Biochemical Systematics and Ecology 78 (2018) 59–62

Contents lists available at ScienceDirect

Biochemical Systematics and Ecology

journal homepage: www.elsevier.com/locate/biochemsyseco

Phenols from the rhizome of *Wikstroemia indica*

Qin Fan[a,1], Yi-Ping Jiang[b,1], Dao-Qi Zhu[a], Wei Xu[a], Wen-Qiang Huang[a], Xiao-Jun Huang[c,**], Meng Shao[a,*]

[a] The Key Laboratory of Molecular Biology, State Administration of Traditional Chinese Medicine, School of Traditional Chinese Medicine, Southern Medical University, Guangzhou, 510515, China
[b] Department of Pharmacy, Zhuhai People's Hospital, Zhuhai, 519000, China
[c] Institute of Traditional Chinese Medicine & Natural Products, College of Pharmacy, Jinan University, Guangzhou, 510632, China

ARTICLE INFO

Keywords:
Wikstroemia indica
Thymelaeaceae
Phenol
Chemotaxonomy

ABSTRACT

A phytochemical investigation on the rhizome of *Wikstroemia indica* led to the isolation of thirteen phenolic compounds, including three flavonoids (**1**-**3**), three lignans (**4**–**6**), two coumarins (**7**, **8**), one stilbene glycoside (**9**), one polyketide (**10**), one caffeoylquinic acid (**11**), one diarylpentanoid (**12**) and one benzoic acid derivative (**13**). The structural elucidation of these compounds was determined by using spectroscopic methods and by comparison with the literature. Compounds **2**, **4**, **5** and **12** were obtained from *W. indica* for the first time, and compounds **9–11** were firstly isolated from the family Thymelaeaceae. Furthermore, the chemotaxonomic significance of these compounds was discussed.

1. Subject and source

The genus *Wikstroemia* Endl. (Thymelaeaceae), comprising approximately 70 species, spreads widely from Southeast Asia, Oceania to Pacific islands. Of these 70 species, 44 and 5 varieties can be found in South and Southwest of China (Wang and Gilbert, 2007a). This area is considered to be the center of diversity for this genus (Halda, 1998). *Wikstroemia indica* (Linn.) C. A. Meyer, one of the most studied species of *Wikstroemia*, is an evergreen arbuscle found in mountain area at an elevation below 1500 m in Guangdong, Guangxi and Yunnan Provinces of China (Wang and Gilbert, 2007b).

In present study, the rhizomes of *W. indica* were collected from Baiyun mountain of Guangzhou, Guangdong Province, China in November 2015, and were authenticated by Dr. Xiao-Jun Huang (College of Pharmacy, Jinan University, Guangzhou). A voucher specimen (No. 201511LG) was deposited at School of Traditional Chinese Medicine, Southern Medical University.

2. Previous work

Extensive previous phytochemical studies on genus *Wikstroemia* had been identified diverse types of phenolic compounds, including lignans from *W. indica* (Chang et al., 2017), *W. scytophylla* Diels (Lei et al., 2017) and *W. lanceolata* Merr. (Lin et al., 2004); flavonoids from *W. scytophylla* (Lei et al., 2017); coumarins from *W. indica* (Chen et al., 2009a) and *W. hainanensis* Merr. (Liao et al., 2006); dilignans from *W. indica* (Chang et al., 2017; Wang et al, 2005a, 2012a,b); biflavonoids from *W. indica* (Huang et al., 2012; Li et al., 2012; Shao et al., 2016), *W. taiwanensis* Chang (Chen et al., 2012) and *W. sikokiana* Franch. & Sav. (Baba et al., 1994; Niwa et al., 1986); bis- and tri-coumarins from *W. indica* (Wang et al., 2018) and *W. virdiflora* Meisn. (Tandon and Rastogi, 1977). In addition, sesquiterpenes isolated from *W. indica* (Wang et al., 2005b), *W. coriacea* B.C. Seemann (Ingert et al., 2013) and diterpenes from *W. chamaedaphne* Meissn (Guo et al., 2012; Guo et al., 2015), *W. retusa* A.Gray (Abe et al, 1997, 1998; Yaga et al., 1993) were also reported.

3. Present study

The air-dried rhizomes of *W. indica* (10.0 kg) were powdered and percolated by 95% ethanol (50 L × 3 times) at room temperature. The combined extract solution was condensed under vacuum at 45 °C affording the crude extract (1.5 kg). The extract was suspended in water and partitioned successively with petroleum ether (60–90 °C, PE), ethyl acetate (EtOAc) and *n*-butyl alcohol (*n*-BuOH) to afford 80 g, 500 g and 600 g of the corresponding residues.

The EtOAc portion was subjected to silica gel chromatography (CC) with a gradient of $CHCl_3$-MeOH (100:0 → 0:100, v/v) to obtain 18 fractions (Frs. E1-18). Fr. E4 (33.8 g) was chromatographied over silica

* Corresponding author.
** Corresponding author.
E-mail addresses: zhyxiaohuang@163.com (X.-J. Huang), shaomeng_smu@163.com (M. Shao).
[1] These authors contributed equally to this work.

https://doi.org/10.1016/j.bse.2018.04.004
Received 31 October 2017; Received in revised form 26 March 2018; Accepted 2 April 2018
Available online 07 April 2018
0305-1978/

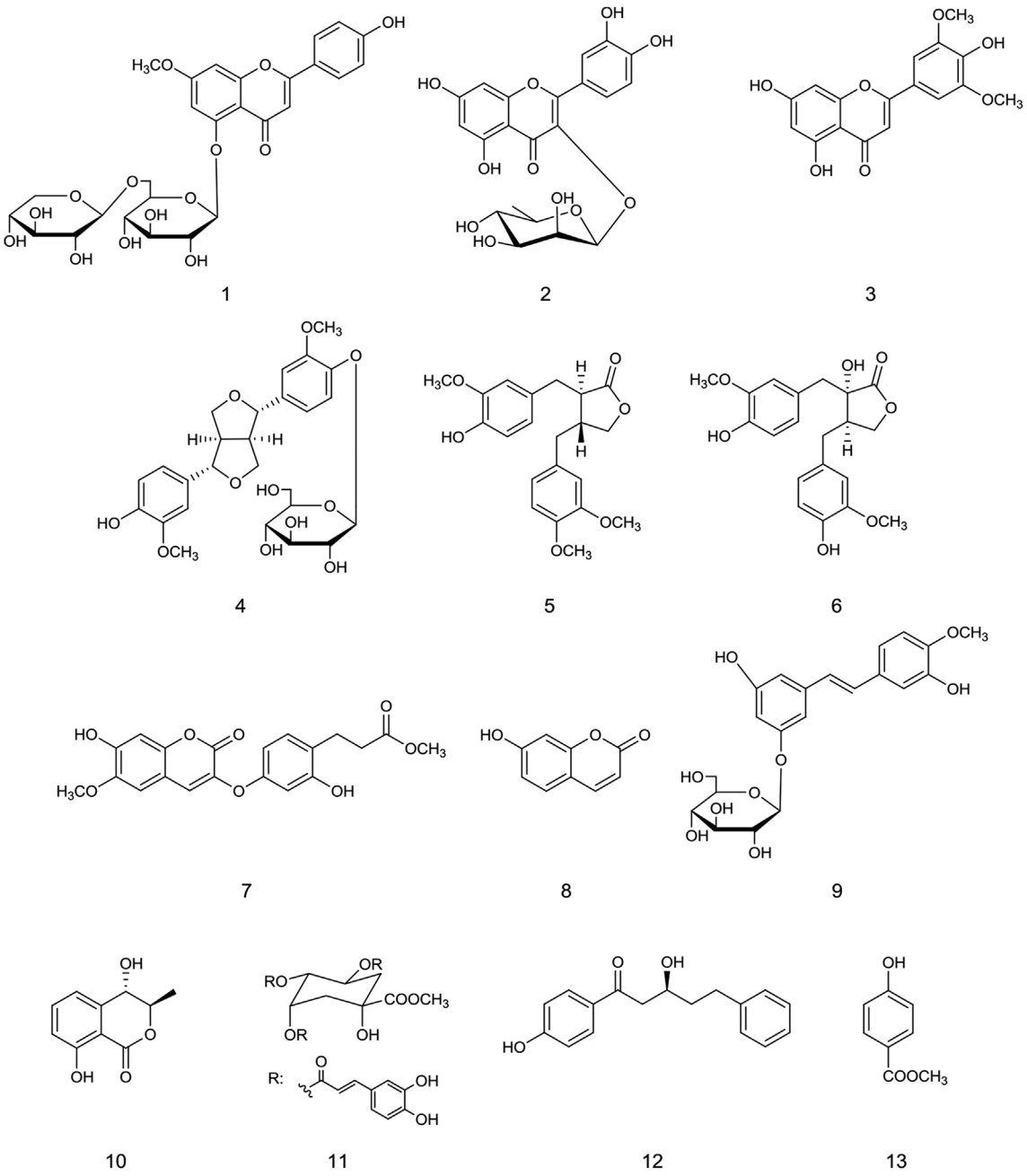

Fig. 1. Chemical structures of compounds **1–13** isolated from *W. indica*.

gel CC using $CHCl_3$-MeOH (100:0 → 100:20, v/v) as mobile phase to give 11 subfractions (Frs. E4a-E4k). Fr. E4a was purified over Sephadex LH-20 gel ($CHCl_3$-MeOH, 1:1, v/v) to afford compound **13** (10.8 mg). Fr. E4d was separated by Sephadex LH-20 gel with $CHCl_3$-MeOH (1:1, v/v) to give 5 subfractions (Frs. E4d1-E4d5), and the mixture of compounds **6** and **8** (30.3 mg) was precipitated out from Fr. E4d5. The supernatant of Fr. E4d5 was applied to Sephadex LH-20 gel with $CHCl_3$-MeOH (1.5:1, v/v) as elution system to give compound **12** (7.6 mg). Compound **3** (22.3 mg) was recrystallized from Fr. E4f in methanol.

Fr. E8 was fractionated by silica gel CC ($CHCl_3$-MeOH, 100:0 → 100:3, v/v) to give 23 subfractions (Frs. E8a-E8w). Fr. E8e was further chromatographed on Sephadex LH-20 gel column eluting with $CHCl_3$-MeOH (1:1, v/v) to give 7 subfractions (Frs. E8e1-E8e7). Compound **7** (13.5 mg) was obtained from Fr. E8e3 through MCI CC eluted with MeOH-H_2O (30:70 → 45:55, v/v), and compounds **10** (8.4 mg) and **5** (44.2 mg) were obtained from Fr. E8q through MCI CC eluted with MeOH-H_2O (30:70 → 50:50, v/v).

Fr. E12 was subjected to silica gel eluted with $CHCl_3$-MeOH (100:5 → 100:20, v/v) to obtain 9 fractions (Frs. E12a-E12i). Fr. E12a was separated by Sephadex LH-20 gel with $CHCl_3$-MeOH (1:2, v/v) followed by purification with semi-preparative HPLC (50% MeOH in H_2O, 8 mL/min) to obtain compound **4** (23.7 mg, t_R = 47 min).

Fr. E15 was chromatographed over silica gel column using a solvent system of $CHCl_3$-MeOH (100:10 → 100:50, v/v) to obtain 26 subfractions (Frs. E15a-E15z). Subfraction E15l was submitted to Sephadex LH-20 gel eluted with $CHCl_3$-MeOH (2:1, v/v) to yield 9 subfractions (Fr. E15l1-E15l9). Fr. E15l4 was further separated by semi-preparative HPLC (40% MeOH in H_2O, 8 mL/min) to obtain compounds **9** (17.8 mg, t_R = 20 min) and **2** (9.3 mg, t_R = 43 min). Fr. E15° was eluted with $CHCl_3$-MeOH (100:5 → 100:50, v/v) through silica gel CC and followed by $CHCl_3$-MeOH (2:1, v/v) elution through Sephadex LH-20 gel to give compounds **11** (23.0 mg) and **1** (45.1 mg).