

SHORT COMMUNICATION

TRITERPENE COMPOUNDS FROM THE LATEX OF *FICUS SUR* I.

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ABSTRACT. Two pentacyclic triterpenoids of oleanane and ursene structures have been isolated from the latex of the *Ficus sur*. The compounds isolated from the latex are naturally acetylated in the 3-position and their structures have been elucidated on the basis of spectroscopic studies.

KEY WORDS: Latex, *Ficus sur*, Moraceae, Triterpenoids

INTRODUCTION

The genus *Ficus* (Moraceae) comprises about 750 species, most of them are found in tropical areas, about 100 of them are found in Africa and over 600 in Asia and Australia [1]. *Ficus sur* is a tree of upland rain forest, a mountain grassland or secondary scrub type, and riveraine forest mainly growing at streams at 1400-2500 m above sea level. It is widely spread in tropical Africa stretching from Senegal to South Africa. There are some researches on the *Ficus* spp. figs volatile compounds, leaves and twigs extracts for their biological activities [2-4].

Children, especially the herders, use the latex of some of *Ficus* species as a chewing gum. In this paper, the characterization of two compounds isolated from *Ficus sur* latex is reported.

RESULTS AND DISCUSSION

The bark of the *Ficus sur* was wounded for artificial excretion of the latex from the plant. The latex is a white viscous liquid substances immediately collected in amber glass bottle to avoid light induced changes. The latex was suspended in small portion of water and extracted with hexane and acetone. The hexane layer was chromatographed on silica gel 60-120 mesh and the fraction eluted with hexane-ether solution by increasing the later from 3 to 10% v/v. Gradient of polarity collected at *ca* 50 mL and the fraction grouped into three parts. The first fraction accounts for over 60%, the second 25% and third 10% of the total sample amount loaded for chromatography.

The first fraction was further purified on column chromatography using silica gel 60-120 mesh. Two fractions with R_f 0.7 and 0.85 were further purified on high performance thin layer chromatography.

The NMR analyses of these samples indicated the presence of 30 carbons, with a double bond, which depicted that the compounds are triterpenes. The base peak m/z 218, indicated that the compound has either ursene or oleanane structure, in which the Retro-Diels-Alder reaction is responsible for the prominent fragmentation of such type structure of terpenes [5, 6].

Compound **1** with R_f 0.7, the molecular structure was established with ¹H and ¹³C NMR, and EIMS experiments. Analysis of ¹³C NMR and DEPT spectra indicated the presence of carbonyl

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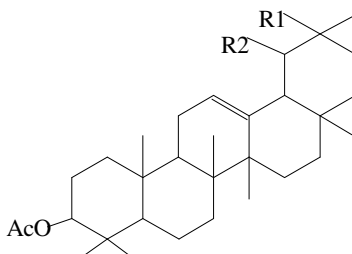
carbon δ 171.4, one double bond (δ 124.7, δ 140.0), eight methyls, an acetyl group, seven methines, eight methylenes and six quaternary carbon signals. The spectral data for the compound given in Table 1 are in agreements with 3 β -acetate-12-ursene [7-10]. The ^1H NMR spectrum of this compound indicated the presence of two methine protons; one at δ 5.15 (1H dt, 8 Hz and 4 Hz) attached to double bond and the other is oxygenated at δ 4.5 (1H, mb) on 3-position, and one acetyl group at δ 2.05 (3H, s). The EI mass spectra $[\text{M}]^+$ m/z 468, with base peak at m/z 218, showed the fragmentation character for ursene type compounds. Based on the above data compound **1** is confirmed as acetate- α -amyrin.

Table 1. ^{13}C NMR spectral assignment for compound **1** and **2** (δ ppm).

Carbon No.	1	2
1	38.8	39.6
2	28.5	27.9
3	81.3	80.8
4	38.1	39.5
5	55.6	55.1
6	18.6	18.1
7	33.2	33.6
8	37.2	38.3
9	48.0	47.4
10	34.1	35.0
11	23.6	23.5
12	124.7	121.5
13	140.0	145.1
14	42.4	42.0
15	28.5	28.2
16	27.0	27.9
17	33.2	32.5
18	59.4	59.0
19	40.0	40.2
20	40.0	41.4
21	31.6	31.0
22	41.9	42.0
23	28.4	29.5
24	16.1	15.8
25	17.1	15.8
26	17.2	16.8
27	23.6	23.5
28	28.4	28.8
29	17.9	17.6
30	21.7	21.2
$\underline{\text{C}}\text{OO}$	171.4	170.8
CH_3COO	21.7	21.2

Compound **2** with R_f 0.85, molecular formula $\text{C}_{32}\text{H}_{52}\text{O}_2$ and its structure was established with ^1H and ^{13}C NMR, and EIMS experiments. The ^{13}C NMR and DEPT spectra indicated the presences of carbonyl carbon δ 170.8, one double bond (δ 121.5, δ 145.1), eight methyl, an acetyl, five methines, ten methylenes and six quaternary carbon signals. The spectral data for the compound (Table 1) is in agreement with acetate- β -amyrin [7, 8, 10, 11]. The ^1H NMR spectrum of this compound indicated the presence of two methine protons at δ 5.1 (1H t, $J = 8$ Hz and $J = 4$ Hz) attached to double bond and at δ 4.4 (1H, m broad) on 3-position, and one acetyl group at δ 2.0 (3H, s). The EI mass spectra $[\text{M}]^+$ m/z 468, with base peak at m/z 218,

showed the fragmentation character for oleanane type compounds. Based on the above data compound **2** is confirmed as acetate- β -amyrin.



- 1** 3-Acetoxy- α -amyrin $R_1 = H, R_2 = Me$
2 3-Acetoxy- β -amyrin $R_1 = Me, R_2 = H$

EXPERIMENTAL

General. Melting points are uncorrected. NMR experiments on a Bruker AMX 500 instrument 1H NMR: 400 MHz, ^{13}C NMR: 100 MHz and Bruker AC 200 MHz; 1H NMR: 200 MHz and ^{13}C NMR: 50 MHz using TMS as internal standard. Optical rotations were measured at 22 $^{\circ}C$ on Bellingham and Stanley P20 polarimeter. Chromatography column: silica gel 60-120 mesh. GC/MS on Finnigan Trace Plus, column: Rtx – 5ms (5% diphenyl and 95% dimethyl polysiloxane), 30 m x 0.25 mm x 0.25 μm . He as a carrier gas 1.5 mL/min, 40-200 $^{\circ}C$ at 5 $^{\circ}C/min$ and 200-250 $^{\circ}C$ at 10 $^{\circ}C/min$, isotherm for 5 min at 250 $^{\circ}C$; injection temperature was 250 $^{\circ}C$.

Plant material. The latex of *Ficus sur* was collected in May 2000 from Wendo Genet College of Forestry Campus. The bark of the *Ficus sur* was wounded for artificial excretion of the latex from the plant. The latex is a white viscous liquid substance, which is immediately collected in amber glass bottle to avoid light induced changes.

Isolation of the compound. The latex, 90 g, was suspended in 20 mL of distilled water and extracted with (10 x 100 mL) hexane at 18-20 $^{\circ}C$ with shaking for 15 min and allowed to stay for 5 min. The hexane extracts were concentrated on vacuum to give a concentrate of 50 g. Hexane extract, 30 g, was subjected to column chromatography using 400 g of silica gel 60-120 mesh, eluted with hexane with increasing polarity of ether from 3 to 10% v/v to give 3 fractions. Fraction 1, 10 g was subjected to column chromatography eluting with hexane and hexane - MeOH (99.5: 0.5).

Acetoxy - α -amyrin 1. White powder, m.p. 224-226 $^{\circ}C$. $[\alpha]_D^{20}$: +76.1 $^{\circ}$ (c 0.38, $CHCl_3$). EIMS m/z (rel. int): 218 (100), 202 (32), 188 (31). 1H NMR δ : 0.80 (6H, s), 0.83 (3H, s), 0.88 (6H, s), 0.92 (3H, s), 0.98 (3H, s), 1.0 (3H, s), 2.05 (3H, s), 4.4 (1H, m, H-3), 5.1 (1H dt, J = 8 Hz and J = 4 Hz, H-12). ^{13}C NMR: Table 1.

Acetoxy - β -amyrin 2. White powder, m.p. 238-240 $^{\circ}C$. $[\alpha]_D^{20}$: +80.8 $^{\circ}$ (c 0.34, $CHCl_3$). EIMS m/z (rel. int): 218 (100), 202 (32), 188 (31). 1H NMR δ : 0.80 (6H, s), 0.83 (3H, s), 0.88 (6H, s), 0.92

(3H, s), 0.98 (3H, s), 1.0 (3H, s), 2.05 (3H, s), 4.5 (1H, m, H-3), 5.15 (1H dt, J = 8 Hz and J = 4 Hz, H-12). ¹³C NMR: Table 1.

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