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Letter

Chemical Constituents from Stems of *Cistanches deserticola*

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ABSTRACT

Objective To study the chemical constituents in the stems of *Cistanches deserticola*. **Methods** The compounds were purified by various column chromatographic methods and their structures were elucidated by spectroscopic (1D, 2D NMR, IR, $[\alpha]_D$, and MS) and chemical analyses. **Results** Seven compounds were isolated and identified as kankanoside D_1 (**1**), ajugol (**2**), cistanin (**3**), uridine (**4**), adenosine (**5**), 6-deoxycatalpol (**6**), and kankanoside D (**7**). **Conclusion** Compound **1** is a new compound.

Key words

Cistanches deserticola Y. C. Ma; iridoids; monoterpenes; Orobanchaceae

1. Introduction

Plants in genus *Cistanche* Hoffmgg. et Link are commonly known as *Roucongrong* in Chinese due to their fleshy stem nature and the calm and leisurely effect in tonifying *Yang*. There are 22 species in genus *Cistanche* Hoffmgg. et Link including *C. deserticola* Y. C. Ma., which belongs to family Orobanchaceae. It is a holoparasitic plant which parasitizes on the roots of *Haloxylon ammodendron* (C. A. Mey.) Bunge or *H. persicum* Bunge ex Boiss. et Buhse (Luo et al, 2002). It is a well-known Chinese materia medica (CMM) for the treatment of kidney deficiency, female infertility, morbid leucorrhea, neurataxia, and senile constipation (Dictionary of Traditional Chinese Drugs, 1977; Chinese Medicinal Herbal, 1988; Chen et al, 2013).

Earlier phytochemical investigation conducted in the isolation of some polar and non-polar constituents. The non-polar constituents were mostly essential oils while those of polar constituents were phenylethanoid glycosides (PhGs), iridoids, lignans, alditols, oligosaccharides, and polysaccharides (Lei et al, 2003; Chen et al, 2013). Pharmacological studies on the plants of genus *Cistanche* Hoffmgg. et Link have shown a wide variety of applications such as treating kidney deficiency and constipation, treating Alzheimer's disease, boosting immune system, enhancing the ability to learn and memorize, anti-aging, and relieving stress (Song et al, 2003; Sato et al, 1985; Tian and Pu, 2005; Ebringerova et al, 2002). PhGs, regarded as the major constituents, have shown the functions such as neuroprotective, hepatoprotective, anti-oxidative, antibacterial, cytostatic, cardioactive, and sexual and immune system modulatory effects (Cheng et al, 1993; Saracoglu et al, 1995; Pennacchio et al, 1996; Xiong et al, 1996; Xiong et al, 1998).

In this paper, we report the isolation and structural elucidation of a new iridoid compound [Kankanoside D_1 (1)] together with six known compounds.

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2. Materials and methods

2.1 General

Optical rotations were measured on a Rudolph Autopol® IV automatic polarimeter (l = 50 mm). IR was recorded on a Varian 640-IR FT-IR spectrophotometer, and UV spectra on a Varian Cary 50 UV-Vis spectrophotometer. ^{1}H-NMR and ^{13}C-NMR spectra were determined on a Bruker 400 MHz NMR at 400 MHz for ^{1}H-NMR and 100 MHz for ^{13}C-NMR with tetra- methylsilane (TMS) as an internal standard. Positive and negative-ion HR-MS was recorded on an Agilent Technologies 6520 Accurate-Mass Q-Tof LC/MS spectrometer.

Silica gel column chromatography (CC) was obtained from Qingdao Haiyang Chemical Co., Ltd., (48−75 μm, Qingdao, China). Sephadex LH-20 was purchased from GE Healthcare Bio-Sciences Co., Ltd., Swiss. The preparative HPLC equipment is Agilent 1200 system including two G1361A preparative pumps, a manual sampler (7725i), a G1365D multiple wavelength detector (MWD), and Agilent LC workstation. HPLC was performed on ODS (Cosmosil C18-MS-II, Tokyo, Japan; 250 mm × 20 mm, flow rate: 10.0 mL/min). HPLC-ELSD (Alltech Grace Evaporative Light Scattering Detector 3300 with the following acquisition parameters Temp: 40 °C; Gas flow: 1.8 L/min; Gain: 10) was used to detect the purity of isolates.

2.2 Plant material

The air dried fleshy stems of *C. deserticola* were collected from Alashan, Inner Mongolia Autonomous Region, People′s Republic of China in June 2009. It was identified by Prof. Li-juan Zhang and a voucher specimen (No 20091001) was deposited in our laboratory.

2.3 Extraction and isolation

The air dried fleshy stems of *C. deserticola* (6.4 Kg) were refluxed two times with aqueous ethanol (60%) for 2 h each time. The combined extracts were concentrated under vacuum, suspended in water and then partitioned with chloroform ($CHCl_3$), ethyl acetate (EtOAc), and *n*-butanol (*n*-BuOH) successively to give three different polar parts. These three fractions were concentrated under vacuum to dryness. The EtOAc extract (11.9 g) was fractionated on an ODS (50 μm, YMC) CC using 30% and 35% methanol to give 13 fractions (A−M). Fractions F (197.6 mg), I (259.8 mg), and B (561.7 mg) were subjected to Sephadex LH-20 CC, and finally to preparative HPLC to afford compounds **2** (8.6 mg) and **3** (15.4 mg), respectively.

The *n*-BuOH extract (33.0 g) was fractionated by silica gel CC using a stepwise gradient of $CHCl_3$-MeOH (10:1 → 1:1 successively) to give eight fractions. Fraction 4 (3.8 g) was further isolated by ODS (50 μm, YMC) CC to yield eight fractions (4a - 4h). Fraction 4a (385.5 mg), 4b (52.1 mg), and 4c (89.3 mg) were further purified by preparative HPLC to yield compound **4** (23.8 mg). Fraction 4e (346.2 mg) was subjected to preparative HPLC to afford compounds **5** (6.8 mg), and **6** (66.9 mg). Compounds **7** (6.8 mg) and **1** (5.6 mg) were obtained from fraction 4f (397.5 mg). The structures of compounds **1**−**7** were showed in Figure 1.

2.4 Hydrolysis of compound 1

A solution of compound **1** (1.5 mg) in 1 mol/L HCl (1 mL) was refluxed for 3 h. After cooling, the reaction mixture was extracted with EtOAc. The aqueous layer was analyzed by HPLC under the following condition: column, Kaseisorb LC NH2-60-5 (250 mm × 4.6 mm, Tokyo Kasei Co. Ltd., Japan); optical rotation detector, Chiralyser (IBZ Messtechnik GMBH, Mozartstrasse 14-16 D-30173 Hannover, Germany); mobile phase, CH_3CN-H_2O (75: 25); flow rate, 1.0 mL/min. The identification of *D*-glucose presents in the aqueous layer was carried out by comparison of its retention time and optical rotation with that of *D*-glucose standard (t_R: 15.6 min, positive optical rotation).

Aglycone (*R*-rotundiol) of compound **1** was obtained by enzymatic hydrolysis with β-glucosidase. A solution of **1** (3.0 mg) in H_2O (1.0 mL) was treated with β-glucosidase (5.0 mg, from almonds, Sigma, USA) and the solution was stirred at 37 °C

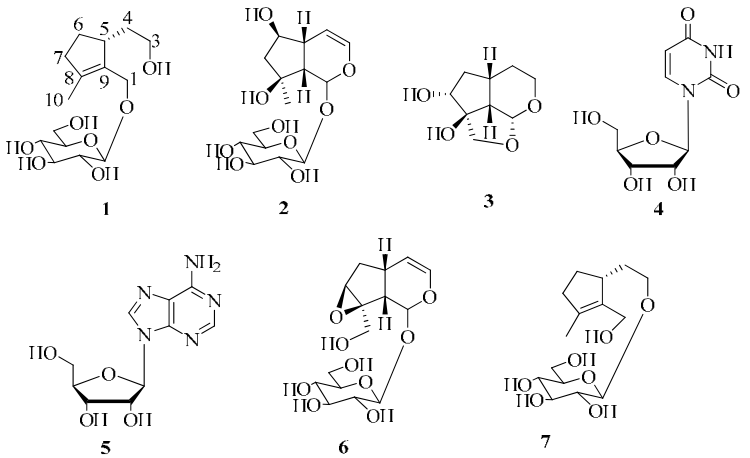

Figure 1 Chemical structures of compounds 1−7