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Flavonoids and Triterpene of *Ficus benghalensis*

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5,6,7,3′,5′-Pentamethoxy-4′-prenyloxyflavone (**1**), rutin, quercetin and 3-acetyl ursa-14:15-en-16-one (**2**) were obtained from the leaves of *Ficus benghalensis* L (Moraceae). Their structures were determined on the basis of spectral data.

Key Words: *Ficus benghalensis*, Moraceae, Leaves, Methoxyflavone, Triterpene.

INTRODUCTION

The genus *Ficus* (Moraceae) is predominantly distributed in the tropics and subtropics. *Ficus benghalensis* L is an ornamental plant cultivated in public gardens in Egypt. It has been reported to be used as antibacterial[1], hypoglycemic[2–5], antioxidant[6], hepatoprotective[7] and anthelmintic[8]. The available reports are those dealing with the isolation of flavonoids[9,10]. Flavonoid and alkaloid were separated from *F. pentoniana*[11]. Triterpenoids were isolated from *F. fistulosa*[12]. The new triterpene lactone from *F. insipida* has been determined[13]. The new ursane and oleanane type triterpenes were isolated from *F. microcarpa*[14]. Leucocyanidin derivative[15], leucopelargonin derivative[16] and bengalenoside[17] were isolated from *Ficus benghalensis*.

RESULTS AND DISCUSSION

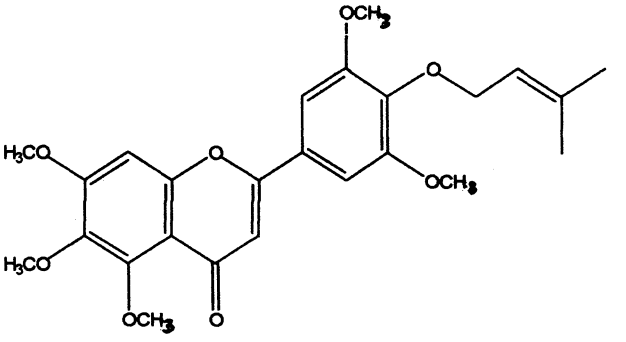
Hexane extract was prepared from the leaves of *Ficus benghalensis* L fractionated by silica gel column chromatography followed by PTLC which afforded quercetin and 5,6,7,3′,5′-pentamethoxy-4′-prenyloxy-flavone (**1**).

The flavone **1** showed UV spectrum at 317 and 275 nm assigned to bands I and II for flavonoid[18]. The ^{1}H NMR spectrum of **1** showed signals at δ 3.88, δ 3.90 and δ 3.95 assigned for five aromatic methoxy groups.

A singlet at δ 6.8 assigned to two equivalent protons (H-2′ and H-6′) and two singlets at δ 6.65 and δ 6.83 assigned to H-3 and H-8 respectively. The signals at δ 4.61(2H, d, J = 7.1 Hz), δ 5.40 (1H, t, J = 7.1 Hz), δ 1.77 (3H, s) and δ 1.71 (3H, s) were attributed to a prenyloxy substitute.

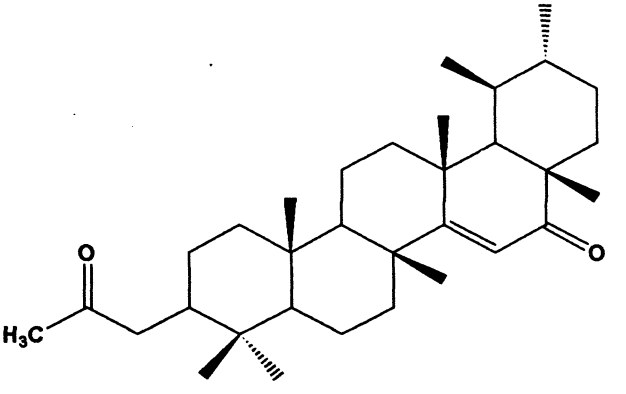
The ^{13}C NMR spectrum of **1** was in total agreement with the 5,6,7-trimethoxy substitutions in the ring A. The two signals downfield, δ 62.20 and 61.50, were chemical shifts characteristic of two methoxy groups at *ortho* position[19].

The comparison of these data with that reported from *F. maxima*[20] proved that compound (**1**) is 5,6,7,3′,5′-pentamethoxy-4′-prenyloxyflavone.



1

The ^1H NMR spectrum of **2** showed six methyl groups at δ 0.88, δ 0.90, δ 0.99, δ 1.14, δ 1.17 and δ 1.30. The chemical shifts of C-29 at δ 1.03 (CH_3, d, J = 6.3 Hz) and C-30 at δ 1.06 (CH_3, d, J = 6.3 Hz) were indicative of two methyl groups attached at C-19 and C-20 in ursane structure[21–23]. The signals at δ 2.03 were assigned to one acetyl group attached at C-3 (δ 4.13). Comparison of ^1H NMR and ^{13}C NMR data was done with that reported from *F. fistulosa*[12]. The structure of compound **2** was deduced as 3-acetyl ursa-14 : 15-en-16-one.



2

Rutin was directly identified by co-chromatography with authentic samples on paper chromatograms. Acid hydrolysis of rutin with 2 M HCl (aqueous methanol 1 : 1) for 30 min and extraction with ethyl acetate gave quercetin. Glucose and rhamnose were detected in aqueous layer after neutralization. The visible characters and retention times, HPLC, in comparison to standard confirmed the identity of quercetin.

EXPERIMENTAL

^{1}H NMR spectrum was recorded at 250 MHz in $CDCl_3$ with TMS as internal standard. UV was recorded using a Perkin-Elmer UV/Vis spectrometer. Silica gel 60 GF_{254} plates were used for PTLC and silica gel G_{60} was used for column chromatography. Whattman No. 1 was used for paper chromatography. Analytical HPLC was recorded using a Gilson equipped with UV/Vis-156 detector on Lichrospher 100RP 18-5 (250 × 4.6 mm) column with solvents system water-methanol 10 per cent gradient and flow rate 1 mL/min.

Plant material: The leaves of *Ficus benghalensis* L were collected in December 2001 from Cairo. Prof. Dr. D. Mohamed, Faculty of Agriculture, Cairo University, made the identity of the plant.

Extraction and isolation: The ground dried leaves were extracted with hexane, chloroform and methanol successively. The hexane extract was submitted to flash column over silica gel using chloroform, ethyl acetate and methanol as eluents. The ethyl acetate fraction obtained from flash column was fractionated over silica gel column with mobile phase hexane-ethyl acetate mixtures of increasing polarity. Fraction eluted with hexane-ethyl acetate 20% was purified by repeated CC on silica gel with mixtures of 5–40% ethyl acetate in hexane followed by PTLC (silica gel : hexane-ethyl acetate, 1 : 1, several runs) to give compound **1** and quercetin.

The chloroform extract was subjected to column chromatography over silica gel using petroleum ether-ethyl acetate (gradient) to obtain nine fractions. Fraction number four was purified over silica gel column using petroleum ether-ethyl acetate (gradient) to give compound **2.**

Rutin was detected in methanol extract. The preparative papers chromatography were used to separate it with solvents system BAW and visualized under UV to show deep purple colour change to yellow with ammonia[19].

Quercetin was identified by HPLC in comparison with authentic samples eluting with methanol-water (gradient). In each analysis 20 μL of the filtered isolated compound was directly injected on to the chromatographic column. Their visible characters and retention times in comparison to standard confirmed the identities of the chromatographic peaks.

Compound (1): 5,6,7,3′,5′-Pentamethoxy-4′-prenyloxyflavone **(1)**: UV λ_{max} nm 317 and 275, ^{1}H NMR (250 MHz, $CDCl_3$) δ: 1.71 (3H, s, H-4″), 1.77 (3H, s, H-5″), 3.88 (3H, s, OCH_3), 3.90 (6H, s, 2 OCH_3), 3.95 (6H, s, 2 OCH_3), 4.61 (2H, d, J = 7.1 Hz, H-1″), 5.4 (1H, t, J = 7.1 Hz, H-2″), 6.65 (1H, s, H-3), 6.83 (1H, s, H-8), 6.80 (2H, s, H-2′ and H-6′). ^{13}C NMR (300 MHz, $CDCl_3$) δ 26.1 (C-2 Me), 56.3 (C-OMe), 65.4 (C-2 OMe), 61.5 (C-OMe), 62.2 (C-OMe), 69.5 (C-1″), 96.2 (C-8), 103.6 (C-6′, 2′), 108.1 (C-3), 112.9 (C-10), 120.4 (C-2″), 126.7 (C-1′), 138.6 (C-3″), 140.1 (C-6), 140.5 (C-4′), 152.7 (C-9), 154.2 (C-5′, 3′), 154.6 (C-5), 157.8 (C-7), 161.1 (C-2), 177.5 (C-4).

Compound (2): 3-Acetyl ursa-14 : 15-en-16-one **(2)**: ^{1}H NMR (250 MHz, $CDCl_3$) δ: 0.88 (3H, s, CH_3-23), 0.9 (3H, s, CH_3-24), 0.99 (3H, s, CH_3-25), 1.03 (3H, δ, CH_3-29, J = 6.5 Hz), 1.06 (3H, δ, CH_3-30, J = 6.5 Hz), 1.14 (3H, s, CH_3-26), 1.17 (3H, s, CH_3-27), 1.3 (3H, s, CH_3-28), 2.03 (3H, s, acetyl CH_3),

4.13 (1H, dd, CH-3), 5.8 (1H, s, CH-15). ^{13}C NMR (300 MHz, $CDCl_3$) δ 15.5 (C-24), 16.5 (C-6, 29), 18.6 (C-11), 21.2 (C-32), 21.9 (C-27), 22.5 (C-23), 23.3 (C-2), 24.8 (C-26), 25.9 (C-25), 27.8 (C-30), 28.5 (C-12), 28.8 (C-1), 30.6 (C-22), 32.3 (C-19), 34.5 (C-28), 36.7 (C-20), 37.2 (C-21), 37.6 (C-4), 37.8 (C-10), 39.6 (C-13), 40.4 (C-8), 40.9 (C-7), 43.1 (C-17), 47.6 (C-9), 55.2 (C-5), 58.5 (C-18), 80.6 (C-3), 119.1 (C-15), 171.0 (C-31), 181.0 (C-14), 208.0 (C-16).

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