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CHEMICAL EXAMINATION OF *CITRUS SINENSIS* STEMS VARIETY BLOOD RED

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ABSTRACT

Chemical examination of Citrus sinensis roots var. blood red has afforded seven compounds, which have been identified as methyl tritriacontanoate, pentanoic acid, nobiletin, sinestein, 5, 4'-dihydroxy-7, 3'-dimethoxy flavanone 3-O-β-glucoside and glucose.

Key words: *Citrus sinensis*, Rutaceae, Methyl tritriacontanoate, Pentanoic acid, Nobiletin, Sinestein, 5,4'-dihydroxy-7, 3'-dimethoxy flavanone 3-O-β-glucoside, Glucose.

INTRODUCTION

Citrus sinensis (L.) var. blood red (family: Rutaceae[1]) is known as sweet orange or mosambi. It migrtated to India from China in the 13th centuary[2]. Sweet oranges are now cultivated throughout the world[3]. The plant is known to cure skin and dental diseases[4]. In view of the fact that there is no previous examination of its stems, we have undertaken the present study.

EXPERIMENTAL

Stems (3 Kg) of the plant were procured from Botanical Gardens, Hisar, and these were chopped into small pieces, which were dried in shade for 24 h under a fan. The extraction was done with hot methanol. Extractives were subjected to column chromatography using silica gel (60-120 mesh). The elution was started with petroleum ether. Polarity was increased slowly. Seven compounds could be isolated.

Melting points were determined on Ganson Electrical Melting Point Apparatus;

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^{1}H NMR on Bruker AC 300 MHz NMR Spectrometer; IR on Hitachi 570 Infra Spectrophotometer; and Mass Spectra on VG 70 S 11-250 J GCMS-DS Spectrometer.

RESULTS AND DISCUSSION

The data obtained for the seven compounds are given as below. Characterizations are based on comparison of the data obtained with those of existing literature data.

Compound A (Methyl tritriacontanoate, 1): It was obtained on elution with benzene-petroleum ether (1 : 19). It crystallized out from methanol as a colourless solid, 20 mg, mp 80°C (lit[5] M.P. 80-82 ^{0}C). IR ($\nu_{max,}$ nujol, cm^{-1}): 1738. ^{1}H NMR (δ, $CDCl_3$): 3.59 (s, 3 H, $COOCH_3$), 1.33-1.18 (60 H, m, 30 x CH_2), 0.89 (3 H, t, *J* 7.5 Hz, CH_2-C**H**$_3$). GCMS (m/z, relative abundance): 508 (M^+, 24).

Compound B (Methyl triacontanoate, 2): It was obtained on elution with benzene-petroleum ether (1 : 9). It crystallized out from MeOH, 18 mg, M.P. 70°C (lit[6] M.P. 71°C). IR ($\nu_{max,}$ nujol, cm^{-1}): 1732. ^{1}H NMR (δ, $CDCl_3$): 3.60 (s, 3 H, $COOCH_3$), 2.47 (2 H, t *J* 7.5 Hz, CH_2-COO), 2.00-0.90 (54 H, m, 27 x CH_2), 0.94 (3 H, t, *J* 7.5 Hz, CH_2-C**H**$_3$). GCMS (m/z, relative abundance): 466 (M^+, 14).

Compound C (Pentacosanoic acid, 3): The elution with benzene-petroleum ether (1 : 1) yielded this compound. It crystallized out from methano, 20 mg, M.P. 77°C (lit[7] M.P. 78-79°C). IR ($\nu_{max,}$ nujol, cm^{-1}): 1706, 3410. ^{1}H NMR (δ, $CDCl_3$) : 2.28 (2 H, t, J 7.5 Hz, CH_2COO), 2.10-1.18 (44 H, m, 22 x CH_2), 0.81 (3 H, t, J 7.5 Hz, CH_2-C**H**$_3$) GCMS (m/z, relative abundance): 380 (M^+, 35).

Compound D (Nobiletin, 4): It was obtained on elution with ethyl acetate-benzene (1 : 9). It crystallized out from MeOH, 60 mg, M.P. 135°C (lit[8] M.P. 134°C). It gave yellow colour with Mg / HCl. IR ($\nu_{max,}$ nujol, cm^{-1}): 1629. ^{1}H NMR (δ, $CDCl_3$): 7.57 (1 H, dd, *J* 7.5 Hz, 2, 5 Hz, H-6'), 7.48 (1 H, d, *J* 2,5 Hz, H-2'), 7.06 (1 H, d, *J* 2,5 Hz, H-5'), 6.63 (1 H, s, H-3), 4.10, 4.03, 3.96, 3.93 (4 x 3 H, 4 s, 4 x OMe), 3.91 (6 H, s, 2 x OMe). GCMS (m/z, relative abundance): 402 (M^+, 100).

Compound E (Sinenstein, 5): It was obtained on elution with ethyl acetate-benzene (1 : 3). It crystallized out from MeOH, 20 mg, M.P. 177°C (lit[9] M.P. 178-179°C). It gave yellow colour with Mg/HCl. IR ($\nu_{max,}$ nujol, cm^{-1}): 1651. ^{1}H NMR (δ, $CDCl_3$): 7.38 (1 H, d, J 2.5 Hz, H-2'), 7.01 (1 H, dd, *J* 7.5 Hz, 2,5 Hz, H-6'), 6.59 (1 H, s, H-8), 6.90 (1 H, s, H-3),

6.84 (1 H, d, J 7.5 Hz, H-5'), 4.01, 3.96, 3.95, 3.92 (5 x 3 H, 5 s, 5 x OMe), GCMS (m/z, relative abundance): 372 (M^+, 100).

Compound F (5,4'-dihydroxy-7,3'-dimethoxy flavanone 3-O-β-glucoside, 6): It was obtained on elution with methanol-ethyl acetate (1 : 9). It crystallized out from ethyl acetate, 90 mg, M.P. 210°C (lit[10] M.P. 210°C). It gave yellow colour with Mg/HCl. GCMS (m/z, relative abundance): 494 (M^+, 3). Molisch test: +ve; Kiliani hydrolysis: glucose, which was confirmed by direct comparison with an authentic sample. Acetate: compd F + Ac_2O + Py. ^1H NMR of the glucodide acetate (δ, $CDCl_3$): 7.19 (1 H, dd, J 7.5, 2.5 Hz, H-6'), 7.10 (1 H, d, *J* 2,5 Hz, H-2'), 7.08 (1 H, d, J 7.5 Hz, H-5'), 6.40 (1 H, d, J 2.5 Hz, H-8), 6.25 (1 H, d, J 2.5 Hz, H-6), 3.82 (6 H, s, 2 x OMe), 5.21-2.70 (9 H, m, H-2, H-3, 7 glu H), 2.02, 2.00, 1.99. 1.97 (4 s, 4 x 3 H, 4 x OAc).

Compound G (Glucose, 7): It was obtained on elution with methanol-ethyl acetate (1 : 4). It crystallized out from ethyl acetate, 50 mg, M.P. 148°C (lit[11] M.P. 146-150°C). IR ($\nu_{max,}$ nujol, cm^{-1}): 3365. GCMS (m/z, relative abundance): 180 (M^+, 3).

Though all the seven compounds turned out to be already known compounds yet the chemistry of the stems of this plant has been revealed.

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