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New *C*-methylated flavonoids and *α*-pyrone derivative from roots of *Talinum triangulare* growing in Nigeria

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ABSTRACT

The first chemical examination of roots of the traditionally used medicinal plant *Talinum triangulare* (Portulacaceae) from Nigeria led to the isolation of two new *C*-methylated flavonoids, 5,6-dimethoxy-7-hydroxy-8-methyl-flavone (**1**), 5,6-dimethoxy-8-methyl-2-phenyl-7*H*-1-benzopyran-7-one (**2**), and one new *α*-pyrone derivative, 4-methoxy-6-(2-hydroxy-4-phenylbutyl)-2*H*-pyran-2-one (**3**), along with thirteen known compounds, including nine amides (**4**–**12**), indole-3-carboxylic acid (**13**), *p*-hydroxy benzoic acid (**14**), and two steroids (**15**–**16**). Their structures were elucidated by extensive spectroscopic measurements including 1D, 2D NMR, MS, and by comparison with the literature. All isolated compounds were screened for their cytotoxic and antifungal activities. However, none of them showed significant activity.

1. Introduction

Talinum triangulare (Portulacaceae) is probably native to tropical America but has been introduced to Nigeria and other tropical regions in Africa as a leaf vegetable. Now it becomes one of the most important vegetables in Nigeria and known as "waterleaf" [1]. The ethnobotanical use of leaves from this plant in Nigeria includes the treatment of peptic ulcer in different parts of Nigeria [2]. Macerated leaves of *T. triangulare* are also applied locally for the treatment of cuts, wounds and scabies in Ovia North East Edo state in Nigeria [3], whereas a decoction of roots from *T. triangulare* is taken three times daily for 7 days in the treatment of hypertension in Iregun area of Ogun state in Nigeria [4–5]. Phytochemical studies of this plant are rare with one exception that is reported frequently encountered metabolites such as steroids and allantoin among others from stems and leaves [6]. No phytochemical studies exist yet on natural products from the roots of *T. triangulare*. Our present study on metabolites from the roots of *T. triangulare* yielded 16 compounds including three new compounds 5,6-dimethoxy-7-hydroxy-8-methyl-flavone (**1**), 5,6-dimethoxy-8-methyl-2-phenyl-7*H*-1-benzopyran-7-one (**2**), and 4-methoxy-6-(2-hydroxy-4-phenylbutyl)-2*H*-pyran-2-one (**3**) (Fig. 1). Herein, we describe the structure elucidation of the new compounds, and screening results on the biological activities of all isolated metabolites.

2. Results and discussions

Compound **1** was isolated as an orange sticky solid. Its UV spectrum showed absorption maxima at 213, 267, and 314 nm, which agreed with a flavonoid skeleton. The molecular formula of **1** was determined as $C_{18}H_{16}O_5$ based on HRESIMS data, indicating 11 degrees of unsaturation. The ^{1}H NMR spectrum (Table 1) showed two aromatic protons at δ_H 7.90 (dd, $J = 7.7$, 2.0 Hz, H-2′ and H-6′), one aromatic proton at δ_H 7.53 (m, H-4′), and two aromatic protons at δ_H 7.52 (m, H-3′ and H-5′), suggesting the presence of a monosubstituted benzene ring *B*, which was further confirmed by the HMBC correlations (Fig. 2) from H-2′ and H-6′ to C-4′ (δ_C 131.4), from H-3′ and H-5′ to C-1′ (δ_C 131.8), and from H-4′ to C-2′ and C-6′ (δ_C 126.0). On the basis of the HMBC correlations from H-3 (δ_H 6.73, s) to C-2 (δ_C 161.3), C-4 (δ_C 178.0), C-10 (δ_C 112.1), and C-1′, and from H-2′ and H-6′ to C-2, the nature of ring *C* was established and ring *B* was deduced to be substituted at C-2. Furthermore, key HMBC correlations from H_3-11 (δ_H 2.44, s) to C-7 (δ_C 152.0), C-8 (δ_C 108.2), and C-9 (δ_C 152.7), from 5-OMe (δ_H 3.96, s) to C-5 (δ_C 148.6), and from both 6-OMe (δ_H 4.05, s) and H-3 to C-6 (δ_C 137.7) confirmed the nature of ring *A* and the attachments of methyl, hydroxy, methoxy and methoxy groups at C-8, 7, 6 and 5 positions, respectively. Thus, compound **1** was elucidated as 5,6-dimethoxy-7-hydroxy-8-methyl-flavone.

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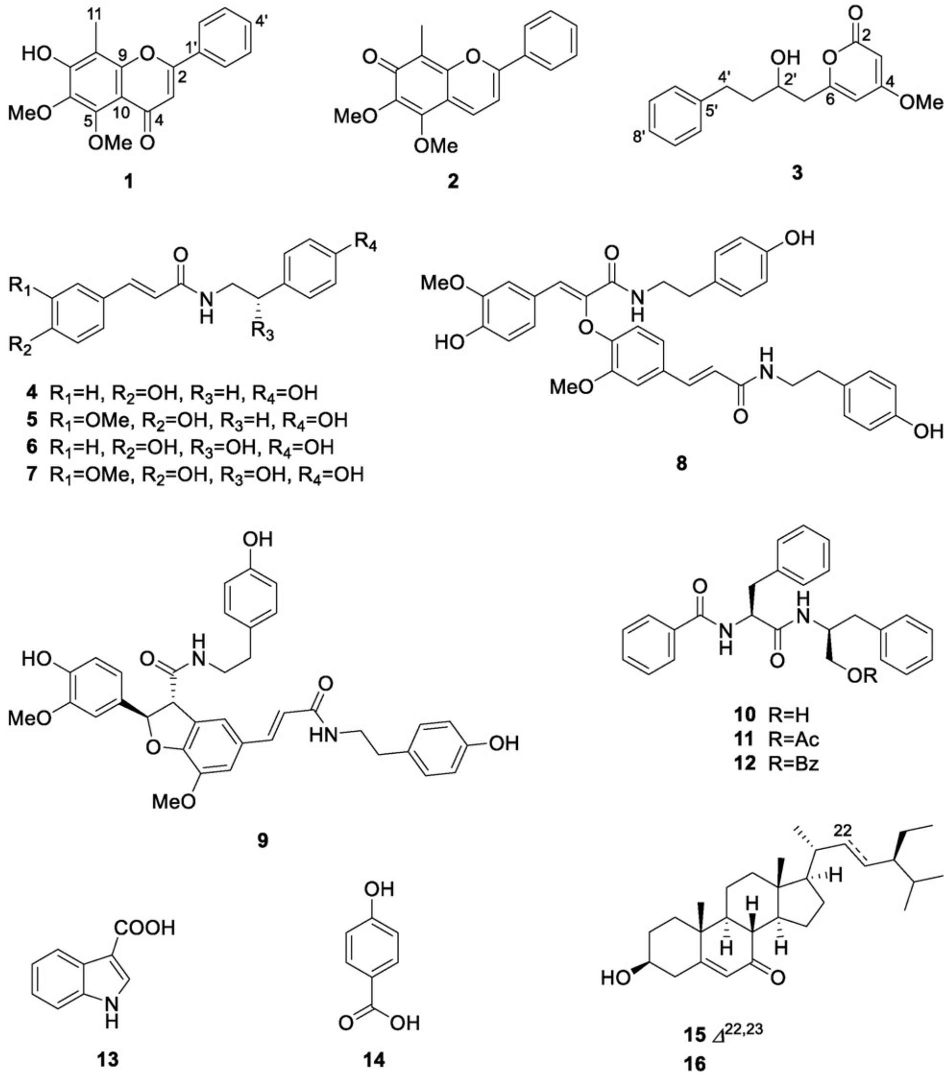

Fig. 1. Structures of compounds **1**–**16** from *Talinum triangulare.*

Compound **2** was obtained as a red solid. It exhibited a pseudomolecular ion peak at *m/z* 297.1122 $[M + H]^+$, which was consistent with the molecular formula of $C_{18}H_{16}O_4$. The ^1H NMR spectrum of **2** (Table 1) showed similar resonances to those of **1**, which included signals for a monosubstituted benzene ring *B* at δ_H 8.07 (dd, J = 7.6, 2.1 Hz, H-2′ and H-6′), 7.59 (m, H-4′), and 7.58 (m, H-3′ and H-5′), a signal for a methyl substituent at δ_H 2.25 (s, H_3-11), and signals for two methoxy groups at δ_H 4.08 (s, 5-OMe) and 3.92 (s, 6-OMe). However, two doublet olefinic protons at δ_H 7.34 (d, J = 7.6 Hz, H-3) and 8.15 (d, J = 7.6 Hz, H-4) were observed in ^1H NMR spectrum of **2** instead of a singlet olefinic proton (H-3) in that of **1**. The COSY correlation between H-3 and H-4, together with the HMBC correlations from H-3 to C-2 (δ_C 161.5), C-10 (δ_C 119.7), and C-1′ (δ_C 132.5), and from H-4 to C-2, C-5 (δ_C 147.5), and C-9 (δ_C 154.6), indicated the presence of two double bonds at C-2/C-3 and C-4/C-10 in ring *C* of **2** (Fig. 2). In

Table 1
^{13}C and ^1H NMR data of compounds **1** and **2.**

Position	1[a]		2[b]	
	δ_C	δ_H (J in Hz)	δ_C	δ_H (J in Hz)
2	161.3, C		161.5, C	
3	107.7, CH	6.73, s	104.7, CH	7.34, d (7.6)
4	178.0, C		137.1, CH	8.15, d (7.6)
5	148.6, C		147.5, C	
6	137.7, C		145.0, C	
7	152.0, C		181.2, C	
8	108.2, C		111.0, C	
9	152.7, C		154.6, C	
10	112.1, C		119.7, C	
11	8.5, CH_3	2.44, s	8.0, CH_3	2.25, s
1’	131.8, C		132.5, C	
2′, 6’	126.0, CH	7.90, dd (7.7, 2.0)	127.2, CH	8.07, dd (7.6, 2.1)
3′, 5’	129.1, CH	7.52, m	130.5, CH	7.58, m
4’	131.4, CH	7.53, m	133.1, CH	7.59, m
5-OMe	62.1, CH_3	3.96, s	62.1, CH_3	4.08, s
6-OMe	61.8, CH_3	4.05, s	60.9, CH_3	3.92, s

[a] Recorded at 600 MHz for ^1H and 150 MHz for ^{13}C in $CDCl_3$.

[b] Recorded at 600 MHz for ^1H and 150 MHz for ^{13}C in CD_3OD.

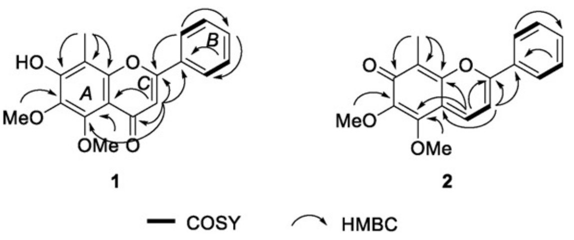

Fig. 2. Key COSY and HMBC correlations of compounds **1** and **2.**