



New phenylpropanoid derivatives from the fruits of *Xanthium sibiricum* and their anti-inflammatory activity



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ABSTRACT

The fruits of *Xanthium sibiricum* Patr yielded five phenylpropanoid derivatives, named as xanthiumnolics A–E (**1–5**). Their structures were elucidated by spectroscopic analysis and comparison with literature data. The isolated ones were tested for their anti-inflammatory activities on lipopolysaccharide (LPS)-induced nitric oxide (NO) production in RAW 264.7, and compound **5** showed strong inhibitory activities with IC₅₀ value of 8.73 μM.

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1. Introduction

The genus *Xanthium* belongs to the Compositae family is mainly distributed in the in North and Central America, Europe, Asia and north Africa [1]. In China, there are about 4 species in this genus and *Xanthium sibiricum* Patr. as one of them was found as Traditional Chinese Medicine for treating nasal sinusitis, numbness of limbs, arthritis, ulcer, pruritus, cancer, and herpes [2–6]. Previous chemical and pharmacological studies on the roots of this plant mainly resulted in the separation of sesquiterpene lactones, ent-kauranoid glycosides, and thiazines with broad bioactivities including antitumour, antibacterial, and inhibition of superoxide anion generation [7–13]. However, the chemistry of the fruits of *Xanthium sibiricum* Patr., which is defined as “*Fructus Xanthii*” has been little researched. In a continuation of our project towards the discovery of new bioactive constituents from *Fructus Xanthii*, the *n*-BuOH extraction of *Fructus Xanthii* was investigated. In this study, five new phenylpropanoid derivatives, xanthiumnolics A–E (**1–5**), were isolated and characterized (Fig. 1). Herein, the isolation and structural

elucidation of the new compounds as well as the evaluation of their anti-inflammatory activity are described.

2. Experimental

2.1. General experimental procedures

Optical rotations were obtained on a Perkin-Elmer 341 digital polarimeter. UV and IR spectra were recorded on Shimadzu UV2550 and FTIR-8400S spectrometers, respectively. CD spectra were recorded on a JASCO J-815 spectropolarimeter. NMR spectra were obtained with a Bruker AV III 400 NMR spectrometer (chemical shift values are presented as δ values with TMS as the internal standard). HRESIMS spectra were performed on a LTQ-Orbitrap XL spectrometer. Preparative HPLC was performed on a Lumtech K-1001 analytic LC equipped with two pumps of K-501, a UV detector of K-2600, and an YMC Pack C₁₈ column (250 mm × 10 mm, i.d., 5 μM, YMC Co. Ltd., Japan) eluted with CH₃OH–H₂O at a flow rate of 2 mL/min. C₁₈ reversed-phase silica gel (40–63 μM, Merk, Darmstadt, Germany), Sephadex LH-20 (Pharmacia, Uppsala, Sweden), MCI gel (CHP 20P, 75–150 μM, Mitsubishi Chemical Corporation, Tokyo, Japan) and silica gel (100–200 and 300–400 mesh, Qingdao Marine Chemical plant, Qingdao, China) were used for column chromatography. And pre-coated silica gel GF₂₅₄ plates (Zhi Fu Huang

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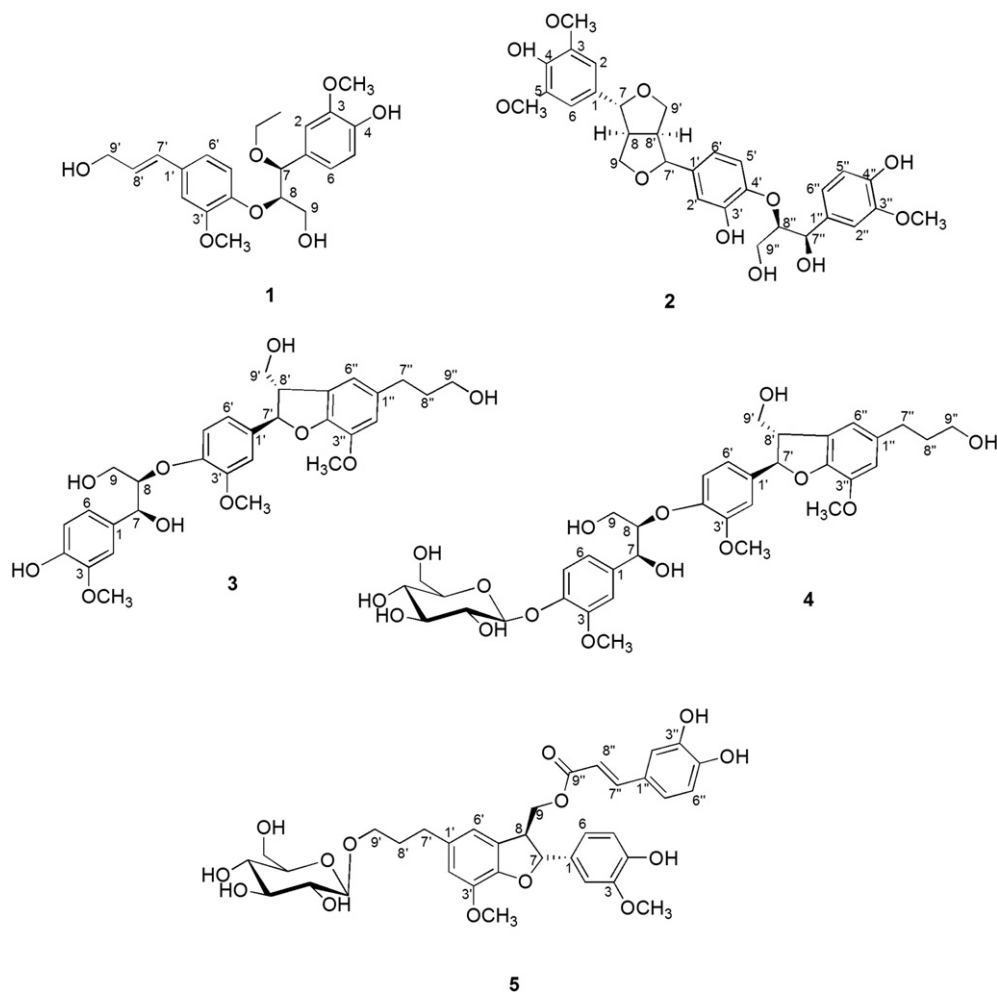


Fig. 1. Structures of compounds 1–5.

Wu Pilot Plant of Silica Gel Development, Yantai, China) were used for TLC. All solvents used were of analytical grade (Beijing Chemical Works).

2.2. Plant material

Fructus Xanthii was collected from Heilongjiang Province, China, in August 2011, and then authenticated by Prof. Wang Zhen-Yue, Heilongjiang University of Chinese Medicine, Harbin, China. A voucher specimen (No. 20111077) was deposited at the herbarium of Heilongjiang University of Chinese Medicine.

2.3. Extraction and isolation

A 70% aqueous EtOH extract of *Fructus Xanthii* (2.7 kg) was suspended to H₂O (3 L) and partitioned by EtOAc and n-BuOH. The n-BuOH extract (59.4 g) was subjected to silica gel chromatography using CH₂Cl₂–MeOH mixtures [20:1 (10 L), 10:1 (7 L), 5:1 (15 L), 2:1 (5 L) v/v] to afford fractions Fr. A–D. Fr. A was subjected to reverse phase MCI chromatography with a gradient of MeOH–H₂O system (40:60; 50:50; 60:40; 80:20; 100:0) as eluents, yielding five fractions (Fr. 1–5). Fr. 1–3 were subjected to Sephadex LH-20 to remove partial pigment and then isolated by HPLC on a YMC RP-18 column, respectively. Finally, compounds **2** (7.5 mg) and **3** (3.2 mg.) were obtained from fraction 1 using a (56:44) MeOH–H₂O system. Compound **1** (4.3 mg) was obtained from fraction 1 using a MeOH–H₂O (52:48) system. Compounds **4** (7.6 mg) and **5** (5.8 mg) were obtained from fraction 3 using a MeOH–H₂O (44:56) system.

2.3.1. Xanthiumnolic A (**1**)

C₂₂H₂₈O₇, pale yellow, viscous oil; $[\alpha]_D^{20} + 12.1$ (c 0.1, MeOH); UV (MeOH) λ_{\max} (log ϵ) 234 (4.22) nm; IR (film) ν_{\max} 3396 (OH), 1605, and 1464 (aromatic ring) cm⁻¹; ¹H NMR and ¹³C NMR see Table 1; HRESIMS m/z 427.1732 (calcd for C₂₂H₂₈O₇ Na [M + Na]⁺, 427.1726).

2.3.2. Xanthiumnolic B (**2**)

C₃₀H₃₄O₁₁, amorphous powder; $[\alpha]_D^{20} - 16.5$ (c 0.1, MeOH); UV (MeOH) λ_{\max} (log ϵ) 235 (3.79) nm; IR (film) ν_{\max} 3408 (OH), 1597, and 1517 (aromatic ring) cm⁻¹; ¹H NMR and ¹³C NMR see Table 1; HRESIMS m/z 571.2101 (calcd for C₃₀H₃₅O₁₁ [M + H]⁺, 571.2179).

2.3.3. Xanthiumnolic C (**3**)

C₃₀H₃₆O₁₀, amorphous powder; $[\alpha]_D^{20} - 37.5$ (c 0.1, MeOH); UV (MeOH) λ_{\max} (log ϵ) 341 (3.83) nm; IR (film) ν_{\max} 3530 (OH), 1590, and 1490 (aromatic ring) cm⁻¹; ¹H NMR and ¹³C NMR see Table 1; HRESIMS m/z 579.2308 (calcd for C₃₀H₃₆O₁₀Na [M + Na]⁺, 579.2206).

2.3.4. Xanthiumnolic D (**4**)

C₃₆H₄₆O₁₅, amorphous powder; $[\alpha]_D^{20} - 46.7$ (c 0.1, MeOH); UV (MeOH) λ_{\max} (log ϵ) 238 (4.02) nm; IR (film) ν_{\max} 3527 (OH), 1604, and 1592 (aromatic ring) cm⁻¹; ¹H NMR and ¹³C NMR see Table 2; HRESIMS m/z 719.2837 (calcd for C₃₆H₄₇O₁₅ [M + H]⁺, 719.2840).

2.3.5. Xanthiumnolic E (**5**)

C₃₅H₄₀O₁₄, amorphous powder; $[\alpha]_D^{20} - 19.7$ (c 0.1, MeOH); UV (MeOH) λ_{\max} (log ϵ) 243 (4.02) nm; IR (film) ν_{\max} 3436 (OH), 1617,

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