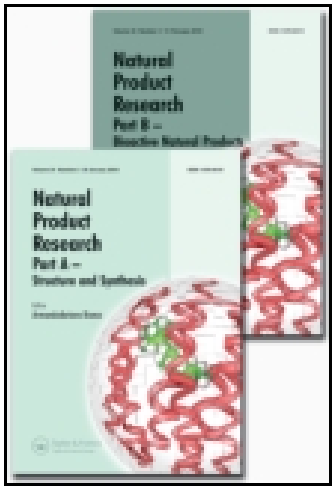

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Three new bioactive phenolic glycosides from Liparis odorata

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Three new bioactive phenolic glycosides from *Liparis odorata*

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Chemical investigation of the ethanol extract from the whole plant *Liparis odorata* (Willd.) Lindl (orchid family) has led to the isolation of three new phenolic glycosides named liparisglycoside A–C (**1–3**) along with six known compounds (**4–9**). The structures of compounds were elucidated by means of extensive spectroscopic data analysis and comparison with the literature data. In addition, compounds **3** and **9** revealed inhibitory effects on lipopolysaccharide-stimulated nitric oxide production, and compounds **1**, **3** and **9** displayed hypolipidaemic effects in *in vitro* bioassays.

Keywords: *Liparis odorata* (Willd.) Lindl; orchid family; phenolic glycosides; anti-inflammatory activity; hypolipidaemic effect

1. Introduction

Liparis odorata (Willd.) Lindl is a plant from the orchid family that has been reported to possess bioactivities, such as inhibiting inflammation, improving blood flow and dispelling dampness (Chinese Materia Medica Editorial Board of State Administration of Traditional Chinese Medicine 1999), and has been used as an anti-hyperlipidaemic treatment in Jiangxi folk medicine of China. However, there are currently few chemical or biological reports on this plant. Thus, to determine its bioactive constituents, our laboratory performed a thorough investigation of the ethanol extract for the first time. So far, three new phenolic glycosides named liparisglycoside A–C (**1–3**) (Figure 1), together with six known compounds, compound **4** (Huang et al. 2013), succinic acid (**5**) (Zhu et al. 2011), phloic acid (**6**) (Wen & Zhou 1998), batatasin III (**7**) (Chen et al. 2008) and luteolin (**8**) (Chai et al. 2004), derivative from enzymatic hydrolysis of the known compound anodendrosin A (**9**) (Abe & Yamauchi 1985) have been isolated. Preliminary evaluation of the effects of the compounds on lipopolysaccharide (LPS)-stimulated nitric oxide (NO) production and hypolipidaemic activities are also reported.

2. Results and discussion

The ethanol extract of *L. odorata* was fractionated by column chromatography and the fractions were purified by repeated column chromatography to yield compounds **1–9**.

Compound **1** was isolated as white needles. Its molecular formula was determined to be $C_{30}H_{44}O_{13}$ based on ^{1}H and ^{13}C NMR data. The pseudomolecular ion peak in the HRESI- MS at *m/z* 635.2785 $[M + Na]^+$ (calcd 635.2674) indicated nine degrees of unsaturation. The ^{13}C

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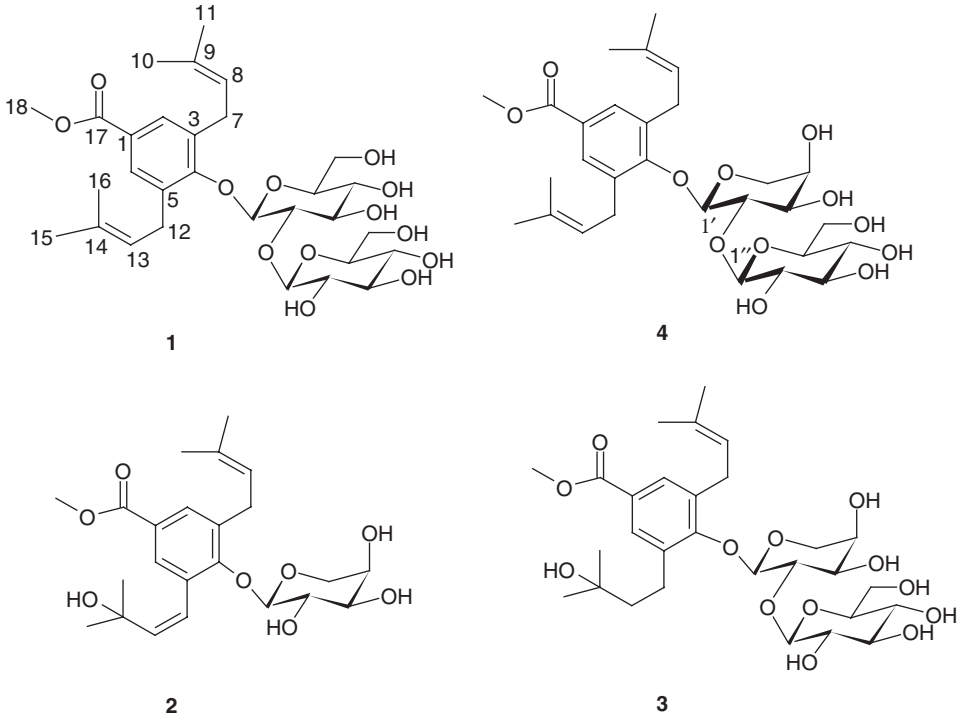

Figure 1. Structures of compound **1–4**.

NMR spectrum revealed 22 carbons, which was 8 carbons less than expected based on the molecular formula, revealing that compound **1** possessed a symmetrical structure. Based on the literature (Yu & Yang 1999), the ^{1}H NMR spectrum of **1** displayed two overlapping aromatic protons at δ 7.60 (s, H-2, 6), indicating the existence of a tetrasubstituted benzene ring, two overlapping olefinic protons at δ 5.23 (t, $J = 7.2$ Hz, H-8, 13), two methylene protons at δ 3.51 (d, $J = 7.2$ Hz, H-7, 12), two sets of methyl protons at δ 1.71 (s, H-10, 15) and 1.70 (s, H-11, 16), declaring there were two isopentenyl groups, as well as protons at δ 3.80 (s, H-18) showing one methoxy. The ^{13}C NMR, DEPT and HSQC spectra indicated two overlapping aromatic methyne carbons at δ 130.3 (C-2, 6), four quaternary aromatic carbons at δ 127.8 (C-1), 137.6 (C-3, 5) and 158.1 (C-4), two overlapping olefinic methyne carbons at δ 124.4 (C-8, 13), two quaternary olefinic carbons at δ 134.3 (C-9, 14), two overlapping methylene carbons at δ 29.9 (C-7, 12), two sets of overlapping methyl carbons at δ 26.3 (C-10, 15) and 18.5 (C-11, 16), one carbonyl resonance at δ 168.9 (C-17) and one methoxy carbon at δ 52.8 (C-18). In the ^{1}H–^{1}H COSY spectrum, the proton at δ 3.51 (H-7, 12) was coupled to the proton at δ 5.23 (H-8, 13) indicating that the two methylene groups were connected to two olefinic methynes. In HMBC, correlations were observed from H-7 to C-2, C-4, C-8, C-9 and from H-12 to C-4, C-6, C-13, C-14 indicating that there were two isopentenyl groups attached to a tetrasubstituted benzene ring at C-3 and C-5, respectively; correlations were observed from H-2, H-6 to C-17 and from H-18 to C-17 indicating that a methyl formate group was attached to C-1 of the benzene ring (Figure 2).

The ^{1}H and ^{13}C NMR spectra of **1** revealed two anomeric protons at δ 4.78 (d, $J = 7.5$ Hz, H-1′) and 4.76 (d, $J = 7.5$ Hz, H-1″), two anomeric carbons at δ 104.5 (C-1′) and 105.6 (C-1″). This indicated the presence of two sugar moieties, and D-glucose was identified by using GC–MS to analyse the trimethylsilyl ether derivatives of the acidic hydrolysis products of **1**. The HMBC correlation between the anomeric proton of one D-glucose and C-4 supported the prediction that

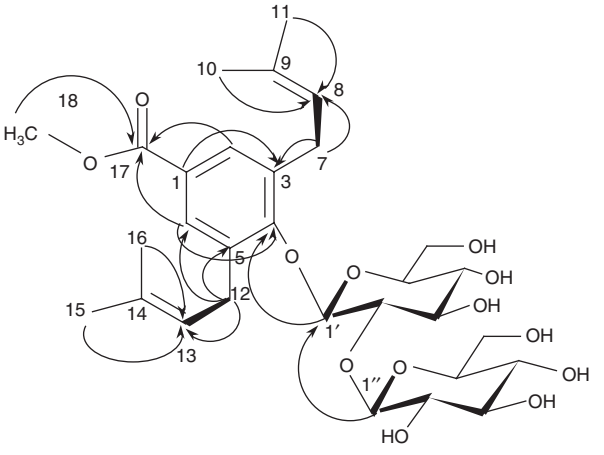

Figure 2. Key HMBC (→) and COSY (▬) correlations observed of compound **1**.

D-glucose was attached to 4-hydroxyl group with a β configuration. The correlation from H-1″ to C-2′ indicated that the two D-glucoses were connected through a 1″ to 2′ linkage with a β configuration.

Furthermore, the NMR spectra of compound **1** and known compound **4** (Hung et al. 2013) were similar, with the exception that **1** had a molecular weight of 30 more than **4**. This is caused by the presence of an additional CH_2O (δ_H at 3.56, 3.73 and δ_C at 62.9) on a sugar moiety. Only D-glucose was identified through GC–MS analysis in 1; thus, compound **1** was determined to be 2′-O-β-D-glucopyranosyl-4-O-β-D-glucopyranosyl-3,5-bis(3-methyl-2-butenyl)-methyl-17-benzoate, known as liparisglycoside A.

Compound **2** was isolated as an amorphous powder. Its molecular formula was determined to be $C_{23}H_{32}O_8$ based on the ^1H and ^{13}C NMR data. The pseudomolecular ion peak in the HR-ESI-MS at *m/z* 459.2008 $[M + Na]^+$ (calcd 459.1989) indicated eight degrees of unsaturation. Comparison of the spectral data of **2** with that of **4** indicated that they were similar with three differences: (1) there were only arabinose signals in **2**, (2) the ^{13}C NMR spectral data indicated the presences of an additional oxygenated tertiary carbon at δ 71.8 (C-14) and the lack of an olefinic quaternary carbon in **2**, which indicated that there was an additional OH connected to C-14, which down-shifted carbon 14 of compound **2**, (3) there were two *trans* olefinic protons at δ 7.10 (1H, d, $J = 16.0$ Hz, H-12) and 6.32 (1H, d, $J = 16.0$ Hz, H-13) in **2**, and in HMBC, these two protons had correlations with C-14, C-4, C-5, C-6, indicating that these two *trans* olefinic carbons were connected to both an aromatic ring and C-14. In addition, in HMBC, the methyl protons at δ 1.36 (3H, s, H-15) and 1.35 (3H, s, H-16) had correlations with C-14 (δ 71.8), indicating that the two methyl groups were connected to C-14. An anomeric proton of L-arabinose δ 4.57 (d, $J = 7.5$ Hz, H-1′) had correlations with C-4 (δ 157.4), which indicated that the L-arabinose moiety was attached to the 4-hydroxyl group with an α-configuration. Therefore, compound **2** was identified as 3-(3-hydroxy-3-methyl-1-butenyl)-5-(3-methyl-2-butenyl)-4-*O*-α-L-arabinose-methyl-17-benzoate, also known as liparisglycoside B.

Compound **3** was isolated as white needles. Its molecular formula was determined to be $C_{29}H_{44}O_{13}$ based on the NMR data. The pseudomolecular ion peak in the HR-ESI-MS at *m/z* 623.2656 $[M + Na]^+$ (calcd 623.2674) indicated eight degrees of unsaturation. Comparison of the spectral data of **3** with **4** indicated that they contained same two sugar groups, there were only four differences in aglycone moiety between them: (1) **3** had a molecular weight of 18 more than **4**, (2) the ^{13}C NMR spectrum indicated that **3** contained an additional oxygenated tertiary carbon at δ 72.1 (C-14) and lacked an olefinic quaternary carbon resonance,

indicating that there was an OH connected to C-14, (3) the ^{1}H NMR spectrum indicated there were six methylene signals at δ 3.50 (2H, d, $J = 7.0$ Hz, H-7), 2.89, 2.81 (2H, dd, $J = 12.0$, 5.0 Hz, H-12), 1.74, 1.69 (2H, dd, $J = 12.0$, 5.0 Hz, H-13), and these signals appeared in the ^{13}C NMR spectrum at δ 29.9 (C-7), 26.6 (C-12) and 45.8 (C-13), (4) one olefinic proton was absent in **3**. In HMBC, the methyl protons at δ 1.23 (3H, d, $J = 2.0$ Hz, H-15) and 1.22 (3H, d, $J = 2.0$ Hz, H-16) had correlations with C-14 (δ 72.1) and C-13 (δ 45.8). In addition, the protons at δ 1.74, 1.69 (H-13) had correlations with the carbons at δ 72.1 (C-14) and 26.6 (C-12), the proton at δ 2.80 (2H, dd, $J = 12.0$, 5.0 Hz, H-12) had correlations with the carbons at δ 158.7 (C-4), 138.8 (C-5), and 130.4 (C-2), all of which indicated that the two methyl fragments were connected to the oxygenated tertiary carbon C-14 and that the olefinic double bond between carbons 13 and 14 in compound **4** was hydrogenated in compound **3**. Therefore, compound **3** was identified as 2′-*O*-α-L-arabinose-4-*O*-β-D-glucopyranosyl-3-(3-hydroxy-3,3-dimethylbutyl)-5-(3-methyl-2-butenyl)-methyl-17-benzoate, named liparisglycoside C.

In addition, the effects of the compounds on LPS-stimulated NO production in peritoneal macrophage cells were determined (Table 1). The results indicated that compounds **3** and **9** inhibited NO production.

A preliminary screening of the hypolipidaemic activities of the compounds was conducted in an *in vitro* experiment (Table 2). Compared with the control group, compounds **1**, **3** and **9**

Table 1. Inhibition of compounds on LPS-stimulated NO production in peritoneal macrophages cells ($n = 3$).

Group	Concentration	NO production	Inhibition rate
LPS	–	38.47	–
Dex	10^{-5}	3.48	90.95
1	10^{-5}	34.38	10.63
2	10^{-5}	34.93	9.21
3	10^{-5}	26.65	30.73
4	10^{-5}	38.80	−0.85
5	10^{-5}	34.38	10.63
6	10^{-5}	39.35	−2.29
7	10^{-5}	33.27	13.52
8	10^{-5}	35.63	7.38
9	10^{-5}	20.82	45.89

Note: Data were expressed as the mean ± SE.

Table 2. Hypolipaemic effects of compounds on triglyceride content in HepG2.

Group	Results
Model	0.3410 ± 0.0113
Simvastatin	0.1799 ± 0.0109**
1	0.2399 ± 0.0057*
2	0.2598 ± 0.0077
3	0.2147 ± 0.0075**
4	0.2450 ± 0.0083
5	0.2488 ± 0.0265
6	0.2474 ± 0.0051
7	0.2402 ± 0.0018
8	0.2414 ± 0.0123
9	0.2362 ± 0.0055*

Note: Data were expressed as mean ± SE.
*$p < 0.05$, **$p < 0.01$ versus control group.

expressed potential hypolipidaemic effects ($p < 0.05$), with compound **3** ($p < 0.01$) being significantly active.

The amount of phenolic glycosides we isolated is sufficient to allow for further investigations on biological activities, which are warranted based on our preliminary results.

3. Experimental

3.1. *General procedures*

UV spectra were recorded on a JASCO V650 spectrophotometer (Jasco International Co., Ltd., Tokyo, Japan). IR spectra were recorded on a Nicolet 5700 FT-IR spectrometer (Thermo Electron, Waltham, MA, USA) using the microscope transmission method. NMR spectra were obtained on an INOVA-500 MHz NMR spectrometer (Varian Medical Systems, Palo Alto, CA, USA) operating at 500 MHz for ^{1}H NMR and at 125 MHz for ^{13}C NMR. Chemical shifts are given in δ (ppm) with solvent (DMSO-d_6, $CDCl_3$ or CD_3OD) peaks used as references. GC–MS spectra were obtained using an Agilent Technologies 7890A instrument (Agilent, Palo Alto, CA, USA). ESI-MS spectra were measured on an Agilent 1100 (Agilent Technologies, Palo Alto, CA, USA) Series LC/MSD ion-trap mass spectrometer. HR-ESI-MS data were recorded on an Autospec Ultima-TOF mass spectrometer (Micromass Communication, Inc., Manchester, England). Analytical HPLC was run on an Agilent 1100 series instrument with a UV/diode array detector using an YMC (YMC Co., Ltd., Kyoto, Japan) column (RP-C18, 4.6 × 250 mm, 5 μm). Preparative HPLC was performed on a Shimadzu LC-6AD instrument (Shimadzu Corporation, Tokyo, Japan) with an SPD-10A detector, using an YMC-Pack ODS-A column (20 × 250 mm, 5 μm). Octodecylcyclohexane silane (ODS) (45–70 μm, Merck KGaA, Darmstadt, Germany) and silica gel (200–30 mesh, Qingdao Marine Chemical, Inc., Qingdao P.R. China) were used for column chromatography, glass pre-coated with silica gel GF254 (200–300 mesh, Qingdao Marine Chemical, Inc.) was used for TLC.

3.2. *Plant material*

L. odorata (Willd.) Lindl was collected in the Jiangxi province of China in 2010, identified by Professor Xuewen Lai in Jiangxi University of Traditional Chinese Medicine in China, and a voucher specimen (No. 002017) has been deposited in the Herbarium of Jiangxi University of Traditional Chinese Medicine.

3.3. *Extraction and isolation*

The air-dried and powdered plant of *L. odorata* (50.0 kg) was extracted with 95% EtOH under reflux three times (each time for 1.0 h). Then, the EtOH extract was concentrated under reduced pressure to give a brown solid material (980 g), which was dissolved in 6000 mL of 0.2 M HCl. The HCl-soluble fraction was basified with $NH_3{\cdot}H_2O$ to a pH of 10, and extracted three times with ethyl acetate. The ethyl acetate-soluble fraction was concentrated under reduced pressure to give a brown solid material (95 g). Next, the total ethyl acetate fraction (95 g) was subjected to neutral silica gel column chromatography, eluted with a $CHCl_3$/MeOH (10:1–1:1) to give eight fractions (A1–A8). Fraction A2 (20 g) was subjected to a neutral silica gel column eluted with CH_2Cl_2/MeOH (40:1–1:1) to give 49 subfractions. Subfractions A2-5–A2-10 (3 g) were subjected to neutral silica gel column chromatography eluted with petroleum ether/acetone (10:1–1:1). Fraction 7 contained crystals that were recrystallised in hexane to obtain compound **7** (50 mg). Fraction 9 contained crystals that were recrystallised in petroleum ether to obtain compound **5** (100 mg). Fraction 15 contained crystals that were recrystallised in petroleum ether to obtain compound **6** (50 mg). Subfractions A2-16–A2-21 (1.3 g) were subjected to neutral

silica gel column chromatography eluted with $CHCl_3/CH_3COCH_3$ (1:1), continually purified by preparative HPLC using 60% MeOH to give, **1** (100 mg) and **4** (90 mg). Subfractions A2-26–A2-29 (0.6 g) were subjected to ODS column chromatography eluted with 60% MeOH, compounds **2** (30 mg) and **8** (60 mg) were obtained by continue preparative HPLC eluted with 25% MeOH. Fraction A3 (17.7 g) was subjected to neutral silica gel column eluted with CH_2Cl_2/MeOH (40:1–0:1) to give 39 subfractions. Subfractions A3-32–A3-36 (4.1 g) were subjected to neutral silica gel column chromatography eluted with $CHCl_3$/MeOH (20:1–0:1), fractions 24–29 were continually purified with another neutral silica gel column to give **9** (30 mg).

3.4. *Liparisglycoside A (1)*

White needles, m.p. 185.3–186.5, $[\alpha]_D^{20}$ − 7.23 ($c = 0.1$, MeOH); UV (MeOH) λ_{max} (log) 207 (3.53), 240 (3.02); IR(KBr) ν_{max} (cm^{-1}) 3322, 1723, 1437, 1320, 1286, 1078, 1024; HR-ESI-MS *m/z* 635.2785 (calcd 635.2674 for $C_{30}H_{44}O_{13}Na$). ^{1}H NMR(500 MHz, CD_3OD): δ_H 1.70 (s, 6H, Me-11, 16), 1.71 (s, 6H, Me-10, 15), 3.51 (d, $J = 7.2$ Hz, 4H, H-7, 12), 3.80 (s, 3H, OMe-18), 4.78 (d, $J = 7.5$ Hz, 1H, H-1′), 4.76 (d, $J = 7.5$ Hz, 1H, H-1″), 5.23 (t, $J = 7.2$ Hz, 2H, H-8, 13),7.60 (s, 2H, H-2, 6). ^{13}C NMR (125 MHz, CD_3OD): δ_C 127.8 (C-1), 130.3 (C-2, 6), 137.6 (C-3, 5), 158.1 (C-4), 29.9 (C-7, 12), 124.4 (C-8, 13), 134.3 (C-9, 14), 26.3 (C-10, 15), 18.5 (C-11, 16), 168.9 (C-17), 52.8 (C-18), 104.5 (C-1′), 83.1 (C-2′), 78.4 (C-3′), 71.6 (C-4′), 78.7 (C-5′), 62.9 (C-6′), 105.6 (C-1″), 76.3 (C-2″), 78.3 (C-3″), 72.0 (C-4″), 78.4 (C-5″), 63.2 (C-6″).

3.5. *Liparisglycoside B (2)*

White amorphous powder, $[\alpha]_D^{20}$ −11.57 ($c = 0.1$, MeOH); UV (MeOH) λ_{max} (log) 209 (3.55), 245 (3.03); IR (KBr) ν_{max} (cm^{-1}) 3393, 2972, 1719, 1599, 1435, 1081, 1015, 909; HR-ESI-MS *m/z* 459.2008 (calcd 459.1989 for $C_{23}H_{32}O_8Na$). ^{1}H NMR (500 MHz, CD_3OD): δ_H 1.35 (s, 3H, Me-16), 1.36 (s, 3H, Me-15), 1.67 (s, 3H, Me-11), 1.69 (s, 3H, Me-10), 3.44 (dd, $J = 7.0$,11.0 Hz, 1H, H-7a), 3.57 (t, $J = 7.0$ Hz, 1H, H-7b), 3.83 (s, 3H, OMe-18), 4.57 (d, $J = 7.5$ Hz, 1H, H-1′), 5.24 (t, $J = 7.0$ Hz, 1H, H-8), 6.32 (d, $J = 16.0$ Hz, 1H, H-13), 7.10 (d, $J = 16.0$ Hz, 1H, H-12), 7.63 (s, 1H, H-6), 7.94 (s, 1H, H-2). ^{13}C NMR (125 MHz, CD_3OD): δ_C 126.8 (C-1), 127.4 (C-2), 132.5 (C-3), 157.4 (C-4), 138.2 (C-5), 130.6 (C-6), 29.8 (C-7), 123.6 (C-8), 134.0 (C-9), 25.9 (C-10), 18.0 (C-11), 123.0 (C-12), 140.7 (C-13), 71.8 (C-14), 29.7 (C-15,16), 168.4 (C-17), 52.6 (C-18), 106.7 (C-1′), 73.1 (C-2′), 74.4 (C-3′), 71.8 (C-4′), 69.8 (C-5′).

3.6. *Liparisglycoside C (3)*

Colourless needles, m.p. 123.3–135.5°C, $[\alpha]_D^{20}$ −13.64 ($c = 0.1$, MeOH); UV (MeOH) λ_{max} (log) 209 (3.50), 245(3.01); IR(KBr) ν_{max} (cm^{-1}) 3379, 2966, 1713, 1086, 776; HR-ESI-MS *m/z* 623.2656 (calcd 623.2674 for $C_{29}H_{44}O_{13}Na$). ^{1}H NMR (500 MHz, CD_3OD): δ_H 1.22 (d, $J = 2.0$ Hz, 3H, Me-16), 1.23 (d, $J = 2.0$ Hz, 3H, Me-15), 1.68 (s, 3H, Me-11), 1.69 (dd, $J = 5.0$, 12.0 Hz, 1H, H-13a), 1.74 (dd, $J = 5.0$, 12.0 Hz, 1H, H-13b), 1.71 (s, 3H, Me-10), 2.81 (dd, $J = 5.0$, 12.0 Hz, 1H, H-12a), 2.89 (dd, $J = 5.0$, 12.0 Hz, 1H, H-12b), 3.50 (d, $J = 7.0$ Hz, 2H, H-7), 3.81 (s, 3H, OMe-18), 4.66 (d, $J = 8.0$ Hz, 1H, H-1″), 4.75 (d, $J = 7.0$ Hz, 1H, H-1′), 5.24 (t, $J = 6.5$ Hz, 1H, H-8), 7.59 (s, 1H, H-2), 7.67 (s, 1H, H-6). ^{13}C NMR (125 MHz, CD_3OD): δ_C 127.9 (C-1), 130.4 (C-2), 137.8 (C-3), 158.7 (C-4), 138.8 (C-5), 130.6 (C-6), 29.9 (C-7), 124.3 (C-8), 134.6 (C-9), 26.4 (C-10), 18.7 (C-11), 26.6 (C-12), 45.8 (C-13), 72.1 (C-14), 30.1 (C-15), 29.7 (C-16), 169.0 (C-17), 53.0 (C-18), 105.1 (C-1′), 81.6 (C-2′), 74.3 (C-3′), 69.5 (C-4′), 67.2 (C-5′), 106.1 (C-1″), 76.2 (C-2″), 78.5 (C-3″), 71.9 (C-4″), 78.6 (C-5″), 63.1 (C-6″).

3.7. *Acidic hydrolysis and sugar analysis of compound 1*

Compound **1** (5 mg) was refluxed in 2 M HCl (3 mL) at 60°C for 3 h and concentrated to give a brown material, which was dissolved in water (3 mL) and partitioned with EtOAc (3 mL × 3). The aqueous layer was evaporated to dryness under reduced pressure to give one monosaccharide residue and the sugars were identified as D-glucose by comparison with retention times of authentic sample of D-glucose (19.84 min) (Kinijo et al. 1992; Wang et al. 2013).

3.8. *Effect of compounds on LPS-stimulated NO production in peritoneal macrophage cells*

The LPS-stimulated NO production effects of the compounds were valued by measuring the peritoneal macrophage cells *in vitro* (Ding et al. 1988; Chen et al. 2006).

3.9. *Hypolipidaemic activity assays*

The hypolipidaemic activities of compounds **1–9** were assayed by measuring the triglyceride content in HepG2 cells *in vitro* (Gómez-Lechón et al. 2007; Wang et al. 2013).

4. Conclusions

Three new phenolic glycosides named liparisglycoside A–C, together with six known compounds, were isolated from *L. odorata* for the first time. In addition, new compound liparisglycoside C and known compound **9** revealed potential anti-inflammatory activities, and new compounds liparisglycoside A, liparisglycoside C, known compound **9** expressed potential hypolipidaemic effects. Further investigations are ongoing in our laboratory.

Supplementary material

Supplementary material relating to this article is available online, alongside Figures S1–S12.

Acknowledgements

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