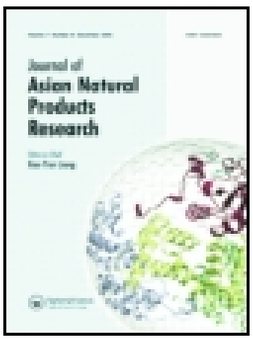

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Two new phenolic compounds from the seeds of *Machilus yunnanensis*

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ABSTRACT

Chemical constituents investigation on the seeds of *Machilus yunnanensis* led to two new phenolic compounds 8-*O*-acetyl-phenylethanoid-4-*O*-*β*-D-glucopyranoside (**1**) and (*E*)-2,3-bis(4-hydroxyphenyl)acrylaldehyde (**2**), together with 16 known compounds. Their structures were elucidated on the basis of spectroscopic data analysis (IR, MS, 1D, and 2D NMR). Meanwhile, compounds **1–3**, **6–13**, **17**, and **18** were evaluated for vasorelaxant effects on the rat endothelium-intact thoracic aorta rings precontracted with phenylephrine (PE) or KCl. The bioassay results showed that compound **17** had significant vasorelaxant effect on the endothelium-intact thoracic aorta rings precontracted with KCl.

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KEYWORDS
Lauraceae; *Machilus yunnanensis*; phenolic compounds; vasorelaxant effect

1. Introduction

Machilus yunnanensis (Lauraceae) is a deep rooted tree, mainly distributed in Yunnan and Sichuan provinces of China [1]. In China, it has been used for the treatments of skin swelling, sore, mumps, burn, bone fractures, and rheumatism [2]. Previous studies on the plants of *Machilus* led to the isolation of different compounds, such as alkaloids [3], triterpenoid glycosides [4], butyrolactones [5], sesquiterpenoids [6], and lignans [7]. However, no phytochemical or biological activities literature on *M. yunnanensis* was available. We made a phytochemical investigation and isolated two new phenolic compounds 8-*O*-acetyl-phenylethanoid-4-*O*-*β*-D-glucopyranoside (**1**) and (*E*)-2,3-bis(4-hydroxyphenyl) acrylaldehyde (**2**) (Figure 1), along with 16 known compounds from the 70% aqueous acetone extract of *M. yunnanensis* seeds. Their structures were established by extensive spectroscopic data analysis and comparison with those of literature values. In addition, previous literature reported that *meso*-dihydroguaiaretic acid isolated from a species of *Machilus* genus, *M. thunbergii*, had aggregation activity and vasorelaxant effect [8]. Therefore, compounds **1–3**, **6–13**, **17,** and **18** were evaluated for their vasorelaxant effects on the rat endothelium-intact thoracic aorta rings precontracted with PE or KCl, but only compound **17**

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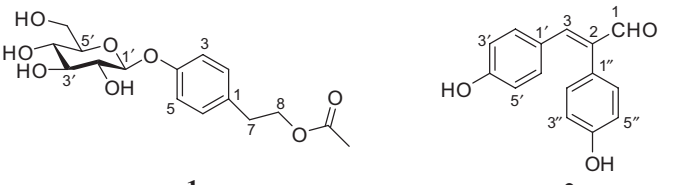
[1]These authors contributed equally to this work.



1 **2**

Figure 1. Structures of compounds **1** and **2**.

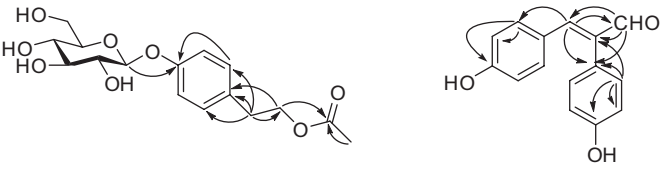
Table 1. ^{1}H NMR and ^{13}C NMR spectral data of **1** and **2** (MeOH-d_4, 600 MHz; δ in ppm, J in Hz).

	1			**2**	
No.	δ_H	δ_C	No.	δ_H	δ_C
1		133.3 (s)	1	9.60 (s)	196.6 (d)
2	7.06 (d, J=8.6 Hz)	131.0 (d)	2		140.5 (s)
3	7.18 (d, J=8.6 Hz)	117.9 (d)	3	7.40 (s)	153.0 (d)
4		158.0 (s)	1′		127.0 (s)
5	7.18 (d, J=8.6 Hz)	117.9 (d)	2′	7.17 (d, J=8.7 Hz)	134.2 (d)
6	7.06 (d, J=8.6 Hz)	131.0 (d)	3′	6.66 (d, J=8.7 Hz)	116.4 (d)
7	2.91 (t, J=6.9 Hz)	35.3 (t)	4′		161.5 (s)
8	4.25 (t, J=6.9 Hz)	66.5 (t)	5′	6.66 (d, J=8.7 Hz)	116.4 (d)
1′	4.92 (d, 7.5 Hz)	102.5 (d)	6′	7.17 (d, J=8.7 Hz)	134.2 (d)
2′	3.47–3.49 (m)	75.0 (d)	1″		126.1 (s)
3′	3.45–3.49 (m)	78.1 (d)	2″	6.99 (d, J=8.4 Hz)	131.8 (d)
4′	3.41–3.43 (m)	71.5 (d)	3″	6.86 (d, J=8.4 Hz)	116.7 (d)
5′	3.45–3.49 (m)	78.2 (d)	4″		158.5 (s)
6′	3.71–3.74 (m)	62.6 (t)	5″	6.86 (d, J=8.4 Hz)	116.7 (d)
	3.92 (d, 12.1 Hz)				
OAc		173.1 (s)	6″	6.99 (d, J=8.4 Hz)	131.8 (d)
	2.03 (s)	20.9 (q)			

showed vasorelaxant effect. Herein, we describe the isolation, structure elucidation, and bioassays of these isolated compounds.

2. Results and discussion

Compound **1** ($[\alpha]_D^{21.6}$ – 28.2) was obtained as a colorless oil. The molecular formula $C_{16}H_{22}O_8$ was deduced by HREIMS at m/z 365.1205 $[M + Na]^+$. The UV absorption maxima at 202, 219, and 271 nm revealed an aromatic ring moiety. The IR showed absorption bands at 3418 (hydroxy), 1729 (ester), 1614, and 1511 cm^{-1} (aromatic ring). The ^{1}H NMR spectral data (Table 1) showed one methyl at δ_H 2.03 (3H, s), two methenes at δ_H 2.91 (2H, *t*, J = 6.9 Hz), and 4.25 (2H, *t*, J = 6.9 Hz), a set of AA′BB′-type aromatic resonances at δ_H 7.06 (2H, d, J = 8.6 Hz) and 7.18 (2H, d, J = 8.6 Hz), one anomeric proton at δ_H 4.92 (1H, d, J = 7.5 Hz), and a series of sugar signals at δ_H 3.38–3.94. The ^{13}C NMR spectral data (Table 1) displayed one methyl (δ_C 20.9), two methenes (δ_C 35.3, 66.5), one ester (δ_C 173.1), six aromatic carbons (δ_C 117.9 × 2,131.0 × 2,133.3, 158.0), and one glucopyranosyl unit (δ_C 102.5, 75.0, 78.1, 71.5, 78.2, and 62.6). The ^{1}H and ^{13}C NMR spectral data of **1** were very similar to **3** (icariside D_2) except for the presence of acetyl group (δ_C 173.1 and 20.9) in **1**, which suggested **1** was the analog of **3**. Acid hydrolysis of **1** liberated a sugar identified as D-glucose by TLC ($CHCl_3$/MeOH; 2:1; R_f 0.26) with an authentic sample [9], and $[\alpha]_D^{21.6}$ + 68.83 of the liberated sugar further confirmed that. The β anomeric configuration for the glucose of **1** was



1 **2**

Figure 2. Key HMBC correlations of **1** and **2**.

determined by its coupling constant (J = 7.5 Hz). The arrangements of the methyl, ester, methenes, aromatic ring, and β-D-glucose were determined by analysis of the proton coupling patterns, HMQC, HMBC data (Figure 2). In the HMBC spectrum, the correlations from H-7 (δ_H 2.91, t) to C-1 (δ_C 133.3), C-2, 6 (δ_C 131.0 × 2), and C-8 (δ_C 66.5); H-8 (δ_H 4.25, t) to C-1 (δ_C 133.3), C-7 (δ_C 35.3), and carbonyl (δ_C 173.1); protons of methyl (δ_H 2.03, s) to carbonyl (δ_C 173.1) proved these links successively. Hence, compound **1** was elucidated as 8-*O*-acetyl-phenylethanoid-4-*O*-β-D-glucopyranoside.

Compound **2** ($[\alpha]_D^{21.6}$ – 3.2) was obtained as a yellowish amorphous powder. The molecular formula $C_{15}H_{12}O_3$ was determined by HREIMS at m/z 263.0678 $[M + Na]^+$. The UV absorption maxima at 202, 231, 329 nm indicated a conjugated system. The IR spectrum showed absorption bands at 3411 (hydroxy), 1661 (α,β-unsaturated carbonyl), 1600 and 1512 cm^{-1} (aromatic ring). The ^1H NMR spectral data (Table 1) displayed a set of AA′BB′-type aromatic resonances at δ_H 6.66 (2H, d, J = 8.7 Hz), 6.86 (2H, d, J = 8.4 Hz), 6.99 (2H, d, J = 8.4 Hz), and 7.17 (2H, d, J = 8.7 Hz), revealing two p-substituted aromatic rings. Moreover, one conjugated olefinic proton and one aldehyde proton were shown at δ_H 7.40 (1H, s) and 9.60 (1H, s), respectively. The ^{13}CNMR spectral data (Table 1) displayed 12 aromatic carbons (δ_C 116.4 × 2, 116.7 × 2, 126.1, 127.0, 131.8 × 2, 134.2 × 2, 158.5, 161.5), one double bond (δ_C 140.5, 153.0), and one aldehyde group (δ_C 196.6). In the HMBC spectrum, the correlations from H-3 (δ_H 7.40, s) to C-1′ (δ_C 127.0), C-2″, 6″ (δ_C 134.2 × 2), and C-1 (δ_C 196.6); H-1 (δ_H 9.60, s) to C-1″ (δ_C 126.1), and C-2 (δ_C 140.5); H-2″, 6″ (δ_H 6.99, d) to C-2 (δ_C 140.5) indicated the two p-substituted aromatic rings were connected to the double bond in C-2 and C-3, respectively, and the aldehyde group was substituted at C-2 in the double bond. The arrangements of the two aromatic rings, double bond, and aldehyde group were determined by analysis of the proton coupling patterns, HMQC, and HMBC data (Figure 2). Furthermore, the *cis*-configuration of the double bond was proved by the correlations from H-3 (δ_H 7.40, s) to H-2′, 6′ (δ_H 7.17, d), and H-1 (δ_H 9.60, s) in the ROESY spectrum (Figure 3). Thus, compound **2** was characterized as (*E*)-2,3-bis(4-hydroxyphenyl)acrylaldehyde.

Sixteen known compounds (**3–18**) were identified on the basis of comparing of their NMR spectral data with those in the literature as icariside D_2 (**3**) [10], 3-[4-(β-D-glucopyranosyloxy) phenyl]propionic acid (**4**) [11], p-2-ethylphenyl-β-D-glucopyranoside (**5**) [12], gastrodin (**6**) [13], koaburasidemonoethyl ether (**7**) [14], phenylethyl-β-D-glucopyranoside (**8**) [15], piceatannol-4′-*O*-β-D-glucopyranoside (**16**) [16], 4,4′-dihydroxychalcone (**10**) [17], tyrosylhydroxylphenyl acetate (**11**) [18], methyl 4-hydroxyphenyl acetate (**12**) [19], p-hydroxylbenzoic acid (**13**) [10], (1′*S*)-1′-(4-hydroxyphenyl)ethane-1′,2′-diol (**14**) [20], hydroxytyrosol (**15**) [21], 4-(2-hydroxyethyl)-1,3-benzenediol (**16**) [22], clovane-2β, 9α-diol (**17**) [23], (–)-oleuropeic acid (**18**) [21]. All above compounds were reported for the first time.

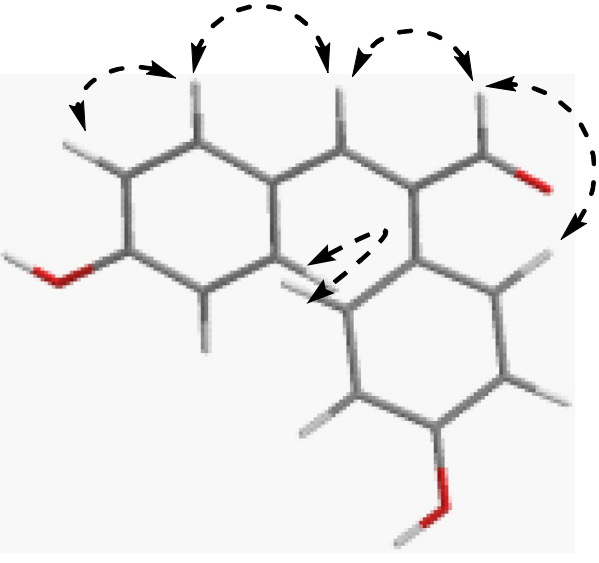


Figure 3. Key ROESY correlations of **2**.

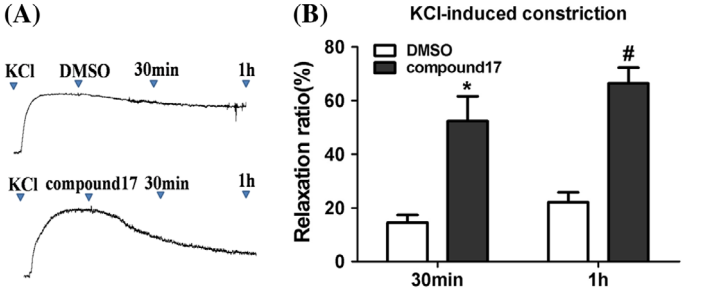


Figure 4. The vasorelaxant effect of compound **17** on rat aortic rings precontracted by KCl. (A) Representative traces showing the effect of **17** on KCl-induced contraction. (B) Statistical analysis of the effect of **17** on KCl-induced contraction. Data were expressed as mean ± SD, $n = 4$ from four rats, $^{*}p < 0.05$ vs. DMSO at 30 min, $^{\#}p < 0.05$ vs. DMSO at 1 h.

Compounds **1–3**, **6–13**, **17,** and **18** were tested for their vasorelaxant effects on the rat endothelium-intact thoracic aorta rings precontracted with phenylephrine (PE) or KCl. The results showed that compound **17** had significant vasorelaxant effect on the rat endothelium-intact aortic rings precontracted with KCl (Figure 4). Other compounds were inactive in current assay.

3. Experimental

3.1. General experimental procedures

Optical rotations were measured on a JASCO P-1020C digital polarimeter (JASCO, Tokyo, Japan). UV spectra were recorded using a Shimadzu UV-2401A spectrophotometer (Shimadzu, Kyoto, Japan). IR spectra were obtained on a Tensor 27 spectrometer (Bruker

Optics, Ettlingen, Germany) with KBr pelets. ESI-MS were recorded on an Agilent 6530 Q spectrometer (Agilent, Palo Alto, CA, USA). HREIMS were measured using a Waters Auto Premier P776 spectrometer (Waters, Milford, MA, USA). 1D and 2D NMR were performed on Bruker AVANCE III-600 spectrometers with TMS as an internal standard (Bruker Optics, Karlsruhe, Germany). Column chromatography was performed over silica gel (100–200 or 200–300 mesh, Qingdao Haiyang Chemical Co. Ltd., Qingdao, China), MCI gel (CHP 20P, 75–150 μm, Mitsubishi Chemical Corporation, Tokyo, Japan), and Sephadex LH-20 (Amersham Pharmacia Biotech, Uppsala, Sweden). Semi-preparative HPLC was performed on an Agilent 1260 apparatus (Agilent, Santa Clara, CA, USA) equipped with a UV detector and a Zorbax SB-C-18 (Agilent, 9.4 mm × 25 cm) column. Thin-layer chromatography was carried out on silicagel plates GF_{254} (Qingdao Haiyang Chemical Inc.) using various solvent systems, and spots were visualized by spraying with 10% H_2SO_4 in EtOH followed by heating.

3.2. Plant material

The air-dried seeds of *M. yunnanensis* (6.0 kg) used in this study were collected from Kunming, Yunnan province, China, in September 2013. The plant was identified by Prof. Xun Gong at Kunming Institute of Botany, Chinese Academy of Sciences (voucher no. KIB-2013114).

3.3. Extraction and isolation

The air-dried seeds of *Machilus yunnanensis* seeds (6.0 kg) was extracted with 70% aqueous acetone (48 h × 3) at room temperature and concentrated under vacuum to give residue, which was partitioned between H_2O and EtOAc. The EtOAc extract (120 g) was chromatographed over a silica gel column ($CHCl_3$/MeOH: 1:0–0:1) to afford six fractions A–F. Fr. C (12 g) was chromatographed on MCI (MeOH/H_2O, 10:95–100:0) to give four fractions C_1–C_4. Fr. C_2 (6 g) was subjected to Sephadex LH-20 (MeOH) to provide four major fractions $C_{2\text{-}1}$–$C_{2\text{-}4}$. Fr. $C_{2\text{-}3}$ (2.5 g) was separated into four subfractions $C_{2\text{-}3\text{-}1}$–$C_{2\text{-}3\text{-}4}$ by repeated silica gel columns ($CHCl_3$/MeOH; 8:1). Fr. $C_{2\text{-}3\text{-}2}$ (36 mg) was further purified by HPLC (MeOH/H_2O, 35:65, flow rate: 3 ml/min) to afford **1** (13.0 mg, t_R 8.2 min) and **3** (10.6 mg, t_R 11.2 min). Compounds **2** (2.6 mg, t_R 9.6 min), **10** (1.5 mg, t_R 14.8 min), and **11** (4.8 mg, t_R 14.8 min) were obtained from $C_{2\text{-}3\text{-}3}$ (16 mg). Fr. $C_{2\text{-}2\text{-}3}$ (57 mg) was subjected to HPLC (MeOH/H_2O, 40:60, flow rate: 3 ml/min) to produce **9** (12 mg, t_R 8.2 min), **13** (5.0 mg, t_R 10.3 min), and **8** (1.2 mg, t_R 12.7 min). Fr. C_3 (3 g) was subjected to repeated silica gel columns ($CHCl_3$/MeOH, 15:1–1:1) to yield **4** (4.6 mg). Fr. B (2.8 g) was dealt with the same steps just as C, then $B_{2\text{-}3\text{-}3}$ (368 mg) yielded **17** (12 mg), **18** (6 mg), and **12** (9.6 mg) by silica gel column ($CHCl_3$/acetone, 20:1–2:1). Compounds **14** (11 mg, t_R 11.3 min), **15** (8 mg, t_R 12.5 min), and **16** (7.5 mg, t_R 14.2 min) were obtained from $B_{2\text{-}3\text{-}2}$ (480 mg) by HPLC (MeOH/H_2O; 60:40; flow rate: 3 ml/min). Compounds **5** (1.8 mg, t_R 6.4 min), **6** (12 mg, t_R 8.2 min), and **7** (8.8 mg, t_R 11.9 min) were afforded from $D_{2\text{-}3\text{-}3}$ (29 mg) by HPLC (MeOH/H_2O; 25:75; flow rate: 3 ml/min).

3.3.1. 8-O-Acetyl-phenylethanoid-4-O-β-D-glucopyranoside (1)

Colorless oil; $[\alpha]_D^{21.6}$ – 28.2 (*c* 0.43, MeOH); UV (MeOH) λ_{max} (log ε): 271 (2.65), 219 (3.58), 202 (3.54) nm; IR (KBr) ν_{max} 3418, 2925, 2854, 1729, 1614, 1511, 1384, 1235, 1072, and

1039 cm^{-1}; For ^{1}H and ^{13}C NMR spectral data, see Table 1; ESI-MS: *m/z* 365 $[M + Na]^+$; HREIMS: *m/z* 365.1205 $[M + Na]^+$ (calcd for $C_{16}H_{22}O_8Na$, 365.1207).

3.3.2. (E)-2,3-Bis(4-hydroxyphenyl)acrylaldehyde (2)

Yellowish amorphous powder; $[\alpha]_D^{21.6} - 3.2$ (*c* 0.52, MeOH); UV (MeOH) λ_{max} (log ε): 329 (4.05), 231 (3.96), 202 (4.09) nm; IR (KBr) ν_{max} 3411, 2929, 2855, 1661, 1600, 1512, 1383, 1269, 1225, 1172, 1086, 837 and 557 cm^{-1}; For ^{1}H and ^{13}C NMR spectral data, see Table 1; ESI-MS: *m/z* 263 $[M + Na]^+$; HREIMS: *m/z* 263.0678 $[M + Na]^+$ (calcd for $C_{15}H_{12}O_3Na$, 263.0679).

3.3.3. Acid hydrolysis of 1

Compound **1** (8.0 mg) was hydrolyzed with 2 M HCl/dioxane (1:1, 4 ml) under reflux for 8 h, then partitioned between H_2O and $CHCl_3$ (2 ml × 3). The aqueous layer was further neutralized with 2 M NaOH and dried to liberate a glucose identified by TLC ($CHCl_3$–MeOH; 2:1; R_f 0.26) with the authentic sample.

3.4. Bioassay for vasorelaxant effect on the rat endothelium-intact thoracic aorta rings

Vasorelaxant effects of 100 μmol/L of selected compounds were evaluated on the rat endothelium-intact thoracic aorta rings precontracted with PE or KCl. Rat aortic rings were prepared according to the literature [24]. Aortic rings were mounted on stainless steel hooks in organ baths containing 37 °C. Krebs solution continuously bubbled with 95% O_2 and 5% CO_2, then equilibrated for 60 min under a resting tension of 1.5 g. After equilibration, the vessels were exposed to 1 μmol/L PE, followed by 10 μmol/L acetylcholine (Ach) to check functional endothelial integrity, and more than 80% relaxation of the rings was considered to be an endothelium-intact ring. Endothelium-intact rings precontracted with 1 μmol/L PE or 60 μmol/L KCl were treated with different compounds or DMSO for 30 min, and the changes in tension of aortic rings were recorded. The vasorelaxant effect was calculated as a percentage of therelaxation in response to PE or KCl on the aortic rings.

Disclosure statement

No potential conflict of interest was reported by the authors.

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References

[1] Editorial Board of the Flora of China, *The Flora of China*. (Science Press, Beijing, 1982), vol. **31**, p. 14.
[2] Chinese Herbal Medicine Company, *Chinese Medicine Resources Annales*, (Science Press, Beijing, 1994), p. 295.

[3] M.T. Liu, S. Lin, Y.H. Wang, W.Y. He, S. Li, S.J. Wang, Y.C. Yang, and J.G. Shi, *Org. Lett*. **9**, 129 (2007).
[4] M.L. Gan, M.T. Liu, L.S. Gan, S. Lin, B. Liu, Y.L. Zhang, J.C. Zi, W.X. Song, and J.G. Shi, *J. Nat. Prod*. **75**, 1373 (2012).
[5] M.T. Liu, S. Lin, M.L. Gan, B. Liu, J.C. Zi, W.X. Song, Y.L. Zhang, X.N. Fan, Y. Liu, W. Tan, S.J. Wang, Y.C. Yang, and J.G. Shi, *J. Asian. Nat. Prod. Res*. **14**, 713 (2012).
[6] W. Cheng, C.G. Zhu, W.D. Xu, X.N. Fan, Y.C. Yang, Y. Li, X.G. Chen, W.J. Wang, and J.G. Shi, *J. Nat. Prod*. **72**, 2145 (2009).
[7] P.B. Bu, Y.R. Li, M. Jiang, F. Wang, X.L. Wang, S. Lin, C.G. Zhu, and J.G. Shi, *J. Asian. Nat. Prod. Res*. **15**, 482 (2013).
[8] M. Miyazawa, Y. Okuno, K. Oshiro, H. Kasahara, H. Shimamura, S. Nakamura, and H. Kameoka, *Biosci. Biotech. Bioch*. **62**, 1425 (1998).
[9] P.Y. Liao, D. Wang, Y.J. Zhang, and C.R. Yang, *J. Agric. Food. Chem*. **56**, 1751 (2008).
[10] T. Miyase, A. Ueno, N. Takizawa, H. Kobayashi, and H. Oguchi, *Phytochemistry*. **28**, 3483 (1989).
[11] N. Ronsted, H. Franzyk, P. Molgaard, J.W. Jaroszewski, and S.R. Jensen, *Plant. Syst. Evol*. **242**, 63 (2003).
[12] C. Socolsky, M.L. Fascio, N.B. D'Accorso, A. Salvatore, E. Willink, Y. Asakawa, and A. Bardon, *J. Chem. Ecol*. **34**, 539 (2008).
[13] Y.W. Li, and C.L. Ma, *J. Serb. Chem. Soc*. **79**, 1205 (2014).
[14] Y. Wang, H. Zhu, D. Wang, R. Cheng, C. Yang, M. Xu, and Y. Zhang, *Bull. Korean. Chem. Soc*. **35**, 631 (2014).
[15] V.T. Nguyen, P.T. Nguyen, L.D. Dat, P.T.T. Huong, S.H. Lee, H.D. Jang, X.C. Nguyen, H.N. Nguyen, P.V. Kiem, C.V. Minh, and Y.H. Kim, *Arch. Pharmacal. Res*. **38**, 1774 (2015).
[16] L. Li, D. Woolridge, S.R. Saing, J. Jennings, G.E. Henry, and N.P. Seeram, *Biochem. Syst. Ecol*. **38**, 1074 (2010).
[17] R.C. Tandel, J. Gohil, and N.K. Patel, *Res. J. Recent. Sci*. **1**, 122 (2012).
[18] I. Aissa, M. Bouaziz, F. Frikha, R. B. Mansour, and Y. Gargouri, *Process. Biochem. (Oxford, U. K.).***47**, 2356 (2012).
[19] S. Yue, Z.Z. Jiao, H.X. Sun, T.Y. Jin, and L. Xiang, *Helv. Chim. Acta*. **98**, 961 (2015).
[20] T. Ishikawa, K. Kondo, and J. Kitajima, *Chem. Pharm. Bull*. **51**, 32 (2003).
[21] M. Pérez-Bonilla, S. Salido, T. A. van Beek, and J. Altarejos, *J. Agric. Food. Chem*. **62**, 144 (2014).
[22] Y. Ozaki, K. Mochida, and S.W. Kim, *Chem. Pharm. Bull*. 35, 1790 (1987).
[23] Y. Wang, Z. Li, H. Zhang, Y. Sha, Y. Pei, and H. Hua, *Chem. Pharm. Bull*. **56**, 843 (2008).
[24] G.J. Chang, T.P. Lin, Y.S. Ko, and M.S. Lin, *Life Sci*. **86**, 869 (2010).