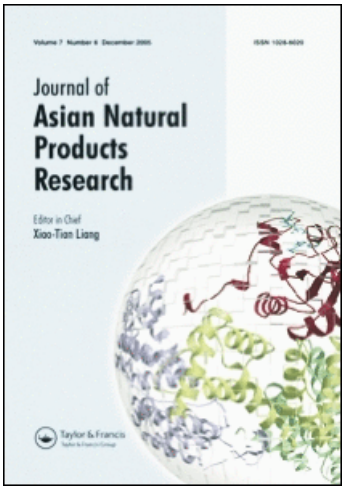

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Ozocardic A: a new alkylanacardic acid from *Ozoroa pulcherrima*

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One new alkylanacardic acid, ozocardic A (**1**), along with the known and related metabolites 6-tridecyl anacardic acid (**2**) and β-sitosterol (**3**) was isolated from *Ozoroa pulcherrima*. The structure of the new compound was elucidated by detailed spectroscopic analysis such as ^{1}H, ^{13}C NMR, COSY, HMQC, HMBC, and HREIMS. The structures of known compounds (6-tridecyl anacardic acid (**2**) and β-sitosterol (**3**)) were identified by the comparison of their spectral data with those published in the literature.

Keywords: alkylanacardic acids; *Ozoroa pulcherrima*; Anacardiaceae; ozocardic A

1. Introduction

Higher plants, as sources of medicinal compounds, continue to play a dominant role in maintenance of human health since antiquities. The African continent is one of the continents endowed with the richest biodiversity in the world, with an avalanche of many food plants used as herbs, health foods, and for therapeutic purposes [1]. This is largely due to the geographical spread, spanning a land mass of approximately 216,634,000 hectares of closed forest areas. Over 5000 different species of plant substances have been recognized to occur in these areas, and many of them have been found to be useful in traditional medicine for prophylaxis and cure of diseases [1]. This great biodiversity, therefore, offers economic promise in the rapidly emerging biotechnology industry. In spite of the heterogeneous nature of the continent and a deluge of information on the composition and biological activity of many plant substances, there has been little effort devoted to the phytochemical investigation. In view of the emerging competitive world, therefore, the evaluation of the constituents, pharmacological properties, and detailed screening of bioactive substances for chemotherapeutic purposes are urgently warranted. In the course of phytochemical studies of medicinal plants from Africa [2–8], we investigate *Ozoroa pulcherrima* (Anacardiaceae) and report on the structural elucidation of one new alkylanacardic acid, named ozocardic A (**1**), along with the known metabolites 6-tridecyl anacardic acid (**2**) and β-sitosterol (**3**). Here, we describe the isolation and structural elucidation of these compounds.

2. Results and discussion

O. pulcherrima (Anacardiaceae) was extracted with $MeOH–CH_2Cl_2$. The

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crude extract was fractionated on a silica gel column and yielded pure compounds **1**–**3**. The structures were elucidated by careful spectroscopic analysis (Figure 1).

Ozocardic A (**1**) was obtained as an amorphous powder. The UV spectrum exhibited absorption maxima at 224 and 274 nm. This was supported by IR bands at 1606 cm^{-1} for aromatic group and a broad signal at 3200 cm^{-1} for hydroxyl group. A $[M]^+$ peak at *m/z* 474.4068 in the HREIMS, along with analysis of ^1H, ^{13}C NMR, and DEPT spectra, showed a molecular formula of $C_{31}H_{54}O_3$, indicating five degrees of unsaturation. The ^1H NMR spectrum of compound **1** exhibited a simple pattern including the aromatic signals at δ 7.38 (1H, t, $J = 8.0$ Hz, H-4), 6.89 (1H, dd, $J = 8.0$, 2.0 Hz, H-3), and 6.79 (1H, dd, $J = 8.0$, 2.0 Hz, H-5), and the ^{13}C NMR and DEPT spectra exhibited three doublet CH-signals at δ 115.8 (C-3), 135.2 (C-4), 122.6 (C-5) and three C-signals at δ 110.2 (C-1), 163.6 (C-2), 147.5 (C-6), for a 1,2,6-trisubstituted benzene moiety. The ^1H NMR spectrum of **1** also showed a peak at δ 11.17 for chelated hydroxyl peak resulting from chelation with $-CO_2H$ group. The presence of $-CO_2H$ group was further confirmed from its peak at δ 174.0 in ^{13}C NMR spectrum. The spectrum further showed a triplet signal of the benzylic methylene at δ 2.97 and the corresponding carbon signal at δ 36.5 (C-1′). Moreover, H-1′ showed HMBC correlations with C-6, C-5, and C-1 (Figure 2). The ^1H NMR spectrum also showed a broad singlet at δ 1.30 for several methylenes in the molecule and a triplet at δ 0.91 for aliphatic methyl group.

The protonated carbons and their bonded protons were unambiguously assigned by the HMQC experiment. In the ^1H–^1H COSY spectrum (Figure 2), the protons at δ 6.89 (H-3) and 6.79 (H-5) were coupled with the proton at δ 7.38 (H-4), and the proton at δ 7.38 (H-4) was coupled with the protons at δ 6.89 (H-3) and 6.79 (H-5), respectively.

In the HMBC spectrum (Figure 2), the proton signal at δ 6.89 (H-3) correlated with carbon signals at δ 110.2 (C-1), 163.6 (C-2), 135.2 (C-4), and 122.6 (C-5); the proton signal at δ 7.38 (H-4) correlated with carbon signals at δ 163.6 (C-2), 115.8 (C-3), 122.6 (C-5), and 147.5 (C-6); the

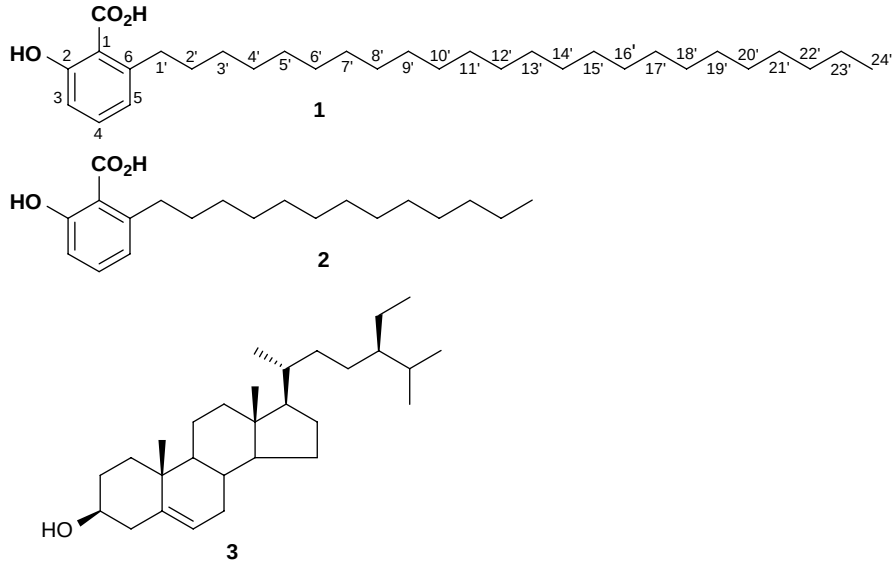

Figure 1. Chemical structures of compounds **1**–**3**.

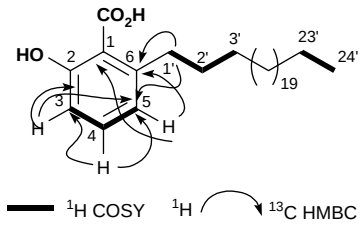

Figure 2. Key COSY and HMBC correlations for ozocardic A (**1**).

proton signal at δ 6.79 (H-5) correlated with carbon signals at δ 110.2 (C-1), 115.8 (C-3), 135.2 (C-4), and 147.5 (C-6). These data accounted for all ^{1}H and ^{13}C NMR resonances. HREIMS required the compound to be monocyclic, considering the subtraction of four double bond equivalents for one carbonyl group and three aromatic double bonds. The above spectral evidences gave indication for the alkylanacardic acid nature of the molecule [9–11]. The length of the side chain was deduced from the molecular ion peak in the mass spectrum (EIMS and HREIMS). The multiplicities of carbons were determined by the DEPT experiment, and the complete assignments were made on the basis of COSY-45, HMQC, and HMBC techniques (Figure 2), and the structure of ozocardic A (**1**) was established as 2-hydroxy-6-tetracosylbenzoic acid which is a new compound.

The known compound was readily identified as 6-tridecyl anacardic acid (**2**) [12] and β-sitosterol (**3**) [13] by the analysis of their NMR spectra and by the comparison with the data reported in literature.

3. Experimental

3.1 General experimental procedures

IR spectra were recorded on a Nicolet-510P spectrophotometer; ν_{max} in cm^{-1}. EI-MS and HREIMS were carried out using MAT 8200 and Micromass LCT mass spectrometers in *m/z*. The ^{1}H NMR spectra were recorded on Bruker AMX-500 instruments using TMS as an internal reference. The chemical shifts are reported in parts per million (δ), while the coupling constants (*J*) in Hertz. The ^{13}C NMR spectra were recorded at 125 MHz on the same instrument.

Column chromatography (CC) was carried out using silica gel (70–230 and 230–400 mesh; E-Merck, Darmstadt, Germany) and Sephadex LH-20 (Amersham Biosciences AB, Uppsala, Sweden). Aluminum sheets precoated with silica gel 60 F 254 (0.2 mm thick; E-Merck) were used for TLC to check the purity of the compounds and were visualized under UV light (254 and 366 nm), followed by ceric sulfate as the spray reagent.

3.2 Plant material

The roots of *O. pulcherrima* Schweinf (Anacardiaceae) were collected near Ngaoundere, about 10 km from Nganha in the Adamawa region of Cameroon, during May 2006, and identified by Mr Elias Ndive (plant taxonomist). A voucher specimen (N0 1905/SRF/CAM) has been deposited at the National Herbarium Yaoundem Cameroon.

3.3 Extraction and isolation

The roots of *O. pulcherrima* Schweinf (Anacardiaceae) were macerated in MeOH–CH_2Cl_2 at room temperature for 48 h and then filtered. The filtrate was concentrated under vacuum to give 89 g of the crude residue. The crude fraction (89 g) was then subjected to CC (silica gel, *n*-hexane, *n*-hexane–EtOAc and EtOAc, MeOH in order of increasing polarity) yielding four fractions. Fraction F_3 was eluted with a mixture of *n*-hexane–acetone (0.5:9.5) yielding ozocardic A (**1**) (6.0 mg) and 6-tridecyl anacardic acid (**2**) (7.0 mg). Finally, fraction F_2 gave β-sitosterol (**3**, 17 mg) on subjecting it to CC using *n*-hexane–EtOAc (8.5:1.5) as eluent.

3.3.1 Ozocardic A (**1**)

White solid. UV (CH_2Cl_2) λ_{max} (log ε) nm: 224 (3.71), 274 (2.10). IR (KBr) ν_{max}: 3200, 2963, 1703, 1697, 1606, 1465, and 1061 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$): 0.91 (3H, t, $J = 7.4$ Hz, H-24′), 1.60 (2H, m, H-2′), 1.30 (m, $n \times CH_2$), 2.97 (2H, t, $J = 8.0$ Hz, H-1′), 6.79 (1H, dd, $J = 8.0$, 2.0 Hz, H-5), 6.89 (1H, dd, $J = 8.0$, 2.0 Hz, H-3), 7.38 (1H, t, $J = 8.0$ Hz, H-4), 11.17 (OH-2). ^{13}C NMR (125 MHz, $CDCl_3$): 174.0 (CO_2H), 163.6 (C-2), 147.5 (C-6), 135.2 (C-4), 122.6 (C-5), 115.8 (C-3), 110.2 (C-1), 36.5 (C-1′), 32.0 (C-2′), 29.8–26.7 ($n \times CH_2$), 22.6 (C-22′), 14.1 (C-13′). EIMS: *m/z* (%) 474.3 (17) $[M]^+$. HREIMS: *m/z* 474.4068 $[M]^+$ (calcd for $C_{31}H_{54}O_3$, 474.4073).

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