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Chemical Constituents of Callicarpa integrifolia: Postprint

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Abstract

Callicarpa integerrima has the effects of dispelling wind and dissipating masses, and treating rheumatic scrofula, but currently there are few reports on its chemical constituents. To investigate the chemical constituents of the roots and stems of Callicarpa integerrima, this experiment utilized silica gel column chromatography, Sephadex LH-20 dextran gel column chromatography, ODS reversed-phase silica gel column chromatography, and high-performance liquid chromatography and other modern separation methods to systematically separate and purify the 95% ethanol extract of the roots and stems of Callicarpa integerrima, and then used NMR and ESI-MS and other modern spectroscopic techniques for structural identification of the compounds. The results showed that: a total of 15 compounds were identified from the 95% ethanol extract of the roots and stems of Callicarpa integerrima: stigmast-4-en-3-one (1), (24R)-5α-stigmastane-3,6-dione (2), 2'-hydroxy-4'-methoxydihydrochalcone (3), α-amyrin (4), β-sitosterol (5), ursolic acid (6), 4-hydroxy-3-methoxybenzoic acid (7), 4-hydroxypyridine (8), p-hydroxybenzoic acid (9), forsythoside B (10), nepetifosides D (11), isoverbascoside (12), verbascoside (13), pedicularioside M (14), β-methoxyforsythoside B (15). Except for compounds 4-6, 12, and 13, all other compounds were isolated from Callicarpa integerrima for the first time, among which compounds 1, 2, 3, 8, 11, and 14 were isolated from the genus Callicarpa for the first time.

Full Text

Preamble

Chemical Constituents of *Callicarpa integerrima*

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Abstract: *Callicarpa integerrima* has traditionally been used for dispelling wind and dissipating masses, as well as treating rheumatism and scrofula. However, few studies have reported on its chemical composition. To investigate the chemical constituents from the roots and stems of *C. integerrima*, this study systematically isolated and purified the 95% ethanol extract of the roots and stems using modern separation techniques including silica gel column chromatography, Sephadex LH-20 gel column chromatography, ODS reversed-phase silica gel column chromatography, and high-performance liquid chromatography (HPLC). The structures of the isolated compounds were then identified using modern spectroscopic techniques such as NMR and ESI-MS. The results identified fifteen compounds from the 95% ethanol extract of *C. integerrima* roots and stems: stigmast-4-en-3-one (1), (24R)-5α-stigmastane-3,6-dione (2), 2'-hydroxy-4' -methoxydihydrochalcone (3), α-amyrin (4), β-sitosterol (5), ursolic acid (6), 4-hydroxy-3-methoxy-benzoic acid (7), 4-pyridinol (8), p-hydroxybenzoic acid (9), forsythoside B (10), nepetifosides D (11), isoacteoside (12), acteoside (13), pedicularioside M (14), and β-methoxy forsythoside B (15). Except for compounds 4–6 and 12–13, all other compounds were isolated from *C. integerrima* for the first time, among which compounds 1, 2, 3, 8, 11, and 14 were reported from the genus *Callicarpa* for the first time.

Keywords: *Callicarpa*; *Callicarpa integerrima*; chemical constituents; phenylethanoids; triterpenoids

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Introduction

Plants of the genus *Callicarpa* have a long history of medicinal use, first documented in *Bencao Shiyi* (Supplement to the Compendium of Materia Medica). They are commonly used in folk medicine for hemostasis and detoxification. The chemical constituents of this genus are primarily classified into terpenoids, phenylethanoid glycosides, and flavonoids, exhibiting pharmacological activities such as hemostasis, antibacterial, anti-inflammatory, and memory improvement effects (Zhan et al., 2020). Currently, the Chinese Pharmacopoeia (2020 edition) includes four *Callicarpa* species: *Callicarpa kwangtungensis*, *Callicarpa nudiflora*, *Callicarpa macrophylla*, and *Callicarpa formosana*. Clinical drugs developed with these four species as the main active ingredients have been widely applied, such as Kangongyan capsules and tablets (*C. kwangtungensis*), Luohuazizhu tablets, antibacterial gel, granules, and capsules (*C. nudiflora*), Zidingxue powder and Sanqi Xueshangning capsules (*C. macrophylla*), and No. 11 hemostatic powder, Zhiyanxiao granules, and Fuyanling capsules (*C. formosana*)

(Mo & Li, 2019; Wu et al., 2019). These examples demonstrate the broad development potential of *Callicarpa* plant resources.

Callicarpa integerrima is a rare vine species in the Lamiaceae family and is endemic to China (Wang et al., 1986). It typically grows in forests on mountain slopes or valleys at altitudes of 200–700 m and is mainly distributed in Guangxi, Guangdong, Jiangxi, Fujian, and other provinces/regions (Editorial Committee of Flora of China, 1982). The roots and leaves of *C. integerrima* are used medicinally for dispelling wind, dissipating masses, and treating rheumatism and scrofula (Chai et al., 2010). Previous studies have identified diterpenoids, triterpenoids, flavonoids, and volatile oil components from this plant (Wang et al., 1986; Chai et al., 2010; Zhu et al., 2012; Di et al., 2021). For decades, domestic and international research has focused primarily on the chemical and pharmacological activities of arboreal and shrub species within the genus, such as *C. nudiflora*, *C. kwangtungensis*, and *C. formosana*, while few reports have addressed the chemical constituents of the rare vine species *C. integerrima*, leaving its pharmacological material basis unclear. As *C. integerrima* is a commonly used folk medicine in southern China, elucidating its chemical constituents is fundamental for ensuring its safe use and resource development. Therefore, to better understand and utilize this medicinal plant, clarify its chemical composition and pharmacological basis, and further develop this distinctive resource, this study identified fifteen compounds from the root and stem extracts of *C. integerrima* [Figure 1: see original paper]: stigmast-4-en-3-one (1), (24R)-5α-stigmastane-3,6-dione (2), 2'-hydroxy-4'-methoxydihydrochalcone (3), α-amyrin (4), β-sitosterol (5), ursolic acid (6), 4-hydroxy-3-methoxy-benzoic acid (7), 4-pyridinol (8), p-hydroxybenzoic acid (9), forsythoside B (10), nepetifosides D (11), isoacteoside (12), acteoside (13), pedicularioside M (14), and β-methoxy forsythoside B (15). Except for compounds 4–6 and 12–13, all other compounds were isolated from *C. integerrima* for the first time, with compounds 1, 2, 3, 8, 11, and 14 being reported from the genus *Callicarpa* for the first time. These findings enrich the compound library of the *Callicarpa* genus, provide a foundation for further systematic investigation of the pharmacological activities and mechanisms of action of *C. integerrima*, and offer a scientific basis for the rational development and utilization of medicinal *Callicarpa* resources.

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1. Instruments and Materials

A DHG-9240 electrothermal constant-temperature blast drying oven (Shanghai Jinghong Experimental Equipment Co., Ltd.), SHB- A circulating water vacuum pump (Gongyi Yuhua Instrument Co., Ltd.), AR224CN electronic analytical balance (Ohaus Instruments Co., Ltd.), CX-1000A pulverizer (Shanghai Shengxi Pharmaceutical Machinery Co., Ltd.), 3510E-DTH ultrasonic cleaner (Branson, USA), KQ-800DE numerical control ultrasonic cleaner (Kunshan Ultrasound Instrument Co., Ltd.), AVIII HD 600 and AVIII 500 NMR spectrometers (Bruker, Switzerland), Shimadzu LC-2030C 3D Plus HPLC system

(Shimadzu, Japan), HH-S4 digital display constant-temperature water bath (Changzhou Jintan Liangyou Instrument Co., Ltd.), Zhengzhou Great Wall DLSB-10/20 low-temperature cooling circulator (Zhengzhou Great Wall Scientific Industrial and Trade Co., Ltd.), Shanghai EYELA OSB-2200 rotary evaporator (Shanghai Ailang Instrument Co., Ltd.), WFH-203B darkroom UV analyzer (Hangzhou Qiwei Instrument Co., Ltd.), MCI GEL CHP20P resin filler (Mitsubishi Chemical, Japan), ODS reversed-phase chromatography filler C18 MB100-40/75 (Fuji Chemical Co., Ltd.), analytical HPLC column YMC-Pack ODS-A (250 mm × 4.6 mm, 5 m), semi-preparative HPLC column YMC-Pack ODS-A (250 mm × 10 mm, 5 m), UPH-II-20T ultra-pure water system (Nanjing Youpu Instrument Equipment Co., Ltd.), silica gel for column chromatography (100–200 mesh and 200–300 mesh, Yantai Jiangyou Silica Gel Development Co., Ltd.), GF254 TLC plates (Qingdao Puke Separation Materials Co., Ltd.), color-developing agent 10% sulfuric acid-ethanol solution applied and heated for visualization, polyamide (Taizhou Lujiang Sijia Biochemical Plastic Factory, Zhejiang), and Sephadex LH-20 dextran gel (40–70 m, GE Healthcare).

Petroleum ether and n-butanol were purchased from Tianjin Fuyu Fine Chemical Co., Ltd., ethyl acetate from Tianjin Damao Chemical Reagent Factory, methanol, dichloromethane, and chloroform from Shanghai Titan Chemical Co., Ltd. (all analytical grade). Deuterated reagents were purchased from Cambridge Isotope Laboratories, Inc. HPLC-grade acetonitrile and methanol were obtained from Shanghai Xingke High Purity Solvents.

Plant samples of *Callicarpa integerrima* were collected in April 2019 near Wenquan Police Station, Wenquan Town, Conghua District, Guangzhou City, Guangdong Province, and identified by Associate Professor MA Zhonghui of the College of Agriculture, Guangxi University. A voucher specimen (collection number: 20190426) is deposited in the Herbarium of the College of Agriculture, Guangxi University (GAUA).

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2. Extraction and Isolation

Air-dried roots (0.55 kg) and stems (9.78 kg) of *C. integerrima* were pulverized and extracted three times each with 95% ethanol at room temperature for one week. The extracts were concentrated under reduced pressure to yield root ethanol extract (41.66 g) and stem ethanol extract (448.50 g). The stem ethanol extract (448.50 g) was suspended in hot water and sequentially extracted with petroleum ether, ethyl acetate, and n-butanol. After concentration under reduced pressure, petroleum ether (46.26 g), ethyl acetate (72.90 g), and n-butanol (106.56 g) fractions were obtained.

The root ethanol extract (41.66 g) was subjected to polyamide column chromatography with a methanol-water gradient elution (0:100 → 30:70 → 50:50 → 70:30 → 100:0) to afford eleven fractions (Fr.1–Fr.11). Fraction Fr.9 (1.00 g) was purified by Sephadex LH-20 column chromatography (chloroform:methanol =

1:1) and recrystallized from chloroform-methanol mixture to yield compounds **1** (7.10 mg) and **2** (11.70 mg). Compound **3** (2.60 mg) was obtained by preparative TLC.

The stem petroleum ether extract (46.26 g) was subjected to silica gel column chromatography with a petroleum ether-dichloromethane gradient elution (98:2 → 5:5) to afford fifteen fractions (Fr.1–Fr.15). Fraction Fr.10 (5.69 g) was repeatedly purified by Sephadex LH-20 column chromatography (chloroform:methanol = 1:1), silica gel column chromatography (petroleum ether:dichloromethane = 19:1), and ODS reversed-phase silica gel column (90% aqueous methanol) to obtain compound **4** (21.20 mg). Compound **5** (10.11 mg) was obtained by recrystallization from chloroform-methanol mixture. Fraction Fr.14 (6.80 g) was purified by MCI column chromatography (85% aqueous methanol) and silica gel column chromatography (petroleum ether:dichloromethane:methanol = 50:99:1) to yield compound **6** (11.40 mg).

The stem ethyl acetate extract (72.90 g) was subjected to silica gel column chromatography with a dichloromethane-methanol gradient elution (98:2 → 5:5) to afford thirteen fractions (Fr.1–Fr.13). Fraction Fr.5 (3.64 g) was purified by Sephadex LH-20 column chromatography (chloroform:methanol = 1:1) and then by semi-preparative HPLC with 18% aqueous acetonitrile isocratic elution at a flow rate of 2 $mL \cdot min^{-1}$ to obtain compound **7** (10.00 mg, tR = 19 min) and compound **8** (4.50 mg, tR = 27 min). Fraction Fr.6 (2.56 g) was purified by Sephadex LH-20 column chromatography (chloroform:methanol = 1:1) and then by semi-preparative HPLC with 20% aqueous acetonitrile (containing 0.1% formic acid) isocratic elution at 2 $mL \cdot min^{-1}$ to yield compound **9** (5.22 mg, tR = 15 min). Fraction Fr.12 (56.11 g) was repeatedly purified by silica gel column chromatography (dichloromethane:methanol = 95:5 → 5:5), medium-pressure ODS reversed-phase column chromatography (acetonitrile-water = 15% → 100%), and Sephadex LH-20 column chromatography (chloroform:methanol = 1:1) to obtain compound **10** (69.90 mg). Subsequent semi-preparative HPLC purification with 21% aqueous acetonitrile (containing 0.1% formic acid) isocratic elution at 2 $mL \cdot min^{-1}$ yielded compound **11** (7.04 mg, tR = 52 min). Using 34% aqueous methanol isocratic elution at 2 $mL \cdot min^{-1}$ gave compound **12** (40.00 mg, tR = 31 min). With 35% aqueous methanol isocratic elution at 2 $mL \cdot min^{-1}$, compounds **13** (94.00 mg, tR = 20 min) and **14** (13.00 mg, tR = 27 min) were obtained. Compound **15** (10.62 mg, tR = 36 min) was obtained using 15% aqueous acetonitrile (containing 0.1% formic acid) isocratic elution at 2 $mL \cdot min^{-1}$.

[Figure 1: see original paper] Chemical structures of compounds 1–15

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3. Structure Identification

Compound 1 was obtained as white needle crystals with molecular formula $C_{29}H_{48}O$. ESI-MS m/z: 413 $[M + H]^+$. ^1H NMR (600 MHz, $CDCl_3$): δH 5.74

(1H, s, H-4), 0.92 (3H, s, H-19), 0.90 (3H, d, J = 7.1 Hz, H-21), 0.86 (3H, t, J = 8.1 Hz, H-29), 0.85 (3H, d, J = 7.1 Hz, H-26), 0.82 (3H, d, J = 6.6 Hz, H-27), 0.70 (3H, s, H-18). ^{13}C NMR (150 MHz, $CDCl_3$): δC 198.8 (C-3), 171.6 (C-5), 123.9 (C-4), 56.1 (C-17), 55.9 (C-14), 53.8 (C-9), 45.9 (C-24), 42.3 (C-13), 39.8 (C-10, C-12), 38.7 (C-8), 36.1 (C-20), 35.6 (C-1), 33.9 (C-2), 33.8 (C-22), 32.9 (C-6), 32.0 (C-7), 29.1 (C-25), 28.2 (C-16), 26.0 (C-23), 24.2 (C-15), 23.0 (C-28), 21.0 (C-11), 19.9 (C-27), 19.1 (C-26), 18.8 (C-21), 17.6 (C-19), 12.1 (C-18/29). These data are consistent with those reported in the literature for stigmast-4-en-3-one (Abdelhameed et al., 2020). Therefore, compound **1** was identified as stigmast-4-en-3-one.

Compound 2 was obtained as white needle crystals with molecular formula $C_{29}H_{48}O_2$. ESI-MS m/z: 429 [M + H]$^+$. ^{1}H NMR (600 MHz, $CDCl_3$): δH 0.95 (3H, s, H-18), 0.92 (3H, d, J = 7.1 Hz, H-21), 0.86 (3H, t, J = 8.1 Hz, H-29), 0.83 (3H, d, J = 6.9 Hz, H-26), 0.81 (3H, d, J = 6.4 Hz, H-27), 0.70 (3H, s, H-19). ^{13}C NMR (150 MHz, $CDCl_3$): δC 211.4 (C-6), 209.1 (C-3), 57.5 (C-5), 56.6 (C-17), 56.0 (C-14), 53.5 (C-9), 46.6 (C-7), 45.8 (C-24), 43.0 (C-13), 41.2 (C-10), 39.4 (C-2), 38.1 (C-1), 38.0 (C-8), 37.4 (C-12), 37.0 (C-4), 36.0 (C-20), 33.9 (C-22), 29.1 (C-25), 28.0 (C-16), 26.0 (C-23), 24.0 (C-15), 23.0 (C-28), 21.7 (C-11), 19.9 (C-26), 19.1 (C-27), 18.6 (C-21), 12.7 (C-18), 12.1 (C-19/29). These data are consistent with those reported in the literature for (24R)-5α-stigmastane-3,6-dione (Li et al., 2014). Therefore, compound **2** was identified as (24R)-5α-stigmastane-3,6-dione.

Compound 3 was obtained as a white amorphous powder with molecular formula $C_{16}H_{16}O_3$. ESI-MS m/z: 257 [M + H]$^+$. ^{1}H NMR (600 MHz, CD_3OD): δH 7.53 (1H, d, J = 8.5 Hz, H-6'), 7.22 (5H, m, H-2/3/4/5/6), 6.36 (1H, d, J = 1.9 Hz, H-3'), 6.40 (1H, dd, J = 8.5, 2.1 Hz, H-5'), 3.83 (3H, s, H-4'), 3.22 (2H, t, J = 7.6 Hz, H-α), 2.90 (2H, t, J = 7.6 Hz, H-β). ^{13}C NMR (150 MHz, CD_3OD): δC 201.8 (C=O), 164.8 (C-4'), 163.0 (C-2'), 143.1 (C-1), 133.6 (C-6'), 129.5 (C-2/3/5/6), 127.0 (C-4), 113.3 (C-1'), 108.9 (C-3'), 99.9 (C-5'), 56.0 (4' -OCH_3), 46.2 (C-α), 32.2 (C-β). These data are consistent with those reported in the literature for 2' -hydroxy-4' -methoxydihydrochalcone (Edyta et al., 2017). Therefore, compound **3** was identified as 2' -hydroxy-4' -methoxydihydrochalcone.

Compound 4 was obtained as white needle crystals with molecular formula $C_{30}H_{50}O$. ESI-MS m/z: 427 [M + H]$^+$. ^{1}H NMR (600 MHz, $CDCl_3$): δH 5.13 (1H, t, J = 3.7 Hz, H-12), 3.22 (1H, dd, J = 11.3, 4.8 Hz, H-3), 1.07 (3H, s, H-27), 1.01 (3H, s, H-26), 1.00 (3H, s, H-23), 0.95 (3H, d, J = 0.8 Hz, H-25), 0.91 (3H, s, H-30), 0.87 (3H, s, H-28), 0.80 (3H, s, H-24), 0.74 (3H, d, J = 11.8 Hz, H-29). ^{13}C NMR (150 MHz, $CDCl_3$): δC 139.6 (C-13), 124.4 (C-12), 79.1 (C-3), 59.1 (C-18), 55.2 (C-5), 47.7 (C-9), 42.1 (C-14), 41.5 (C-22), 40.0 (C-8), 39.7 (C-19), 39.6 (C-20), 38.8 (C-1), 38.8 (C-4), 36.9 (C-10), 33.8 (C-17), 33.0 (C-7), 31.3 (C-21), 28.8 (C-15), 28.1 (C-23), 28.1 (C-28), 27.3 (C-2), 26.6 (C-16), 23.4 (C-11), 23.3 (C-27), 21.4 (C-29), 18.4 (C-6), 17.5 (C-30), 16.9 (C-26), 15.7 (C-25), 15.6 (C-24). These data are consistent with those reported in the

literature for α-amyrin (Mo et al., 2020). Therefore, compound **4** was identified as α-amyrin.

Compound 5 was obtained as a white powder with molecular formula $C_{29}H_{50}O$. ESI-MS m/z: 415 $[M + H]^+$. ^1H NMR (600 MHz, $CDCl_3$): δH 5.35 (1H, m, H-6), 3.52 (1H, m, H-3), 1.01 (3H, s, H-19), 0.92 (3H, d, J = 6.5 Hz, H-21), 0.84 (3H, d, J = 2.9 Hz, H-26), 0.83 (3H, m, H-29), 0.81 (3H, m, H-27), 0.69 (3H, s, H-18). ^{13}C NMR (150 MHz, $CDCl_3$): δC 140.8 (C-5), 121.7 (C-6), 71.8 (C-3), 56.8 (C-14), 56.1 (C-17), 50.1 (C-9), 45.9 (C-24), 42.3 (C-4), 42.3 (C-13), 39.8 (C-12), 37.3 (C-1), 36.5 (C-10), 36.2 (C-20), 34.0 (C-22), 31.9 (C-7), 31.9 (C-8), 31.9 (C-2), 29.2 (C-25), 28.3 (C-16), 26.0 (C-23), 24.3 (C-15), 23.0 (C-28), 21.1 (C-11), 19.8 (C-26), 19.4 (C-19), 19.0 (C-27), 18.8 (C-21), 12.0 (C-29), 11.9 (C-18). These data are consistent with those reported in the literature for β-sitosterol (Cha et al., 2021). Therefore, compound **5** was identified as β-sitosterol.

Compound 6 was obtained as a white powder with molecular formula $C_{30}H_{48}O_3$. ESI-MS m/z: 457 $[M + H]^+$. ^1H NMR (600 MHz, CD_3OD): δH 5.26 (1H, t, J = 3.7 Hz, H-12), 3.29 (1H, dd, J = 3.3, 1.6 Hz, H-3), 1.32 (3H, m, H-27), 1.30 (3H, s, H-29), 1.27 (3H, s, H-24), 1.16 (3H, s, H-23), 0.90 (3H, m, H-30), 0.83 (3H, s, H-25), 0.75 (3H, s, H-26). ^{13}C NMR (150 MHz, DMSO): δC 179.3 (C-28), 138.9 (C-13), 127.1 (C-12), 78.2 (C-3), 55.9 (C-5), 53.6 (C-18), 48.1 (C-17), 48.1 (C-9), 42.6 (C-14), 40.1 (C-8), 39.6 (C-1), 39.5 (C-4), 39.4 (C-19), 39.2 (C-20), 37.5 (C-22), 37.4 (C-10), 33.7 (C-7), 31.1 (C-21), 28.8 (C-23), 28.8 (C-2), 28.2 (C-15), 25 (C-16), 24 (C-11), 23.7 (C-27), 21.4 (C-30), 18.8 (C-6), 17.5 (C-26), 17.5 (C-29), 16.5 (C-24), 15.7 (C-25). These data are consistent with those reported in the literature for ursolic acid (Chen et al., 2020). Therefore, compound **6** was identified as ursolic acid.

Compound 7 was obtained as a white powder with molecular formula $C_8H_8O_4$. ESI-MS m/z: 169 $[M + H]^+$. ^1H NMR (600 MHz, CD_3OD): δH 7.56 (1H, d, J = 1.8 Hz, H-2), 7.55 (1H, d, J = 2.0 Hz, H-6), 6.86–6.81 (1H, m, H-5), 3.89 (3H, s, 8-OCH_3). ^{13}C NMR (150 MHz, CD_3OD): δC 167.6 (C-7), 152.7 (C-4), 148.8 (C-3), 125.3 (C-6), 123.5 (C-1), 115.9 (C-2), 114.1 (C-5), 56.6 (8-OCH_3). These data are consistent with those reported in the literature for 4-hydroxy-3-methoxy-benzoic acid (Li et al., 2006). Therefore, compound **7** was identified as 4-hydroxy-3-methoxy-benzoic acid.

Compound 8 was obtained as a white powder with molecular formula C_5H_5NO. ESI-MS m/z: 96 $[M + H]^+$. ^1H NMR (600 MHz, DMSO-d_6): δH 9.76 (1H, s, 4-OH), 7.74 (2H, d, J = 8.6 Hz, H-2/6), 6.91 (2H, d, J = 8.2 Hz, H-3/5). ^{13}C NMR (150 MHz, DMSO-d_6): δC 160.7 (C-4), 149.8 (C-2/6), 115.9 (C-3/5). These data are consistent with those reported in the literature for 4-pyridinol (Wang, 2008). Therefore, compound **8** was identified as 4-pyridinol.

Compound 9 was obtained as a white powder with molecular formula $C_7H_6O_3$. ESI-MS m/z: 139 $[M + H]^+$. ^1H NMR (600 MHz, CD_3OD): δH 7.87 (2H, d, J = 8.7 Hz, H-2/6), 6.80 (2H, d, J = 8.7 Hz, H-3/5). ^{13}C NMR (150 MHz, CD_3OD):

δC 171.6 (C-7), 163.1 (C-4), 133.0 (C-2/6), 124.1 (C-1), 116.0 (C-3/5). These data are consistent with those reported in the literature for p-hydroxybenzoic acid (Li et al., 2014). Therefore, compound **9** was identified as p-hydroxybenzoic acid.

Compound 10 was obtained as a brownish-red solid with molecular formula $C_{34}H_{44}O_{19}$. ESI-MS m/z: 779 $[M+Na]^{+}$. ^{1}H NMR (600 MHz, CD_3OD): δH 7.62 (1H, d, J = 15.8 Hz, Caf-7' '), 7.10 (1H, d, J = 2.0 Hz, Caf-2' '), 6.99 (1H, dd, J = 8.2, 2.1 Hz, Caf-6' '), 6.82 (1H, d, J = 8.2 Hz, Caf-5' '), 6.75–6.71 (2H, m, Agl H-2/5), 6.60 (1H, dd, J = 8.1, 2.1 Hz, Agl H-6), 6.31 (1H, d, J = 15.9 Hz, Caf H-8' '), 5.21 (1H, d, J = 1.8 Hz, Rha H-1' ' '), 4.98 (1H, m, Glu H-4'), 4.94 (1H, d, J = 2.3 Hz, Api H-1' ' ' '), 4.40 (1H, d, J = 7.9 Hz, GluH-1'), 4.02 (1H, m, Agl H-8), 3.96–3.90 (2H, m, Rha-2' ' ', Api-4' ' ᵒ a), 3.83 (1H, m, Glu-3'), 3.80–3.69 (4H, m, Glu-5' /6' , Api-2' ' ' '/4' ' ᵒ b), 3.64–3.60 (1H, m, Rha-3' ' '), 3.58 (2H, s, Api-5' ' ' '), 3.51 (1H, dd, J = 11.2, 5.7 Hz, Glu-2'), 3.35–3.30 (2H, m, Rha-4' ' '/5' ' '), 2.82 (2H, m, Agl-7), 1.11 (3H, d, J = 6.2 Hz, Rha-6' ' '). ^{13}C NMR (150 MHz, CD_3OD): δC 168.3 (Caf C-9' '), 149.8 (Caf C-4' '), 148.2 (Caf C-7' '), 146.8 (Caf C-3' '), 146.1 (Agl C-3), 144.7 (Agl C-4), 131.6 (Agl C-1), 127.7 (Caf C-1' '), 123.4 (Caf C-6' '), 121.5 (Agl C-6), 117.3 (Agl C-2), 116.7 (Caf C-5' '), 116.5 (Agl C-5), 115.4 (Caf C-2' '), 114.8 (Caf C-8' '), 111.1 (Api C-1' ' ' '), 104.2 (Glu C-1'), 103.1 (Rha C-1' ' '), 81.8 (Glu C-3'), 80.7 (Api C-3' ' ' '), 78.3 (Api C-2' ' ' '), 76.2 (Glu C-2'), 75.2 (Api C-4' ' ' '), 74.6 (Glu C-5'), 73.8 (Rha C-4' ' '), 72.4 (Rha C-2' ' '), 72.4 (Agl C-8), 72.1 (Rha C-3' ' '), 70.9 (Glu C-4'), 70.5 (Rha C-5' ' '), 68.5 (Glu C-6'), 65.7 (Api C-5' ' ' '), 36.7 (Agl C-7), 18.5 (Rha C-6' ' '). These data are consistent with those reported in the literature for forsythoside B (Yamasaki et al., 2007). Therefore, compound **10** was identified as forsythoside B.

Compound 11 was obtained as a yellow-green solid with molecular formula $C_{36}H_{48}O_{20}$. ESI-MS m/z: 823 $[M +Na]^{+}$. ^{1}H NMR (600 MHz, DMSO-d_6): δH 7.54 (1H, d, J = 15.8 Hz, Acyl H-7' '), 7.29 (1H, d, J = 2.0 Hz, Acyl H-2' '), 7.09 (1H, dd, J = 8.2, 2.0 Hz, Acyl H-6' '), 6.79 (1H, d, J = 8.1 Hz, Acyl H-5' '), 6.70 (2H, d, J = 8.1 Hz, Agl H-2/5), 6.58 (1H, dd, J = 8.1, 2.1 Hz, Agl H-6), 6.42 (1H, d, J = 15.9 Hz, Acyl H-8' '), 5.03 (1H, d, J = 1.7 Hz, Rha H-1' ' '), 4.76 (1H, d, J = 2.9 Hz, Api H-1' ' ' '), 4.68 (1H, t, J = 9.7 Hz, Glu H-4'), 4.42 (1H, d, J = 7.9 Hz, Glu H-1'), 4.25 (1H, dd, J = 7.9, 3.8 Hz, Agl H-7), 3.80 (3H, s, 3' '-OCH_3), 3.78 (1H, s, Agl H-8a), 3.69 (3H, m, Glu H-5' , Rha H-2' ' ', Api H-2' ' ' '), 3.62 (3H, m, Api H-3' ' ' '/4' ' ' '), 3.57–3.48 (3H, m, Agl H-8b, Glu H-2' /3'), 3.40–3.30 (2H, m, Glu H-6' , Rha H-5' ' '), 3.26 (3H, m, Rha H-3' ' ', Api H-5' ' ' '), 3.13 (3H, s,7-OCH_3), 3.09 (1H, d, J = 9.4 Hz, Rha H-4' ' '), 0.98 (3H, d, J = 6.2 Hz, Rha H-6' ' '). ^{13}C NMR (150 MHz, DMSO-d_6): δC 165.8 (Acyl C-9' '), 149.5 (Acyl C-3' '), 147.9 (Acyl C-4' '), 145.8 (Agl C-4), 145.2 (Acyl C-7' '), 145.0 (Agl C-3), 129.7 (Agl C-1), 125.5 (Acyl C-1' '), 123.3 (Acyl C-6' '), 118.1 (Agl C-6), 115.5 (Agl C-5, Acyl C-5' '), 114.1 (Agl C-2), 113.9 (Acyl C-8' '), 111.1 (Acyl C-2' '), 109.1 (Api C-1' ' ' '), 102.8 (Glu C-1'), 101.3 (Rha C-1' ' '), 82.3 (Agl C-7), 78.9 (Api C-3' ' ' '), 78.8 (Glu C-3'), 75.9 (Api C-2' ' ' '), 74.3 (Glu C-2'), 73.7 (Agl C-8), 73.4 (Api C-4' ' ' '), 72.8 (Glu C-5'

), 71.7 (Rha C-4'' '), 70.5 (Rha C-2'' '), 70.4 (Rha C-3'' '), 69.3 (Glu C-4'), 68.8 (Rha C-5'' '), 67.1 (Glu C-6'), 63.2 (Api C-5''' '), 56.0 (7-OCH_3), 55.6 (3' '-OCH_3), 18.1 (Rha C-6'''). These data are consistent with those reported in the literature for nepetifosides D (Xu et al., 2019). Therefore, compound **11** was identified as nepetifosides D.

Compound 12 was obtained as an orange-red paste with molecular formula $C_{29}H_{36}O_{15}$. ESI-MS m/z: 647 $[M+Na]^+$. ^1H NMR (600 MHz, CD_3OD): δH 7.57 (1H, d, J = 15.8 Hz, Caf H-7' '), 7.05 (1H, d, J = 2.1 Hz, Caf H-2' '), 6.89 (1H, dd, J = 8.2, 2.1 Hz, Caf H-6' '), 6.78 (1H, d, J = 8.2 Hz, Caf H-5' '), 6.69 (1H, d, J = 2.0 Hz, Agl H-2), 6.65 (1H, d, J = 8.0 Hz, Agl H-5), 6.54 (1H, dd, J = 8.0, 2.0 Hz, Agl H-6), 6.30 (1H, d, J = 15.9 Hz, Caf H-8' '), 5.20 (1H, d, J = 1.7 Hz, Rha H-1'' '), 4.51 (1H, dd, J = 11.9, 2.2 Hz, Glu H-6a'), 4.39–4.36 (1H, m, Glu H-6b'), 4.34 (1H, d, J = 7.9 Hz, Glu H-1'), 4.04–3.93 (3H, m, Agl H-8b, Rha H-2'' '/4'' '), 3.75–3.69 (2H, m, Agl H-8a, Rha H-3'' '), 3.58–3.55 (1H, m, Glu H-5'), 3.54 (1H, d, J = 8.9 Hz, Glu H-3'), 3.42 (1H, m, Glu H-4'), 3.35–3.30 (1H, m, Glu H-2'), 2.83–2.73 (2H, m, Agl H-7), 1.26 (3H, d, J = 6.2 Hz, Rha H-6'' '). ^{13}C NMR (150 MHz, DMSO-d_6): δC 169.2 (Caf C-9' '), 149.5 (Caf C-4' '), 147.2 (Caf C-7' '), 146.7 (Caf C-3' '), 146.0 (Agl C-3), 144.6 (Agl C-4), 131.4 (Agl C-1), 127.6 (Caf C-1' '), 123.2 (Caf C-6' '), 121.3 (Agl C-6), 117.1 (Agl C-2), 116.5 (Agl C-5), 116.4 (Caf C-5' '), 115.1 (Caf C-2' '), 114.8 (Caf C-8' '), 104.3 (Glu C-1'), 102.6 (Rha C-1'' '), 83.9 (Glu C-3'), 75.6 (Glu C-2'), 75.3 (Glu C-5'), 73.9 (Rha C-4'' '), 72.4 (Agl C-8), 72.3 (Rha C-3'' '), 72.2 (Rha C-2'' '), 70.3 (Glu C-4'), 70.0 (Rha C-5'' '), 64.6 (Glu C-6'), 36.6 (Agl C-7), 17.9 (Rha C-6'''). These data are consistent with those reported in the literature for isoacteoside (Saimaru & Orihara, 2010). Therefore, compound **12** was identified as isoacteoside.

Compound 13 was obtained as an orange-red paste with molecular formula $C_{29}H_{36}O_{15}$. ESI-MS m/z: 647 $[M+Na]^+$. ^1H NMR (600 MHz, DMSO-d_6): δH 7.46 (1H, d, J = 15.8 Hz, Caf H-7' '), 7.03 (1H, d, J = 2.0 Hz, Caf H-2' '), 6.97 (1H, dd, J = 8.2, 2.1 Hz, Caf H-6' '), 6.76 (1H, d, J = 8.1 Hz, Caf H-5' '), 6.65–6.61 (2H, m, Agl H-2/5), 6.49 (1H, dd, J = 8.1, 2.1 Hz, Agl H-6), 6.20 (1H, d, J = 15.9 Hz, Caf H-8' '), 4.72 (1H, m, Glu H-4'), 4.35 (1H, d, J = 7.9 Hz, Glu H-1'), 3.88 (1H, dd, J = 9.2, 6.4 Hz, Rha H-2'' '), 3.72–3.07 (10H, m, Agl H-8, Rha/Glu-H), 2.76–2.63 (2H, m, Agl H-7), 0.96 (3H, d, J = 6.1 Hz, Rha H-6'' '). ^{13}C NMR (150 MHz, DMSO-d_6): δC 166.0 (Caf C-9' '), 148.8 (Caf C-3' '), 145.8 (Caf C-7' '), 145.2 (Agl C-3, Caf C-4' '), 143.8 (Agl C-4), 129.4 (Agl C-1), 125.7 (Caf C-1' '), 121.7 (Caf C-6' '), 119.8 (Agl C-6), 116.5 (Caf C-5' '), 116.0 (Agl C-2), 115.7 (Agl C-5), 114.9 (Caf C-8' '), 113.8 (Caf C-2' '), 102.5 (Glu C-1'), 101.5 (Rha C-1'' '), 79.3 (Glu C-3'), 74.7 (Glu C-5'), 74.7 (Glu C-2'), 71.9 (Rha C-4'' '), 70.8 (Rha C-2'' '), 70.6 (Rha C-3'' '), 70.5 (Agl C-8), 69.4 (Rha C-5'' '), 69.0 (Glu C-4'), 61.0 (Glu C-6'), 35.2 (Agl C-7), 18.4 (Rha C-6'''). These data are consistent with those reported in the literature for acteoside (Lan et al., 2018). Therefore, compound **13** was identified as acteoside.

Compound 14 was obtained as a yellow solid with molecular formula

$C_{35}H_{46}O_{19}$. ESI-MS m/z: 795 $[M+Na]^+$. ^{1}H NMR (600 MHz, DMSO-d_6): δH 7.54 (1H, d, J = 15.8 Hz, Acyl H-7' '), 7.29 (1H, d, J = 2.0 Hz, Acyl H-2' '), 7.09 (1H, dd, J = 8.3, 2.0 Hz, Acyl H-6' '), 6.79 (1H, d, J = 8.1 Hz, Acyl H-5' '), 6.59 (2H, m, Agl H-2/5), 6.50 (1H, dd, J = 8.1, 2.1 Hz, Agl H-6), 6.41 (1H, d, J = 15.9 Hz, Acyl H-8' '), 5.03 (1H, d, J = 1.7 Hz, Rha H-1'' '), 4.78 (1H, d, J = 2.9 Hz, Api H-1''' '), 4.64 (1H, t, J = 9.7 Hz, Glu H-4'), 4.38 (1H, d, J = 7.9 Hz, Glu H-1'), 3.80 (3H, s, 3' '-OCH_3), 3.76–3.63 (7H, m, Glu H-3' /5' , Api H-2''' '/3''' '/4''' ', Rha H-2'' '), 3.57–3.48 (2H, m, Agl H-8), 3.32–3.18 (6H, m, Api H-5''' ', Glu H-2' /6' , Rha H-3'' '/5'' '), 3.10 (1H, t, J = 9.4 Hz, Rha H-4'' '), 2.80–2.65 (2H, m, Agl-7), 0.98 (3H, d, J = 6.2 Hz, Rha H-6'' '). ^{13}C NMR (150 MHz, DMSO-d_6): δC 166.3 (Acyl C-9' '), 150.0 (Acyl C-3' '), 148.4 (Acyl C-4' '), 146.2 (Acyl C-7' '), 145.5 (Agl C-3), 144.0 (Agl C-4), 129.6 (Agl C-1), 126.0 (Acyl C-1' '), 123.7 (Acyl C-6' '), 120.0 (Agl C-6), 116.8 (Agl C-2), 116.0 (Agl C-5), 115.7 (Acyl C-8' '), 114.3 (Acyl C-5' '), 111.5 (Acyl C-2' '), 109.6 (Api C-1''' '), 102.7 (Glu C-1'), 101.7 (Rha C-1'' '), 79.3 (Glu C-3' , Api C-3''' '), 76.4 (Api C-2''' '), 74.8 (Glu C-2'), 73.9 (Api C-4''' '), 73.3 (Glu C-5'), 72.1 (Rha C-4'' '), 71.0 (Rha C-2'' '), 70.8 (Rha C-3'' '), 70.8 (Agl C-8), 69.9 (Glu C-4'), 69.2 (Rha C-5'' '), 67.6 (Glu C-6'), 63.6 (Api C-5''' '), 56.1 (3' '-OCH_3), 35.5 (Agl C-7), 18.6 (Rha C-6'''). These data are consistent with those reported in the literature for pedicularioside M (Jia & Gao, 1993). Therefore, compound **14** was identified as pedicularioside M.

Compound 15 was obtained as a yellow-green solid with molecular formula $C_{35}H_{46}O_{20}$. ESI-MS m/z: 809 $[M+Na]^+$. ^{1}H NMR (500 MHz, DMSO-d_6): δH 7.47 (1H, d, J = 15.8 Hz, Caf H-7' '), 7.03 (1H, d, J = 2.1 Hz, Caf H-2' '), 6.98 (1H, dd, J = 8.3, 2.1 Hz, Caf H-6' '), 6.76 (1H, d, J = 8.1 Hz, Caf H-5' '), 6.71–6.68 (2H, m, Agl H-2/5), 6.58 (1H, dd, J = 8.0, 2.0 Hz, Agl H-6), 6.20 (1H, d, J = 15.9 Hz, Caf H-8' '), 5.02 (1H, d, J = 1.5 Hz, Rha H-1'' '), 4.76 (1H, d, J = 2.8 Hz, Api H-1''' '), 4.68 (1H, t, J = 9.7 Hz, Glu H-4'), 4.42 (1H, d, J = 7.8 Hz, Glu H-1'), 4.25 (1H, dd, J = 7.8, 3.9 Hz, Agl H-7), 3.79 (1H, d, J = 9.3 Hz, Agl H-8a), 3.72–3.57 (7H, m, Glu H-3' /5' , Api H-2''' '/3''' '/4''' ', Rha H-2'' '), 3.57–3.46 (2H, m, Agl H-8b, Glu H-6' a), 3.36–3.17 (6H, m, Api H-5''' ', Glu H-2' /6' , Rha H-3'' '/5'' '), 3.13 (3H, d, J = 6.1 Hz,7-OCH_3), 3.09 (1H, d, J = 9.4 Hz, Rha H-4'' '), 0.96 (3H, d, J = 6.2 Hz, Rha H-6'' '). ^{13}C NMR (125 MHz, DMSO-d_6): δC 165.7 (Caf C-9' '), 148.5 (Caf C-4' '), 145.8 (Caf C-7' '), 145.6 (Caf C-3' '), 145.2 (Agl C-4), 145.0 (Agl C-3), 129.7 (Agl C-1), 125.5 (Caf C-1' '), 121.4 (Caf C-6' '), 118.1 (Agl C-6), 115.8 (Caf C-5' '), 115.4 (Agl C-5), 114.8 (Caf C-2' '), 114.0 (Agl C-2), 113.4 (Caf C-8' '), 109.1 (Api C-1''' '), 102.8 (Glc C-1'), 101.2 (Rha C-1'' '), 82.2 (Agl C-7), 78.9 (Glc C-3'), 78.8 (Api C-3''' '), 75.9 (Api C-2''' '), 74.3 (Glc C-2'), 73.7 (Agl C-8), 73.4 (Api C-4''' '), 72.7 (Glc C-5'), 71.6 (Rha C-4'' '), 70.5 (Rha C-5'' '), 70.4 (Rha C-3'' '), 69.3 (Glc C-4'), 68.7 (Rha C-5'' '), 67.0 (Glc C-6'), 63.2 (Api C-5''' '), 56.0 (7-OCH_3), 18.1 (Rha C-6'''). Analysis of the MS and NMR data revealed that compound **15** has a structure similar to that of forsythoside B (Wang et al., 2005). The main difference is the presence of an additional methoxy group signal [δH 3.13 (3H, d, J = 6.1 Hz,7-OCH_3), δC 56.0 (7-OCH_3)] and downfield-shifted methine

CH signals at C-7 due to the methoxy group: δC 73.4 (Agl C-8), 82.2 (Agl C-7), 129.7 (Agl C-1). In the HMBC spectrum, a long-range correlation was observed between δH 3.13 (3H, d, J = 6.1 Hz,7-OCH_3) and δC 82.2 (Agl C-7), confirming that the methoxy group is attached to the phenylethanol moiety at Agl C-7. Therefore, compound **15** was identified as β-methoxy forsythoside B.

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4. Discussion and Conclusion

This study employed multiple modern chromatographic separation techniques and spectroscopic identification methods to isolate and identify fifteen compounds from the root and stem extracts of the endemic Chinese plant *Callicarpa integerrima*. These compounds include three steroids (1, 2, 5), two triterpenoids (4, 6), six phenylethanoid glycosides (10–15), one flavonoid (3), two benzoic acid derivatives (7, 9), and one alkaloid (8).

The results indicate that phenylethanoid glycosides are characteristic constituents of *C. integerrima*. These compounds are natural water-soluble glycosides derived from phenylethanol aglycones substituted with caffeoyl, glucose/rhamnose/apiose, and other sugar moieties (Tian et al., 2020). Modern pharmacological studies have demonstrated that phenylethanoid glycosides exhibit diverse biological activities, including antioxidant, memory-enhancing, hepatoprotective, neuroprotective, and antitumor effects, with particularly promising potential for treating Alzheimer' s disease and improving cognitive function (Lee et al., 2006). *Callicarpa* plants have long been used as folk medicines for hemostasis. Yu et al. (2015) reported that phenylethanoid glycosides are the main active components responsible for the hemostatic effect of *C. kwangtungensis*. Most of the phenylethanoid glycosides identified in this study were isolated from *C. integerrima* for the first time, with compounds **11** and **14** being reported from the genus *Callicarpa* for the first time. Therefore, this study not only enriches the secondary metabolite library of *C. integerrima* but also provides a material basis for further exploration of novel hemostatic active constituents and their pharmacological mechanisms in this species.

The chemical constituents of the petroleum ether fraction from *C. integerrima* were found to be primarily low-polarity substances such as steroids and terpenoids. Previous studies have shown that α-amyrin (compound **4**) exhibits significant insecticidal activity against female *Sogatella furcifera*; β-sitosterol (compound **5**) demonstrates good larvicidal effects against *Aedes aegypti* and *Culex quinquefasciatus* mosquitoes (Chenniappan & Kadarkari, 2012; Gangadhara & Radhakrishnan, 2018) and inhibits the growth of *Aspergillus fumigatus*, *Aspergillus niger*, and *Rhizopus* species (Prince et al., 2019); ursolic acid (compound **6**) exerts antibacterial effects by reducing planktonic bacterial adhesion and disrupting biofilm formation (Sycz et al., 2022); and p-hydroxybenzoic acid (compound **9**) inhibits the growth of *Microcystis aeruginosa* by promoting reactive oxygen species production (Zhang et al., 2008). These findings suggest

that the lipophilic constituents of *C. integerrima* hold promise for plant disease control applications, providing a scientific basis for further development and utilization of this plant.

The *Callicarpa* species included in the 2020 edition of the Chinese Pharmacopoeia—*C. kwangtungensis*, *C. nudiflora*, *C. macrophylla*, and *C. formosana* —are all common arboreal or shrub species that typically grow in open, sunny environments such as roadsides and forest edges that are susceptible to external disturbance. In contrast, *C. integerrima*, as one of the few vine species in the genus, usually inhabits understory environments and relies on climbing other plants to obtain sunlight and growing space. Whether these specific understory environmental stresses affect the secondary metabolic activities of *C. integerrima* remains an open question. Notably, six of the fifteen compounds identified in this study represent first reports from the genus *Callicarpa*. These unexpected discoveries will motivate further investigation into the natural product constituents and pharmacological activities of *C. integerrima*.

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