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A new fatty aldol ester from the aerial part of *Mimosa invisa* (Mimosaceae)

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A new aldol ester named 17-*O*-triacontanoylheptadecanal (**1**) was isolated from the aerial part of *Mimosa invisa* (Mimosaceae) together with eight known compounds identified as *β*-sitosterol (**2**), *α*-amyrine (**3**), lupeol (**4**), 4′-*O*-methylepinumisoflavone (**5**), alpinumisoflavone (**6**), betulinic acid (**7**), 3-*O*-*β*-D-glucopyranoside of sitosterol (**8**) and epirobinetinidol (**9**). The structures of compounds were determined on the basis of NMR and mass spectrometry data as well as by comparing the data reported in the literatures. The antimicrobial activities of the crude extract and compounds **1** and **9** were investigated against seven microbial species. The natural products showed moderate activities compared to that of the crude extract.

Keywords: Mimosaceae; *Mimosa invisa*; fatty aldol ester; antimicrobial activity

1. Introduction

Mimosa invisa Linn (Mimosaceae) is a creeping leguminous plant widespread in the humid climate zone and some desert parts of Africa. It grows up in the southern part of Cameroon (Letouzey, 1970) and is difficult to handle because of prickles on its branches. This plant is used to restore the soil fertility (Toxopeus, 1952) and in traditional pharmacopeia, people prepare it as a decoction with some ingredients to treat bronchitis and asthma (Berhaut, 1975). Its extract obtained by infusion is used as a mouthwash by some tribes to relieve tooth pain (Bouquet, 1969). With the aim of promoting the ethnopharmacological uses, these species were biologically and phytochemically studied.

We herein report the structure elucidation of new fatty aldol ester and its antimicrobial activities as well as that of the crude extract.

2. Results and discussion

The ethnopharmacological use of *M. invisa* (Mimosaceae) prompted us to investigate the antimicrobial activities of the crude extract and some isolated compounds. Thus, the crude extract showed good antimicrobial activities against seven microbial strains with the MIC values ranging from 128 to 256 μg mL^{-1} (Table 1). In order to identify the natural products responsible for these activities, the remaining extract was subjected to repeated column

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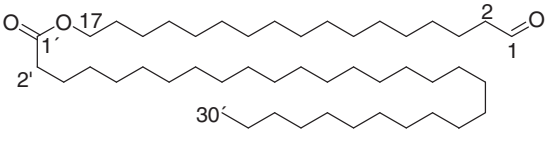
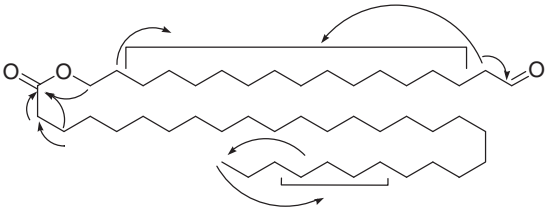
Table 1. MIC values of compounds **1** and **9**, crude extract and reference molecules.

	Microorganisms[a] and MIC ($\mu g\,mL^{-1}$)						
Samples	EC	EA	SA	PA	KP	ST	CA
Crude extract	512	258	128	128	128	256	512
Compound **1**	256	256	256	256	64	128	256
Compound **9**	128	256	512	128	64	128	>512
RA[b]	2	16	2	32	4	1	1

Notes: The final concentration of DMSO was lower than 2.5% and does not affect the microbial growth.
[a]Microorganisms: EC, *Escherichia coli*; EA, *Enterobacter aerogenes*; SA, *Staphylococcus aureus*; PA, *Pseudomonas aeruginosa*; KP, *Klebsiella pneumoniae*; ST, *Salmonella typhi*; CA, *Candida albicans*.
[b]RA, reference antibiotics (ciprofloxacin for bacteria and nystatin for fungi).



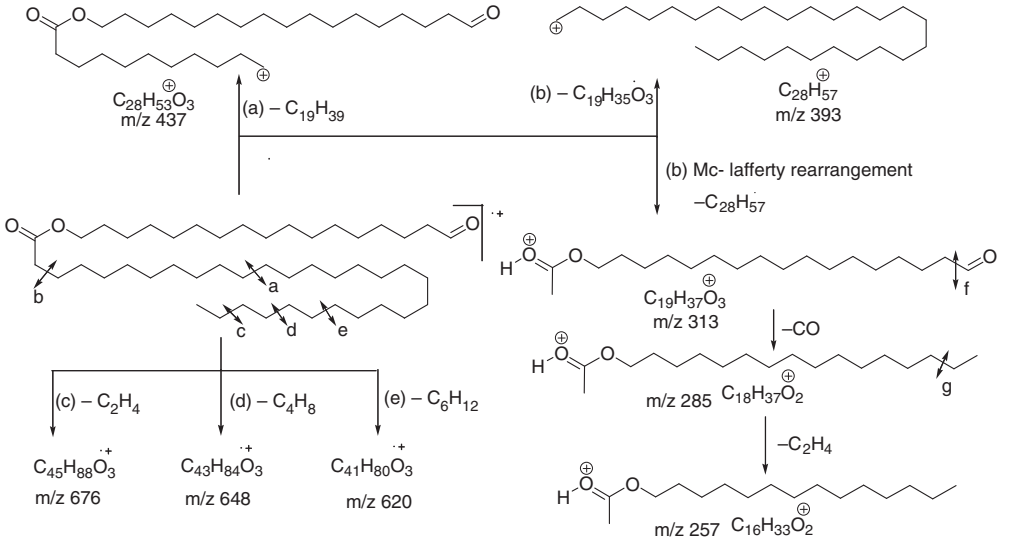
Compound 1



Scheme 1. Some relevant HMBC correlations.

chromatography on silica gel affording nine secondary metabolites. Among them, a new fatty aldol identified as 17-*O*-triacontanoylheptadecanal (**1**) and eight known compounds as β-sitosterol (**2**) (De-Eknamkul & Potduang, 2003), α-amyrine (**3**) (Mahato & Kundu, 1994), lupeol (**4**) (Mahato & Kundu, 1994), 4′-*O*-methylepinumisoflavone (**5**) (Nkengfack et al., 2001), alpinumisoflavone (**6**) (Nkengfack et al., 2001), betulinic acid (**7**) (Mahato & Kundu, 1994), 3-*O*-β-D-glucopyranoside of sitosterol (**8**) (Walter, 1963) and epirobinetinidol (**9**) (Cronje, Steynberg, Brandt, Young, & Ferreira, 1993).

Compound **1** was obtained as a white powder from the mixture hexane/ethyl acetate 3-1 (v/v). Its HREIMS displayed the peak at *m/z* 704.7057 (calcd 704.7046) corresponding to the molecular formula $C_{47}H_{92}O_3$ accounting for two degrees of unsaturation. The IR spectrum presented the absorption band of carbonyl at 1733 cm^{-1}. The ^{1}H- and ^{13}C-NMR spectra (Exp. part) showed the characteristic signals of fatty acid ester (Zu-Jian, Ming-An, & Qing-Wei, 2009), which were the terminal CH_3 group at δ_H/δ_C 1.01 (t, 6.7)/14.1, the sequence of CH_2 groups at δ_H/δ_C 1.42 (br s)/[23.0–33.9] and the CH_2 groups at δ_H/δ_C 2.59 (t, 6.5)/34.9 and 2.50 (br t, 6.7)/44.0 in α of aldehyde and carboxylic functions, respectively. A methylene oxide at δ_H/δ_C 4.36 (t, 6.2)/64.9 and an unresolved resonance of aldehyde at δ_H/δ_C 9.94 (s)/202.6 were further observed. The HMBC spectrum exhibited a correlation (Scheme 1) between the resonance at δ_H 4.36 and the carbon signal at δ_C 173.5, justifying the esterification of fatty acid moiety. Methanolysis of **1** led to the fatty acid



Scheme 2. Fragmentation pattern of compound **1**.

methyl ester identified at *m/z* 466 corresponding to the molecular formula $[C_{31}H_{62}O_2]^{\bullet+}$. These data suggested that **1** contains a fatty acid and fatty aldol both linked by an ester function with the methylene oxide borne by the aldehyde moiety. Besides, some fragments collected from the mass spectrometry spectrum supported the proposed structure. Thus, the fragments at *m/z* 313 and 393 (Scheme 2) from residual ions of Mc Lafferty rearrangement confirmed the length of aldol moiety and consequently those of fatty acid unit. The foregoing data led the identification of **1** as 17-*O*-triacontanoylheptadecanal.

The structures of known compounds were determined by the comparison of their NMR data with those reported in the literatures as well as their physical data.

2.1. *Antimicrobial assays*

The antimicrobial activities of some obtained compounds were already investigated. In fact, compound **6** was found to be active on several bacterial species, including *Mycobacterium smegmatis*, *Candida albicans* and some Gram-positive and Gram-negative bacteria (Kuete, Ngameni, et al., 2008). Compound **4** also exhibited moderate inhibitory effects against *Escherichia coli* and *M. smegmatis*, (Kuete, Wansi, et al., 2008). Meanwhile, the antimicrobial activities of **7** (Mbaveng et al., 2008), **2** and **8** (Kuete et al., 2007; Manríquez-Torres, Zúñiga-Estrada, González-Ledesma, & Torres-Valencia, 2007) were also reported.

Compounds **1** and **9** were subjected to biological assays (Table 1), and **1** displayed activity against the seven tested microorganisms (MIC values ranged from 64–256 $\mu g\,mL^{-1}$) with *Klebsiella pneumoniae* being the most sensitive species (MIC: 64 $\mu g\,mL^{-1}$). Compound **9** was also active on six tested bacterial species but not against *C. albicans*. However, the activities of **1** and **9** could be considered as moderate, since the MIC values obtained were greater than those of ciprofloxacin and nystatin used as reference drugs. Therefore, both tested compounds significantly contribute to the antimicrobial activity of *M. invisa*, together with other isolated metabolites.

3. Experimental

3.1. *General*

Vacuum column chromatography (VCC), column chromatography (CC) and thin-layer chromatography (TLC) were performed over silica gel 60 H (particle size 90% <45 mm), 200–300 mesh silica gel silica gel GF254, respectively. The melting point (m.p.) was measured by an Electro thermal IA 9000 digital m.p. apparatus; uncorrected. IR spectra were carried out on a JASCO A-302 spectrophotometer in $CHCl_3$. Studies on 1D and 2D NMR spectra were carried out with Bruker DRX-400 MHz. HR–EI–MS and LR–EI–MS were recorded with variant JEOL MS mass spectrometer instrument.

3.2. *Plant material*

Mimosa invisa (Mimosaceae) was collected from Yaoundé central region of Cameroon in October 2007. A voucher specimen has been deposited in the National Herbarium, Yaoundé, Cameroon under the registration number 42881/HNC.

3.2.1. *Extraction and isolation*

The aerial part of *Mimosa invisa* was cut into small pieces, air-dried and extracted with 5 L of mixture methylene chloride/methanol (1 : 1, v/v) for 2 days. In this method, 200.0 g of crude extraction obtained from 1.0 kg of plant material was rotary evaporated and subjected to VCC. The VCC was carried out on silica gel in the gradient condition using hexane, hexane/ethyl acetate (3 : 1, 1 : 1 and 1 : 3) and ethyl acetate, yielding five fractions (A–E). Fraction A was purified by CC with hexane yielding the compounds **3** (7.2 mg) and **5** (9.0 mg). Fraction B was further purified by CC with different mixtures of hexane/ethyl acetate in the order of increasing polarity, affording 6.0 mg of compound **1**, 10.0 mg of **2** and 5.2 mg of **4**. Fractions C and D were mixed on the basis of comparative TLC and purified in the gradient condition with the mixture of hexane/ethyl acetate affording 4.0 and 6.2 mg of compounds **6** and **7**, respectively. Also, 11.0 and 5.0 mg of compounds **8** and **9**, respectively, were obtained from the fraction E eluted with the mixture of MeOH and methylene chloride in the gradient condition.

3.2.1.1. *17-O-triacontanoylheptadecanal (**1**)*. White powder, m.p 83.7–84.0°C, IR 2918, 2848, 1733 and 1465 cm^{-1}; HR–EI–MS *m*/*z*: 704.7057 [calcd for $C_{47}H_{92}O_3$, 704.7046]; LR–EI–MS: Scheme 2; NMR data: ^{1}H-NMR (400 MHz, Pyridin-d_5): 9.94 (1H, s, H-1), 2.59 (2H, t, 6.5, H-2), 1.42 [80H, br s, H-(3–16, 4′–29′)], 1.88 (2H, m, H-17), 4.36 (2H, t, 6.2, H-18), 2.50 (2H, br t, 6.7, H-2′), 1.72 (2H, m, H-3′) and 1.01 (3H, t, 6.7, H-30′); ^{13}C-NMR (100 MHz, Pyridin-d_5): 202.6 (C-1), 34.9 (C-2), 23.0–33.9 (C-3), 26.4 (C-17), 64.9 (C-18), 173.5 (C-1′), 44.0 (C-2′), 29.2 (C-3′) and 14.1 (C-30′).

3.2.1.1.1. *Methanolysis of compound **1***
In this method, 1.5 mg of **1** was refluxed (70°C) for 18 h in 2.5 mL of MeOH containing 1.5 mL of 0.9 N HCl under magnetic stirring. The mixture was neutralised with an aqueous solution of $NaHCO_3$ and extracted with hexane. The fatty acid methyl ester was carefully characterised by EI–MS at *m*/*z* 466 corresponding to $[C_{31}H_{62}O_2]^{\bullet+}$.

3.2.1.2. *Compounds **2–9***. The NMR and some physical data are reported as supplementary data.

3.3. *Antimicrobial assay*

The crude extract, compound **1**, compound **9** and the reference antibiotics were analysed for their antimicrobial activities using the XTT colorimetric assay performed according to Eloff (1998) as modified by Kuete, Mbaveng, et al. (2008). The antibacterial activities against *Staphylococcus aureus* (ATCC 25922), *Salmonella typhi* (ATCC 6539), *Pseudomonas aeruginosa* (PAO1), *E. coli* (ATCC 10536), *K. pneumoniae* ATCC 29916, *Enterobacter aerogenes* ATCC 13048 and the antifungal activities against *C. albicans* (ATCC 9002) were carried out, with ciprofloxacin (Sigma–Aldrich, St. Quentin Fallavier, France) and nystatin (Sigma), respectively, as positive control.

4. Conclusions

The ethnopharmacological uses of some plant species sometimes bring information about the expected biological activities. Thus, *Mimosa invisa* could enter in the list of plants of which the biological activity corroborates with the traditional use, although the significant activity of its crude extract is a synergic effect of phytocomponents. Therefore, its traditional use against lung diseases could be attributed to flavonoids since some species rich in this class of compounds display antitussive activities (Garcia, Arts, Sterne, Thompson, & Shaheen, 2005). The expectorant effect of some sterols and triterpenoids was further reported (Gairola, Gupta, Bansal, Singh, & Maithani, 2010), which matches with its employment as anti-asthmatic. This plant appears to have a wide spectrum of biological activities since some known compounds obtained were reported to be endowed with anticancer (Cichewicz & Kouzi, 2004), hypoglycaemic (Panda, Jafri, Kar, & Meheta, 2009) and analgesic properties (Villaseñor, Angelada, Canlas, & Echegoyen, 2002).

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