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A New Prenylated Chalcone from the Stem-bark of *Lonchocarpus sericeus* Poir (Papilionaceae)

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ABSTRACT
Lonchocarpus *sericeus* belonging to the papilionaceae family is a plant used in folk medicine for the treatment of convulsion, **backache,** constipation and anorexia. 2',4'-dihydroxy-3'-(4-hydroxy-4-methyl pentenyl)-chalcone was isolated from the **chloroform** soluble fraction of the methanol extract of *Lonchocarpus sericeus* bark using a combination of silica gel column **chromatography** and gel filtration,. The structure of this compound was elucidated with the help of 1D and 2D NMR **spectroscopic** analysis. This is the first report of isolation of this compound. Detailed HSQC and HMBC have been provided **for the new** compound.

KEYWORDS: Lonchocarpus sericeus, Papilionaceae, NMR analysis, 2',4'-dihydroxy-3'-(4-hydroxy-4-methyl pentenyl)-**chalcone.**

INTRODUCTION

Lonchocarpus sericeus commonly known as Cube root or Senegal lilac is a dry deciduous tree that often grows wild and may reach up to 16m high in Nigeria and Ghana. The main use of the bark in African traditional medicine is as a stomachic and laxative particularly in young children. It also has common application in treating convulsions and back ache[1]. Preliminary phytochemical screening of the methanol extract of the stem bark revealed the presence of alkaloids, saponins, flavonoids, tannins, triterpenes and steroids[2] while that of the chloroform fraction revealed the presence of phenolic compounds, flavonoids, triterpenoids and steroids[3]. Phytochemical investigation of the root and seed led to the isolation of bioactive chalcones; Lonchocarpin and Derricin[4].

The ethnomedicinal use of the stem-bark in the treatment of convulsion was validated scientifically [2]. The chloroform extract of the stem-bark has also been shown to posses some anticonvulsant activity [3]. Despite its medicinal potentials, there is no report yet on the isolation and characterization of any compound from the stem-bark of the plant. In this paper we report the isolation of a prenylated chalcone from the chloroform- soluble fraction of the methanol extract of the stem-bark of *L. sericeus.*

MATERIALS AND METHODS

Collection and identification of the plant material

The stem-bark of *Lonchocarpus sericeus* was collected from Kongo Campus of Ahmadu Bello University, Zaria, after identification of the tree on the field using descriptions in the monograph [1]. The leaves of the plant were also collected alongside to ease identification of the bark. The identity of the bark was confirmed and authenticated by Messrs Musa Muhammad and Umar Gallah of the Herbarium Unit, Department of Biological Sciences, Ahmadu Bello University, Zaria by comparison with the existing herbarium specimen of voucher number 1085.

Preparation of the extract

The stem-bark was dried at room temperature for several weeks and size reduced manually using mortar and pestle.

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The size reduced stem-bark (2kg) was extracted with Methanol in a soxhlet apparatus for 72 hr and concentrated *in vacuo*. This yielded 100 g of a gummy extract. The extract was suspended in distilled water and filtered. The water insoluble portion was successively washed with n-hexane. chloroform and ethyl acetate to yield n-Hexane (nHxF), chloroform (CF) and ethyl acetate (EAF) fractions respectively. CF was subjected to further phytochemical investigations because of the promising anticonvulsant activity of CF [3].

Isolation and Purification of Compound R_2

10g of CF was subjected to column chromatography on a silica gel (60 – 120 mesh) with gradient elution using Hexane and Chloroform. Eluents were collected in 25ml aliquot and TLC was used to monitor the progress of elution.

A total of 104 fractions were collected and pooled together into 9 major fractions based on their TLC profiles. Fraction 4, (10 mg) which showed four major spots was subjected to repeated gel filtration using sephadex LH_{20} and eluted with absolute methanol to give 5.2 mg of yellow amorphous compound coded as R_2. R_2 gave a single homogenous spot with Hexane: ethylacetate 9:1 and 5:1. R_2 was subjected to chemical test and spectral analysis to elucidate its chemical structure.

Chemical Test on R_2

R_2 gave a Prussian blue colour with freshly prepared Ferric chloride solution indicating the presence of a phenolic nucleus. It also produced red colour with concentrated Hydrochloric acid in the presence of Magnesium chips. This is indicative of a flavonoid nucleus.

Spectroscopic Characterization

^{1}H NMR (600 MHz, $CDCl_3$) and ^{13}C NMR (150 MHz, $CDCl_3$), HSQC, COSY and HMBC spectra were recorded on a Bruker AVANCE-600 Japan using the residual solvent peak as reference.

RESULTS AND DISCUSSION

The ^{1}H NMR spectrum of R_2 exhibited signals for ortho coupled hydrogens at δ_H 6.37 (d, 1H, J=8.8Hz, H-5') and δH 7.71 (d, IH, J=8.9Hz, H-6') due to 1,2,3,4 tetra substituted aromatic ring. It also revealed the presence of two hydrogens at δH 7.55 (d, IH, J=15.5Hz, H-α) and δH 7.86 (d, IH, J=15.4Hz, H-β); this is typical of olefinic hydrogen in trans arrangement. It also showed two multiplets at δH 7.40 (3H) and δH 7.63 (2H) corresponding to the five protons of a second monosubstituted aromatic ring. The down field signal at δH 13.7 (s) indicates the presence of a free hydroxyl group. Other resonances at δ_H 5.58 (d, 1H, J=10.1Hz, H-2") and δ_H 6.74 (d, IH, J=10Hz, H-1") typical of olefinic protons in cis-arrangement, δ_H 1.26 (d, J=4.6Hz, H-3") due to methylene group integrating for two protons and 0.87(m) integrating for two methyl groups are indicative of the presence of a prenyl moiety as a side chain of the molecule.

The ^{13}C spectrum of R_2 showed 21 signals due to two benzene rings, a prenyl group at δ_C 115.9, a carbonyl carbon at δ_C 191.9 and the α and β carbon resonances appeared at δ_C 120.3 and δ_C 144.2 respectively.

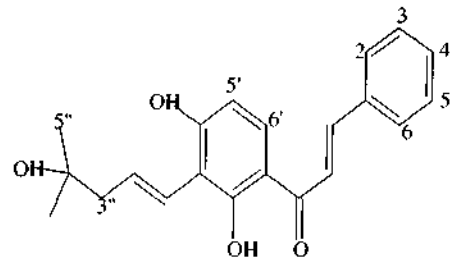
The DEPT experiment disclosed two carbon resonances for methyl groups at δ_C 14.2 and δ_C 28.4, a methylene carbon at δ_C 29.7 and nine methine carbons at δ_C 144.2, 130.8, 130.7, 129.0, 128.6, 128.2, 120.3, 115.9 and 78.0.

The assignment of the carbons and the placement of the prenyl side chain were achieved using 2D NMR experiments.

The ^{1}H-^{1}H COSY spectrum of R_2 showed four important cross peaks between δ_H 7.55 (H-α) and δ_H 7.86 (H-β); 5.58(H-2") and δ_H 6.74(H-1"); δ_H 6.37 (H-5') and δ_H 7.71 (H-6'); δ_H 7.40 (H-2, 4, 6) and δ_H 7.63 (3, 5) indicating protons that are attached on adjacent carbons.

In the HMBC spectrum, a common J_3 correlation between δ_H 7.63 (H-3, H-5) and δ_H 7.55 (H-α) and a J_2 correlation between δ_H 7.86 (H-β) and δ_H 7.40 (H-2, H-6) with a quaternary carbon at δ_C 134.8 confirms its assignment as C-1. Also a common J_3 correlation between δ_H 7.86 (H-β) and δ_H 7.71 (H-6') to a quaternary carbonyl carbon at δ_H 191.9 confirms that C=O is linked to C-1' of ring A.

In the prenyl side chain, the methine proton signal at δ_H 5.58 (H-2") showed cross peak with a quaternary carbon signal at δ_C 78.0 through a J_3 correlation and a methylene carbon signal at δ_C 29.7 by J_2 correlation. The methyl hydrogens showed J_3 correlation to the methyl carbons of their respective partners and to the methylene carbon at δ_C 29.7. They also showed J_2 correlation to the quaternary carbon bearing the hydroxyl group at δ_C 78.0 on which they are attached. A J_3 correlation between δ_H 5.58 and a J_2 correlation between δ_H 6.74 to a quarternary carbon at δ_C 114.1 (C-3') indicates that the prenyl moiety is joined to the molecule through C-3' carbon. The combination of 1D and 2D NMR as enunciated above and Table 1 led us to propose the structure below for R_2



2',4'-dihydroxy-3'-(4-hydroxy-4-methyl penteny

To the best of our search, this is the first report of the isolation of this compound. However, some similar prenylated chalcones such as dihydrocordoin was previously reported from the same genus [5].

Table 1: ^{1}H NMR, ^{13}C NMR, DEPT and HMBC information about R_2

Position	δ_C (ppm)	δ_H (J) Hz	DEPT	HMBC
1	134.8		C	
2, 4	128.6	7.40 (1.5, 6.4)	CH	C-1,2,4,3,5,C-α
3, 5	128.2	7.63 (1.7, 9.0)	CH	C-1, C-3, C-5,C-2,C-4
6	130.7	7.40 (1.5, 6.4)	CH	
1'	130.8		C	
2'	161.0		C	
3'	114.1		C	
4'	159.9		C	
5'	108.4	6.37 (8.8)	CH	C-4', C-3', C-1', C-5'
6'	109.5	7.71 (8.9)	CH	C=O, C-2', C-4', C-1'
C=O	191.9		C	
1"	115.9	6.74 (10.0)	CH	C-2', C-4', C-1"
2"	129.0	5.58 (10.1)	CH 3"	C-2", C-1", C-4", C-
3"	29.7	1.26 (4.6)	CH_2	
4"	78		C	
5"	14.2	0.87 (6.7)	CH_3	
6"	28.4	1.50	CH_3	
α to C=O	120.3	7.55 (15.5)	CH	C=O, C-β, C-1
β to C=O	144.2	7.86 (15.4)	CH	C=O, C-1,C-6,C-2,C-α

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