions (TPC-1 to TPC-6). Compounds **1** (18.2 mg, petroleum ether:$CHCl_3$, 7.2:2.8), **2** (32.6 mg, petroleum ether:$CHCl_3$, 7:3), **3** (56.8 mg, petroleum ether:$CHCl_3$, 6.5:3.5), and **4** (9.2 mg, petroleum ether:$CHCl_3$, 2:8) were isolated from these column fractions by using repeated CC and preparative thin-layer (TLC) chromatographic techniques.

Results

3β-Hydroxylup-20(29)-ene (lupeol) (1): IR ($CHCl_3$) ν_{max}: 3260 (OH), 1640, 1490 (C=C), 1382 cm^{-1} (C-O); UV (MeOH) λ_{max} (log ε): 202 (2.40), 320 nm (3.42); EI-MS$m/z$ (rel. int. %): 426 (17), 279 (7), 218 (48); HREI-MS m/z: 426.3664 (M^+, $C_{30}H_{50}O$; calcd 426.3861); ^{1}H-NMR ($CDCl_3$, 400 MHz): δ 0.75 (3H, *s*, H-24), 0.77 (3H, *s*, H-28), 0.82 (3H, *s*, H-25), 0.97 (3H, *s*, H-27), 0.98 (3H, *s*, H-23), 1.02 (3H, *s*, H-26), 1.67 (3H, *s*, H-30), 3.16 (1H, *dd*, H-3), 4.55 and 4.67 (each 1H, *m*, H-29).

β-Sitosterol (2): IR ($CHCl_3$) ν_{max}: 3406 (OH), 1642 cm^{-1} (C=C); UV (MeOH) λ_{max} (log ε): 206 nm (2.42); EI-MS$m/z$ (rel. int. %): 414 (68), 400 (19), 381 (16), 329 (16), 289 (24); HREI-MS$m/z$: 414.3860 (M^+, $C_{29}H_{50}O$; calcd 414.3812); ^{1}H-NMR ($CDCl_3$, 400 MHz): δ 0.66 (3H, *s*, H-18), 0.75 (3H, *d*, $J_{27,25}$ = 6.2 Hz, H-27), 0.80 (3H, *d*, $J_{26,25}$ = 6.2 Hz, H-26), 0.82 (3H, *t*, $J_{29,28}$ = 7.4 Hz, H-29), 0.90 (3H, *d*, $J_{21,20}$ = 6.5 Hz, H-21), 0.99 (3H, *s*, H-19), 3.50 (1H, *m*, H-3), 5.33 (1H, *br.s*, H-6).

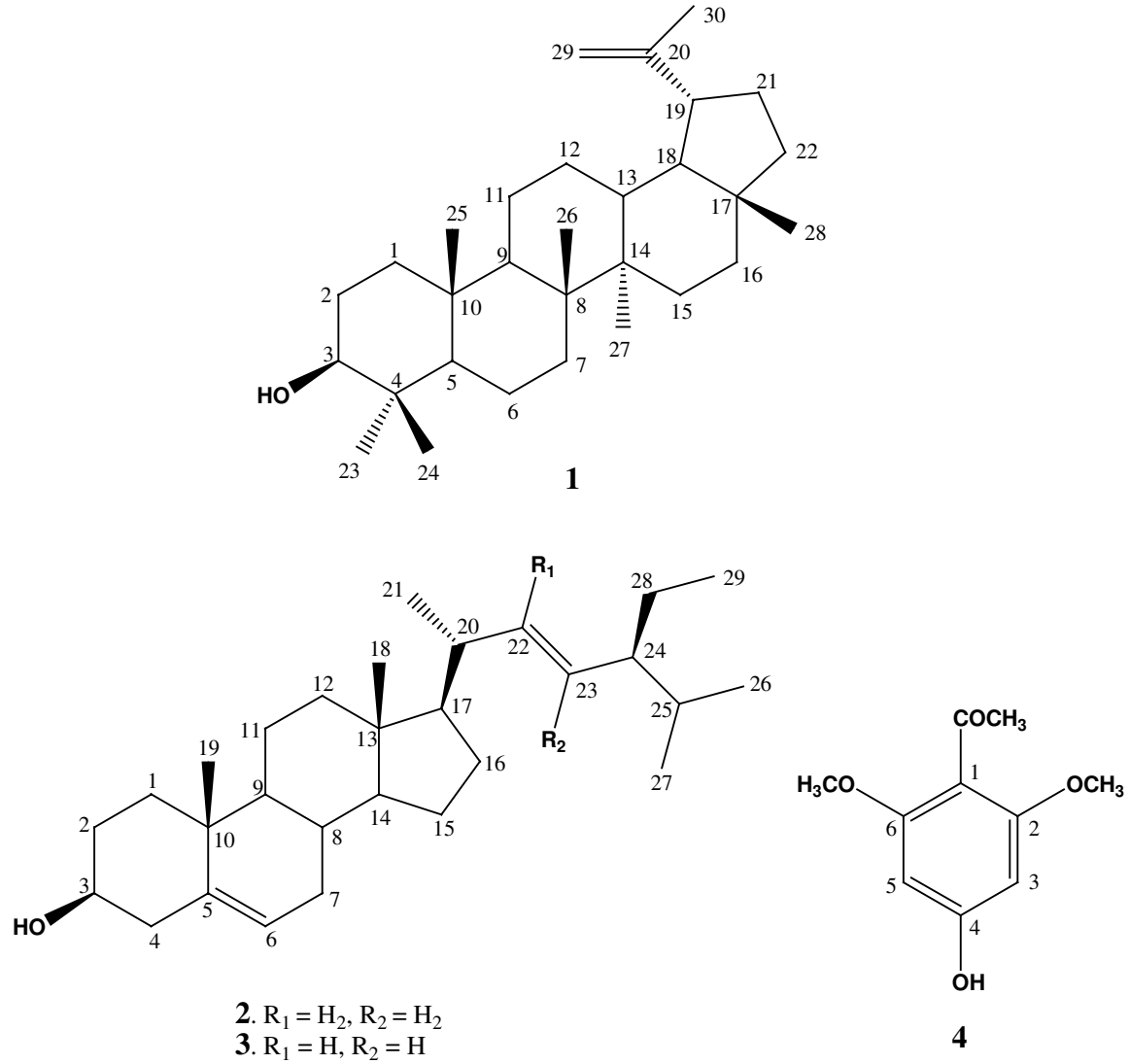
Stigmasterol (3): IR ($CHCl_3$) ν_{max}: 3406 (OH), 1648 cm^{-1} (C=C); UV (MeOH) λ_{max} (log ε): 208 nm (2.46); EI-MS$m/z$ (rel. int. %): 412 (56), 400 (24), 372 (18), 336 (42), 294 (12); HREI-MS$m/z$: 412.2164 (M^+, $C_{29}H_{48}O$; calcd 412.2160); ^{1}H-NMR ($CDCl_3$, 400 MHz): δ 0.68 (3H, *s*, H-18), 0.76 (3H, *d*, $J_{27,25}$ = 6.5 Hz, H-27), 0.83 (3H, *d*, $J_{26,25}$ = 6.5 Hz, H-26), 0.82 (3H, *t*, $J_{29,28}$ = 7.2 Hz, H-29), 1.00 (3H, *s*, H-19), 1.02 (3H, *d*, $J_{21,20}$ = 6.6 Hz, H-21), 3.50 (1H, *m*, H-3), 5.33 (1H, *br.s*, H-6), 5.02 (1H, *dd*, $J_{23,22}$ =

15.2 and $J_{23,24}$ = 8.1 Hz, H-23), 5.13 (1H, *dd*, $J_{22,23}$ = 15.2 and $J_{22,20}$ = 8.4 Hz, H-22).

2,6-Dimethoxy-4-hydroxyacetophenone (4): IR ($CHCl_3$) ν_{max}: 3230 (OH), 1665 ($COCH_3$), 1610, 1486, 1430 cm^{-1} (aromatic C=C); UV (MeOH) λ_{max} (log ε): 286 (3.04), 318 (3.34); EI-MS$m/z$ (rel. int. %): 196 (27), 181 [$(M$-$CH_3)^+$, (100)], 166 [$(181$-$CH_3)^+$, (8)], 138 [$(166$-$C{\equiv}O)^+$, (5)]; HREI-MS$m/z$: 196.0755 (M^+, $C_{10}H_{12}O_4$; calcd 196.0736); ^{1}H-NMR ($CDCl_3$, 400 MHz): δ 2.59 (3H, *s*, $COCH_3$), 3.80 (3H, *s*, 6-OCH_3), 3.83 (3H, *s*, 2-OCH_3), 5.90 (1H, *d*, $J_{3,5}$ = 2.4 Hz, H-3), 6.04 (1H, *d*, $J_{5,3}$ = 2.4 Hz, H-5), 13.90 (1H, *s*, 4-OH).

Discussion

The present study on the methanolic extract of *Tannacetum polycephalum* of Pakistani origin resulted in the isolation and characterisation of compounds **1-4** (Figure). The structures of these compounds were



2. $R_1 = H_2$, $R_2 = H_2$
3. $R_1 = H$, $R_2 = H$

1 **4**

Figure. Structures of conpounds 1-4 isolated from Tannacetum polycephalum.

identified on the basis of spectroscopic methods and comparison with the literature. They were found to be lupeol (**1**),[3] β-sitosterol (**2**),[4,5] stigmasterol (**3**),[5] and 2,6-dimethoxy-4-hydroxy acetophenone (**4**).[6] These compounds were isolated from this plant for the first time.

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