

Isoflavonoid Derivatives from *Lophira alata* Stem Heartwood

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Six isoflavonoid derivatives among which three are new have been isolated from the stem heartwood of *Lophira alata*. The structures were elucidated from spectroscopic and chemical evidences. Two have unusual carbon skeletons, possibly resulting from a variant of isoflavonoid biogenesis. The two compounds form the first members of a new subclass of flavonoid compounds which we call “isobiflavonoids”. The presence of these isoflavonoid compounds in this plant of the Ochnaceae family has important chemotaxonomic implications since it modifies the botanic distribution of isoflavonoid compounds in non-leguminous plants.

Key words: *Lophira alata*, Ochnaceae, Isobiflavonoids

Introduction

The stem heartwood of *Lophira alata* Banks ex C. F. Gaertn. (Ochnaceae), also called “bon-gossi”, shows strong resistance against destructive fungi and bacteria and is extensively exploited as first-grade timber. Both aqueous and alcohol extracts of either the leaves, the stem bark or the stem heartwood of this plant are extensively used as antiseptics by many tribes in the central and southern regions of Cameroon. The observed antimicrobial properties of the MeOH extract of its stem bark (Malcolm and Sofowora, 1969; Murakami *et al.*, 1991) initiated its phytochemical investigation that led to the characterization of the antimicrobial biflavonoids mbamichalcone (Tih *et al.*, 1988) and bongosin (Tih *et al.*, 1990), the tetraflavonoids lophirachalcone, lophiraflavan A (Tih *et al.*, 1992a), lophiraflavans B and C (Tih *et al.*, 1992b), and the hexaflavonoid azobechalcone (Tih *et al.*, 1999). Recently, investigation of the MeOH leaf extract gave luteolin, lithospermoxide (Tih *et al.*, 2003), and lophirones L and M (Tih *et al.*, 2006). The women of the Bafia and the Yambassa tribes living in the Ombessa district of the Mbam division in the centre region of Cameroon use the aqueous extract of the stem heartwood for gynaecological cleansing. In order to understand the antiseptic action of this extract, we have

extended our phytochemical investigation to the stem heartwood methanol extract of this plant from which we have isolated and characterized, using spectroscopic and chemical evidences, six isoflavonoid derivatives of which three are new.

Results and Discussion

The MeOH extract of the stem heartwood purified by multiple chromatographic steps, first over Sephadex LH20 and then over silica gel, gave six isoflavonoid derivatives. Their chemical structures were established from spectroscopic (IR, UV, MS, 1D and 2D NMR, HSQC, HMBC, and NOE) and chemical evidences. Three of these compounds, lophirones N (**2**) and O (**5**) and mbamiloside A (**4**), are novel with important structural particularities and chemotaxonomic significations. The previously described lophirone A (**1**) was identified (Ghogomu *et al.*, 1987) by comparing its spectral data with those of a reference sample isolated earlier from the stem bark of *Lophira lanceolata* while genistein and 2'-hydroxygenistein reported for the first time in this species were identified by comparing their spectra with those published (Pelter *et al.*, 1978; Lane and Newman, 1987).

Lophirone N (**2**) was obtained as an amorphous beige solid of the molecular formula C₃₀H₂₂O₉

determined by HRMS measurements (found 526.1258, calcd. for $C_{30}H_{22}O_9$ 526.1264). With a molecular mass of 526, it has 20 unsaturated sites and gave a positive phenol test. Its IR spectrum was very similar to that of lophirone A (**1**) as it presented absorption bands for the same functional groups: hydroxy (3252 cm^{-1}), conjugated carbonyl (1692 and 1648 cm^{-1}), and conjugated double bond (1624 cm^{-1}). The 400-MHz 1D ^1H NMR spectrum displayed similar signals observed for most of the protons found in the ^1H NMR spectrum of lophirone A (**1**). Cross peaks in the 2D COSY ^1H - ^1H NMR spectrum of **2** defined these protons as follows: three residual protons on ring B' at δ 6.76 ppm (1H, d, $J = 2.1\text{ Hz}$, H-8'''), δ 6.91 ppm (1H, dd, $J = 8.1$ and 2.1 Hz , H-5'''), and δ 8.33 ppm (1H, d, $J = 8.1\text{ Hz}$, H-6'''); two disubstituted benzene rings B and A, each bearing four protons. Protons on ring B had signals at δ 6.60 ppm (2H, d, $J = 8.9\text{ Hz}$, H-3' and H-5') and at δ 7.25 ppm (2H, d, $J = 8.9\text{ Hz}$, H-2' and H-6'), while those on ring A' appeared at δ 6.66 ppm (2H, d, $J = 9.1\text{ Hz}$, H-3'' and H-5'') and at δ 7.22 ppm (2H, d, $J = 9.1\text{ Hz}$, H-2'' and H-6''). The lone proton on ring C gave a singlet signal at δ 8.25 ppm (1H, s, H-2), while the remaining two aliphatic protons formed an AB system with a chemical shift of δ 4.99 ppm (1H, d, $J = 12.0\text{ Hz}$, H- β) and δ 6.14 ppm (1H, d, $J = 12.0\text{ Hz}$, H- α). The main difference observed between the spectra of both compounds was the replacement of the signals of the three protons on ring A which originally appeared at δ 6.77 ppm (1H, d, $J = 2.3\text{ Hz}$, H-8), 6.91 ppm (1H, dd, $J = 8.8$ and 2.3 Hz , H-6), and 7.94 ppm (1H, d, $J = 8.0\text{ Hz}$, H-5) in the ^1H NMR spectrum of **1** by those of a different three-proton system appearing at δ 6.21 ppm (1H, d, $J = 1.8\text{ Hz}$, H-8), 6.37 ppm (1H, d, $J = 1.8\text{ Hz}$, H-6), and 13.04 ppm (1H, s, H-5) in the ^1H NMR spectrum of lophirone N (**2**). This suggests that the proton H-5 at δ 7.94 ppm and *peri* to the carbonyl group in **1** has been replaced by a chelated phenolic hydroxy group at δ 13.04 ppm in compound **2**. This should imply that lophirone N has six phenolic groups on the same carbon skeleton like lophirone A (**1**). The confirmation was obtained from the ^1H NMR spectral data of compound **3**, the completely acetylated (Ac_2O /pyridine) derivative of compound **2**, which has the molecular formula $C_{42}H_{34}O_{15}$ determined by HRMS measurements and showed no residual hydroxy absorption in its IR spectrum. The mass difference of 252 amu no-

ticed between the molecular mass of compound **2** and that of compound **3** implies that all six OH groups were transformed to six CH_3COO groups during acetylation. This was also confirmed by the ^1H NMR spectrum of **3** which displayed sharp singlet signals for six CH_3COO groups at δ 2.12 (3H, s), 2.19 (6H, s), 2.21 (3H, s), 2.28 (3H, s), and 2.29 ppm (3H, s), hence confirming the isobiflavonoid structure **2** for lophirone N. The analysis of the HSQC and HMBC spectra of compounds **2** and **3** led to the complete assignment of the carbon atom resonances in both compounds thus confirming the carbon skeleton of compound **2**.

Compound **4**, obtained as a yellow amorphous powder, is a phenolic glycoside since it gave positive phenol and Molish tests. A molecular mass of 448 and a molecular formula of $C_{21}H_{20}O_{11}$ were assigned to compound **4** from the data established from high-resolution CI-MS in which the $[\text{M}+\text{H}]^+$ ion was observed at m/z 449.1076. Acid hydrolysis of compound **4** gave an aglycone and a sugar which we identified, respectively, as the isoflavonoid 2'-hydroxygenistein and the sugar glucose by comparison of their IR, MS, NMR, and UV spectra with those of reference samples. This evidence implies that compound **4** is a glucoside of 2'-hydroxygenistein. Proof that the anomeric carbon hydroxy group of glucose is linked to the 2' position of ring B of the aglycone came from NOE difference measurements. Irradiation of the anomeric proton at δ 5.15 ppm (H-1'') gave NOE difference values of 4%, 0%, and 9%, respectively, with each of the aglycone protons at δ 8.03 (H-2), 6.74 (H-6), and 6.51 ppm (H-3'), respectively. The large coupling constant ($J = 8\text{ Hz}$) between the anomeric sugar proton H-1'' and that adjacent, H-2'', confirms the β -configuration of the glucoside bond implying that compound **4** is the isoflavonoid derivative 2'-*O*- β -D-glucoside of 2'-hydroxygenistein for which we have given the name mbamiloside A. The complete attribution of the carbon resonances was made using its HMQC and HMBC spectra and the results are in good agreement with the carbon skeleton of structure **4** (Fig. 1).

Lophirone O (**5**), obtained as a yellow non-crystalline powder, gave no reaction with FeCl_3 solution, confirming the absence of any phenolic function in its structure. HRMS established a molecular mass of 622 and a molecular formula of $C_{40}H_{30}O_7$ (found 622.1986, calcd. 622.1991) for this compound. Its UV spectrum showed absorp-

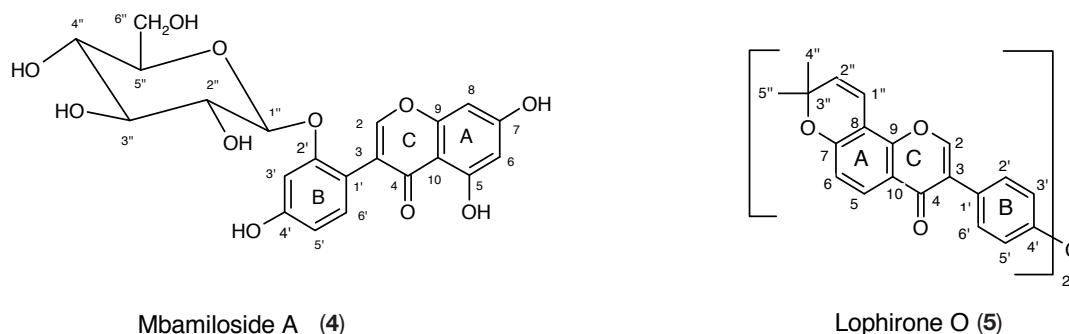


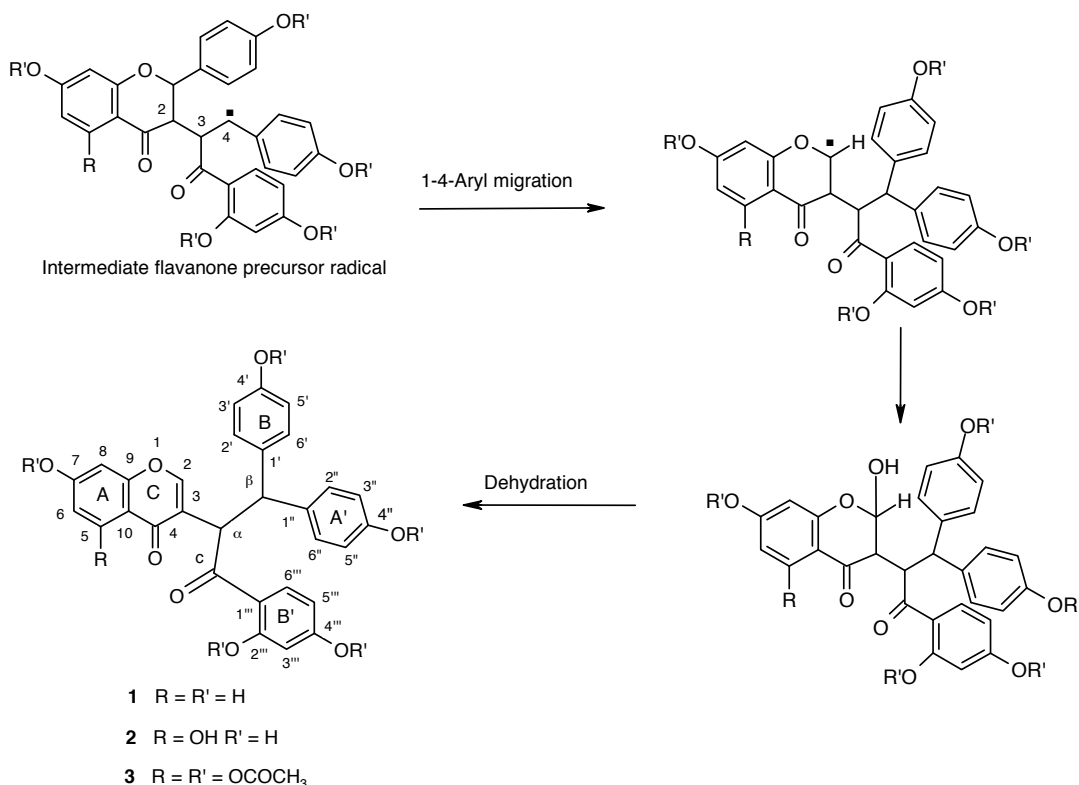
Fig. 1. Chemical structure of mbamiloside A (4) and lophirone O (5).

tion bands at 261 and 320 nm suggesting the presence of an isoflavonoid nucleus (Hideyuki *et al.*, 2001). From the careful analysis of the 400-MHz ^1H and 100-MHz ^{13}C NMR spectra of compound **5**, it was noticed that signals of only half the number of protons and carbon atoms, respectively, required by the molecular formula were observed. This suggests that lophirone O must have a symmetric structure. From its 1D and 2D COSY ^1H - ^1H NMR spectra, it was established that the structure of compound **5** has the following proton systems: four protons on the disubstituted ring B at δ 7.21 ppm (2H, d, J = 8.6 Hz, H-2 and H-6) and δ 6.70 ppm (2H, d, J = 8.6 Hz, H-3 and H-5); the H-2 proton on ring C appearing at δ 7.21 ppm (1H, s, H-2); an AB system of two *ortho*-coupled protons on ring A at δ 6.81 ppm (1H, d, J = 8.4 Hz, H-6) and 8.32 ppm (1H, d, J = 8.4 Hz, H-5); and the characteristic signals of the 2,2-dimethylchromene protons at δ 6.13 ppm (1H, d, J = 10.0 Hz, H-2''), 6.32 ppm (1H, d, J = 10.0 Hz, H-1''), and 1.36 ppm (6H, s, CH_3 -4 and CH_3 -5). The presence of the *ortho*-coupled protons H-5 (δ 8.32 ppm) and H-6 (δ 6.81 ppm) on ring A unequivocally locates the C-phenyl substituent at the position 8 of ring A. This substituent must have undergone an oxidative cyclization involving the hydroxy group at the 7 position of ring A to give the chromene ring. This information leads to the establishment of the structure of only exactly one half of the molecule in which only four out of seven oxygen atoms required by the molecular formula of lophirone O are implicated. For a symmetric structure, the second half of the molecule can only be linked to this first substructure by an ether bond using the oxygen atom at the position 4' of ring B, thus leading to structure **5** (Fig. 1). The complete assignment of the chemical shifts

of the carbon atoms in **5** was made after a careful study of its HSQC and HMBC spectra, thus confirming the carbon skeleton. Lophirone O is thus a novel symmetric di-prenylated isoflavonoid ether with structure **5**, which adds new structural diversity to *Lophira alata*.

Both lophirones A (**1**) and N (**2**) have a unusual sequence of carbon atoms in their flavonoid skeleton which neither corresponds to that of biflavonoids nor to that of biisoflavonoids. This shows a natural deviation of the known isoflavonoid biosynthetic pathway, thus leading to a new subclass of biflavonoids which we call isobiflavonoids. This subclass has only **1** and **2** as representatives until now. It is possible that this transposed skeleton may result from an 1,4-aryl group migration similar to that of the 1,2-aryl shift in isoflavonoid biogenesis (Scheme 1).

These results modify the isoflavonoid distribution in non-leguminous plants because, despite of the large structural diversity of isoflavonoids, their distribution has mainly been restricted to leguminous plants, especially in the subfamily Papilionoideae (Dewick, 1994). In other subfamilies like Caesalpinoideae and Mimosoideae as well as in non-leguminous plants, isoflavonoid derivatives have been very rarely found and described only in a few isolated plant genera belonging to the families Chenopodiaceae, Compositae, Moraceae, Iridaceae, Cupressaceae, Podocarpaceae, and Bryaceae (Mackova *et al.*, 2006). Earlier reports on two isoflavonoids from the genus *Ochna* (Messanga *et al.*, 1992, 1998) raised some doubts from our local botanists. The compounds described here add to those described earlier in the genus *Ochna* to confirm that the Ochnaceae family is also one of the sources of isoflavonoids in non-leguminous plants.



Scheme 1. Possible biogenesis pathway for lophirone A (**1**) and lophirone N (**2**).

Experimental

General experimental procedures

UV spectra were determined with a Kontron-Uvikon 930 spectrophotometer (San Diego, CA, USA). IR spectra were recorded using transparent KBr discs on a JASCO FTIR-3000E spectrometer (Tokyo, Japan). Optical rotation was measured on a Perkin-Elmer 341 polarimeter (Überlingen, Germany). The 400-MHz ¹H and 100-MHz ¹³C NMR spectra were recorded on a Bruker WM400 spectrometer (Rheinstetten, Germany) with compounds dissolved in CD₃COCD₃. Chemical shifts are expressed in δ (ppm), with reference to TMS, and coupling constants (*J*) in Hertz. For HMBC spectra, the delay was 70 μ s with $J_{CH} \sim 7$ Hz. Cims was carried out on a Riber Nermag V_{3.0} instrument (Rueil-Malmaison, France), with NH₃ as ionizing gaz. EI mass spectra were recorded on a JEOL JMS-110 spectrometer (Tokyo, Japan). Sephadex LH20 (Pharmacia, Uppsala, Sweden) and Kieselgel 60 (0.063D, 0.200 mm; Merck, Darmstadt, Germany) were used for column chromatography. Thin

layer chromatography (TLC) was carried out on aluminium sheets precoated with fluorescent silica gel 60 F₂₅₄ (Merck) and developed in a CH₂Cl₂/MeOH mixture (10:1, v/v). Chromatograms were visualized by spraying TLC plates with 3% H₂SO₄ solution, followed by heating in an oven at 60 °C for 10 min. Preparative TLC plates on glass support were prepared using fluorescent silica gel (F₂₅₄), and after applying the mixture, they were developed in the same eluent mixture as above. Bands resulting from the separation were visualized using a UV lamp at 254 nm, scraped off after, and finally eluted with methanol to obtain the pure compounds.

Plant material

The stem heartwood of *Lophira alata* Banks ex C. F. Gaertn. (Ochnaceae) was harvested in March 2006 in the Ombessa district in the Mbam division of the centre region of Cameroon and was identified by Victor Nana, botanist at the National Herbarium Yaounde, Cameroon where a voucher specimen (no. VN 1895) is deposited.

Extraction and isolation

The stem heartwood was cut into small pieces, sundried, and ground into fine powder (0.8 kg) which was extracted with methanol in a Soxhlet extractor. The removal of the solvent gave a dark brown gum (106 g) which was washed with hot ethyl acetate. The EtOAc-soluble fraction was evaporated to dryness to give a brown crude extract (38 g) which was fractionated by gel permeation column chromatography on a Sephadex LH20 column with MeOH as eluent to give five fractions: F1 (22.3 g), F2 (11.1 g), F3 (2.4 g), F4 (1.4 g), and F5 (0.8 g). First, column chromatography of F4 over silica gel eluted with a gradient mixture of CH_2Cl_2 and MeOH gave 54 fractions of 30 ml each which were recombined following TLC analysis into four subfractions: F4a (738 mg), F4b (282 mg), F4c (198 mg), and F4d (164 mg). Fraction F4b subsequently purified by preparative TLC on silica gel plates, using the technique of multiple migration with the mixture $\text{CH}_2\text{Cl}_2/\text{MeOH}$ (10:1, v/v) as eluent, gave two pure compounds, lophirone A (**1**) (124 mg) and lophirone N (**2**, 68 mg). This purification procedure repeated with fraction F4c gave more lophirone A (92 mg), mbamiloside A (**4**, 42 mg) and 2'-hydroxygenistein (28 mg). The last fraction F4d also subjected to the same purification procedure gave genistein (26 mg) and lophirone O (**5**, 18 mg).

Acetylation of **2**

Compound **2** (8 mg) was dissolved in a mixture of Ac_2O (2 ml) and anhydrous pyridine (2 ml) placed in a 10-ml round-bottom flask. The well-corked flask was placed in an oven at 80 °C for 2 h after which it was allowed to cool and evaporate to dryness under vacuum. The residual solid obtained was purified on an LH20 Sephadex column with MeOH as eluent to give lophirone N hexaacetate (**3**, 6 mg) as a white amorphous powder.

Acid hydrolysis of **4**

Compound **4** (10 mg) was dissolved in 5 ml 30% HCl, placed in a 25-ml round-bottom flask and heated under reflux at 80 °C for 2 h in a water bath. The reaction mixture was allowed to cool, 5 ml of water were added, and the reaction medium was extracted with CH_2Cl_2 (10 ml x 3). The combined CH_2Cl_2 extracts were evaporated to afford the aglycon, which was identified as

2'-hydroxygenistein by comparing its spectra with those in the literature. Acetone was added to the aqueous layer until a white precipitate was deposited, which was further filtered, washed with more acetone, dried, and identified as glucose by comparing its IR and NMR spectra with those of a commercial reference sample.

Spectroscopic data

Lophirone N (2): Amorphous beige powder. – $[\alpha]_{\text{D}}^{20} +48^\circ$ (c 0.24, MeOH). – UV (MeOH): λ_{max} (log ϵ) = 264 nm (4.28). – IR (KBr): ν_{max} = 3252, 1692, 1648, 1624 cm^{-1} . – ^1H NMR (CD_3COCD_3 , 100 MHz): δ = 4.99 (1H, d, J = 12.0 Hz, H- β), 6.14 (1H, d, J = 12.0 Hz, H- α), 6.21 (1H, d, J = 1.8 Hz, H-8), 6.37 (1H, d, J = 1.8 Hz, H-6), 6.60 (2H, d, J = 8.9 Hz, H-3' and H-5'), 6.66 (2H, d, J = 9.1 Hz, H-3'' and H-5''), 6.77 (1H, d, J = 2.3 Hz, H-8), 6.92 (1H, dd, J = 8.8 and 2.3 Hz, H-6), 7.22 (2H, d, J = 9.1 Hz, H-2'' and H-6''), 7.25 (2H, d, J = 8.9 Hz, H-2' and H-6'), 7.94 (1H, d, J = 8.0 Hz, H-5), 8.25 (1H, s, H-2), 8.33 (1H, d, J = 8.0 Hz, H-5), 13.04 (1H, s, H-5). – ^{13}C NMR (CD_3COCD_3 , 100 MHz): δ = 43.4 (C- α), 53.9 (C- β), 94.3 (C-8), 98.1 (C-6), 104.1 (C-3'''), 104.6 (C-10), 108.3 (C-5'''), 114.5 (C-1'''), 115.4 (C-3'), 115.4 (C-5'), 116.1 (C-3''), 116.1 (C-5''), 122.3 (C-3), 129.9 (C-2'), 129.9 (C-2''), 129.9 (C-6''), 129.9 (C-6'), 133.9 (C-6'''), 135.1 (C-1'), 135.2 (C-1''), 155.8 (C-4'), 156.2 (C-4''), 156.7 (C-2), 157.9 (C-9), 162.4 (C-5), 164.2 (C-7), 165.9 (C-4'''), 166.4 (C-2'''), 178.3 (C-4), 203.8 (C-c). – EI-HRMS: m/z = 526.1258 (calcd. for $\text{C}_{30}\text{H}_{22}\text{O}_9$ 526.1264).

Lophirone N hexaacetate (3): Amorphous white powder. – $[\alpha]_{\text{D}}^{20} +52^\circ$ (c 0.16, MeOH). – UV (MeOH): λ_{max} (log ϵ) = 269 nm (4.6). – IR (KBr): ν_{max} = 3048, 2923, 2848, 1740, 1690, 1644 cm^{-1} . – ^1H NMR (CD_3COCD_3 , 400 MHz): δ = 2.12 (3H, s), 2.19 (6H, s), 2.21 (3H, s), 2.28 (3H, s), 2.29 (3H, s), 6.13 (1H, d, J = 12.0 Hz, H- α), 4.96 (1H, d, J = 12.0 Hz, H- β), 6.56 (1H, d, J = 2.3 Hz, H-8), 6.88 (1H, d, J = 2.3 Hz, H-6), 6.91 (1H, d, J = 2.1 Hz, H-3''), 7.00 (4H, d, J = 9.0 Hz, H-3', H-5', H-3''', H-5'''), 7.13 (1H, dd, J = 2.1 and 8.9 Hz, H-5''), 7.53 (4H, d, J = 9.0 Hz, H-2', H-6', H-2'', H-6''), 7.63 (1H, d, J = 8.9 Hz, H-6''), 8.34 (H, s, H-2). – ^{13}C NMR (CD_3COCD_3 , 100 MHz): δ = 44.4 (C- α), 53.1 (C- β), 94.8 (C-8), 99.6 (C-6), 104.3 (C-3''), 104.8 (C-10), 108.1 (C-5''), 114.3 (C-1''), 115.4 (C-3'), 115.4 (C-5'), 116.4 (C-3'''), 122.8 (C-3), 123.1 (C-2'''), 123.1 (C-6'''), 129.5 (C-2'), 129.5 (C-6'),

134.6 (C-1'''), 135.4 (C-1'), 123.1 (C-5'''), 133.9 (C-6''), 156.6 (C-4'''), 156.8 (C-4'), 156.9 (C-2), 158.4 (C-9), 162.2 (C-5), 166.2 (C-4''), 166.4 (C-2''), 168.4 (C-7), 175.3 (C-4), 203.4 (C-c). – EI-HRMS: m/z = 778.1892 (calcd. for $C_{42}H_{34}O_{15}$ 778.1898).

Mbamiloside A (4): Amorphous yellow powder. – UV (MeOH): λ_{\max} (log ϵ) = 281 (4.13); + $AlCl_3$: 285 (4.14); + $NaOAc$: 283 nm (4.13). – IR (KBr): ν_{\max} = 3285, 3048, 1636 cm^{-1} . – 1H NMR (CD_3COCD_3 , 400 MHz): δ = 3.19 (1H, dd, J = 8.8 and 7.0 Hz, H-2''), 3.26 (1H, ddd, J = 8.8, 5.3 and 2.1 Hz, H-5''), 3.28 (1H, t, J = 8.8 and 8.8 Hz, H-3''), 3.37 (1H, t, J = 8.8 and 8.8 Hz, H-4''), 3.51 (1H, dd, J = 11.5 and 5.3 Hz, H-6a''), 3.71 (1H, dd, J = 11.5 and 2.1 Hz, H-6b''), 5.15 (1H, d, J = 7.0 Hz, H-1''), 6.39 (1H, d, J = 2.4 Hz, H-8), 6.51 (1H, d, J = 2.1 Hz, H-3'), 6.69 (1H, dd, J = 8.9 and 2.1 Hz, H-5'), 6.74 (1H, d, J = 2.4 Hz, H-6), 7.38 (1H, d, J = 8.9 Hz, H-6'), 8.03 (1H, s, H-2). – ^{13}C NMR (CD_3COCD_3 , 100 MHz): δ = 62.0 (C-6''), 70.7 (C-4''), 74.2 (C-2''), 77.2 (C-5''), 77.5 (C-3''), 94.1 (C-8), 99.4 (C-6), 102.2 (C-1''), 102.7 (C-3'), 106.6 (C-5'), 106.8 (C-1'), 121.1 (C-3), 132.4 (C-6'), 155.8 (C-9), 156.9 (C-2'), 156.9 (C-2), 158.9 (C-4'),

163.2 (C-5), 164.5 (C-7), 179.3 (C-4). – CI-HRMS: m/z = 449.1076 (calcd. for $C_{21}H_{21}O_{11}$ 449.1083).

Lophirone O (5): Amorphous yellow powder. – UV (MeOH): λ_{\max} (log ϵ) = 261 (4.41), 320 nm (2.8). – IR (KBr) = ν_{\max} 3050, 1642 cm^{-1} . – 1H NMR (CD_3COCD_3 , 400 MHz): δ = 1.36 (6H, s, CH_3 -4), 1.36 (6H, s, CH_3 -5), 6.13 (1H, d, J = 10 Hz, H-2''), 6.32 (1H, d, J = 10.0 Hz, H-1''), 6.70 (2H, d, J = 8.6 Hz, H-3 and H-5), 6.81 (1H, d, J = 8.4 Hz, H-6), 7.21 (2H, d, J = 8.6 Hz, H-2 and H-6), 7.23 (1H, s, H-2), 6.81 (1H, d, J = 8.4 Hz, H-5). – ^{13}C NMR (CD_3COCD_3 , 100 MHz): δ = 27.1 (C-4''), 27.1 (C-5''), 80.3 (C-3''), 109.0 (C-8), 116.2 (C-3'), 114.8 (C-6), 116.2 (C-5'), 118.4 (C-10), 125.1 (C-3), 126.1 (C-2''), 126.4 (C-5), 128.7 (C-2'), 128.7 (C-6'), 131.8 (C-1'), 131.8 (C-1''), 151.8 (C-2), 157.2 (C-7), 152.2 (C-9), 157.4 (C-4'), 175.4 (C-4). – EI-HRMS: m/z = 622.1986 (calcd. for $C_{40}H_{30}O_7$ 622.1991).

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