

马槟榔果实的化学成分研究

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摘要: 采用硅胶柱色谱和 Sephadex LH-20 等分离方法, 对马槟榔 *Capparis masaikai* 果实的化学成分进行分离纯化, 依据理化性质及波谱数据分析进行结构鉴定, 从中分离鉴定了 10 个单体化合物, 分别为: 杜仲树脂酚 (1)、erythro-guaiacylglycerol- β -O-4'-sinapyl ether (2)、hedytol C (3)、hedytol A (4)、hedytol B (5)、ozoroalide (6)、5 α ,6 α -epoxy-3 β -hydroxyergosta-22-ene-7-one (7)、松柏醛 (8)、3-羟基-5-(对羟基苯基)戊酸 (9)、 β -hydroxypropiovanillone (10)。分离得到的化合物结构类型包括木脂素、大环内酯、甾醇及酚类。化合物 1~10 为首次从该植物中分离, 其中 1~7 为首次从该属植物中分离得到。

关键词: 马槟榔; 山柑属; 化学成分; 木脂素

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Chemical Constituents from the Fruits of *Capparis masaikai*

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Abstract: Ten compounds were isolated and purified by silica gel column chromatography and Sephadex LH-20 from the fruits of *Capparis masaikai*. Their structures were identified as medioresinol (1), erythro-guaiacylglycerol- β -O-4'-sinapyl ether (2), hedytol C (3), hedytol A (4), hedytol B (5), ozoroalide (6), 5 α ,6 α -epoxy-3 β -hydroxyergosta-22-ene-7-one (7), coniferyl aldehyde (8), 3-hydroxy-5-(*p*-hydroxyphenyl) pentanoic acid (9) and β -hydroxypropiovanillone (10) by physicochemical properties and spectral data. The structural types of isolated compounds include lignans, macrocyclic lactone, sterols and phenols. Compounds 1-10 were isolated from this plant for the first time, among of them, compounds 1-7 were firstly reported from the genus *Capparis*.

Key words: *Capparis masaikai*; *Capparis*; chemical constituents; lignans

Introduction

The genus *Capparis* comprises about 250 species in the world, which are distributed principally in tropical and subtropical regions^[1]. About 30 species of the genus are distributed in China, including Guangdong, Guangxi, Guizhou and Yunnan provinces^[2]. The fruits of

Capparis masaikai are used as a traditional folk Chinese medicine and its seeds are commonly chewed for their sweet taste, due to the presence of sweet proteins^[3]. In order to investigate the chemical constituents of this plant, ten compounds were isolated from 90% ethanol extract of *C. masaikai* and identified as medioresinol (1), erythro-guaiacylglycerol- β -O-4'-sinapyl ether (2), hedytol C (3), hedytol A (4), hedytol B (5), ozoroalide (6), 5 α ,6 α -epoxy-3 β -hydroxyergosta-22-ene-7-one (7), coniferyl aldehyde (8), 3-hydroxy-5-(*p*-hydroxyphenyl) pentanoic acid (9) and β -hydroxypropiovanillone (10). Compounds 1-10 were isolated from this plant material for the first time and compounds 1-7 were reported from genus

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Capparis for the first time.

Materials and Methods

General

NMR spectra were recorded on a Bruker AM-400, DRX-500, and Avance III 600 spectrometers with TMS as an internal standard. A Bruker HCT/E squire was used to measure ESI-MS spectra. Column chromatography was performed on silica gel (100-200, 200-300 and 300-400 mesh, Qingdao Marine Chemical Inc.), Sephadex LH-20 (40-70 μm , Amersham Pharmacia Biotech AB, Uppsala, Sweden), MCI gel CHP 20P (75-150 μm , Mitsubishi Chemical Corporation, Tokyo), and Chromatorex RP-C₁₈ gel (20-45 μm , Merck, Darmstadt, Germany). TLC spots were visualized under UV light and by dipping into 5% H₂SO₄ in EtOH followed by heating.

Plants materials

The fruits of *C. masaikai* were collected in Wenshan region of Yunnan Province, China, in September 2012, and identified by Prof. Hua Peng (Kunming Institute of Botany, Chinese Academy of Sciences). A voucher specimen (*kmust* 20120910) was deposited at Faculty of Life Science and Technology, Kunming University of Science and Technology.

Extraction and isolation

The air-dried and powdered fruits of *C. masaikai* (5.0 kg) were extracted by refluxing with 90% EtOH for three times. In a vacuum, the EtOH solvent was concentrated to give a crude residue (225.0 g), which was then suspended in water. The water layer was partitioned with EtOAc. The EtOAc portion (80.0 g) was submitted to column chromatography (CC) over silica gel (100-200 mesh) using PE (petroleum ether)/acetone (100:1 \rightarrow 6:4) and chloroform/MeOH (9:1 \rightarrow 0:1) to give seven fractions (Fr. A - Fr. G). Fr. C (9.87 g) was subjected to chromatography on a RP-C₁₈ (MeOH/H₂O from 6:4 \rightarrow 10:0) to give ten fractions (Fr. C1 - Fr. C10). Fr. C3 (0.35 g) was purified by Sephadex LH-20 (chloroform/MeOH) and was then chromatographed over repetitive silica gel eluted by gradient PE/acetone (50:1 \rightarrow 1:1) to yield compounds **6** (8.0 mg), **7** (14.0 mg), and **8** (2.0 mg).

Fr. C7 (0.85 g) was first subjected to an MCI gel column eluted with a gradient MeOH/H₂O (8:2 \rightarrow 10:0) to obtain three subfractions (C7A-C7C). Compounds **9** (20.0 mg) and **10** (15.0 mg) were obtained from Fr. C7B (0.25 g) by CC over silica gel and Sephadex LH-20 (acetone). Fr. E (10.15 g) was repeatedly chromatographed over a RP-C₁₈ (MeOH/H₂O from 3:7 \rightarrow 10:0), Sephadex LH-20 (MeOH), and silica gel columns to yield compounds **1** (66.0 mg), **2** (2.8 mg), **3** (4.0 mg), **4** (17.0 mg) and **5** (10.0 mg).

Structural identification

Compound 1 Yellow oil, C₂₁H₂₄O₇, ¹H NMR (CDCl₃, 400 MHz) δ : 6.89 (1H, d, J = 1.8 Hz, H-2), 6.87 (1H, d, J = 7.8 Hz, H-5), 6.82 (1H, dd, J = 7.8, 1.8 Hz, H-6), 6.58 (2H, s, H-2', 6'), 5.79 (1H, brs, OH-4), 5.65 (1H, brs, OH-4'), 4.74 (1H, d, J = 4.0 Hz, H-7'), 4.72 (1H, d, J = 4.0 Hz, H-7) 4.23-4.29 (2H, m, H-9a, 9'a), 3.90 (9H, s, OCH₃-3, 3', 5'), 3.89 (2H, m, H-9b, 9'b), 3.09 (2H, m, H-8, 8'); ¹³C NMR (CDCl₃, 100 MHz) δ : 147.1 (C-3, 5), 146.7 (C-3'), 145.2 (C-4'), 134.2 (C-4), 132.8 (C-1'), 132.0 (C-1), 118.9 (C-6'), 114.3 (C-5'), 108.6 (C-2'), 102.6 (C-2, 6), 86.1 (C-7), 85.8 (C-7'), 71.8 (C-9), 71.6 (C-9'), 56.3 (OCH₃-3', 5'), 55.9 (OCH₃-3), 54.3 (C-8), 54.0 (C-8'). It was identified as medioresinol by comparison of the physical and spectral data with the reported data^[4,5].

Compound 2 White powder, C₂₁H₂₆O₈, ESI-MS m/z : 429 [M + Na]⁺, ¹H NMR (CD₃OD, 600 MHz) δ : 6.98 (1H, d, J = 1.8 Hz, H-2), 6.78 (1H, dd, J = 7.8, 1.8 Hz, H-6), 6.75 (1H, d, J = 7.8 Hz, H-5), 6.74 (2H, s, H-2', 6'), 6.55 (1H, d, J = 16.2 Hz, H-7'), 6.32 (1H, dd, J = 16.2, 6.0 Hz, H-8'), 4.92 (1H, d, J = 4.8 Hz, H-7), 4.22 (2H, dd, J = 5.4, 1.2 Hz, H-9'a, 9'b), 4.21 (1H, m, H-8), 3.88 (1H, dd, J = 12.0, 5.4 Hz, H-9a), 3.82 (9H, s, OCH₃-3, 3', 5'), 3.55 (1H, dd, J = 12.0, 3.0 Hz, H-9b); ¹³C NMR (CD₃OD, 150 MHz) δ : 154.7 (C-3', 5'), 148.8 (C-4), 147.0 (C-3), 136.5 (C-4'), 134.9 (C-1'), 133.9 (C-1), 131.5 (C-7'), 130.0 (C-8'), 120.7

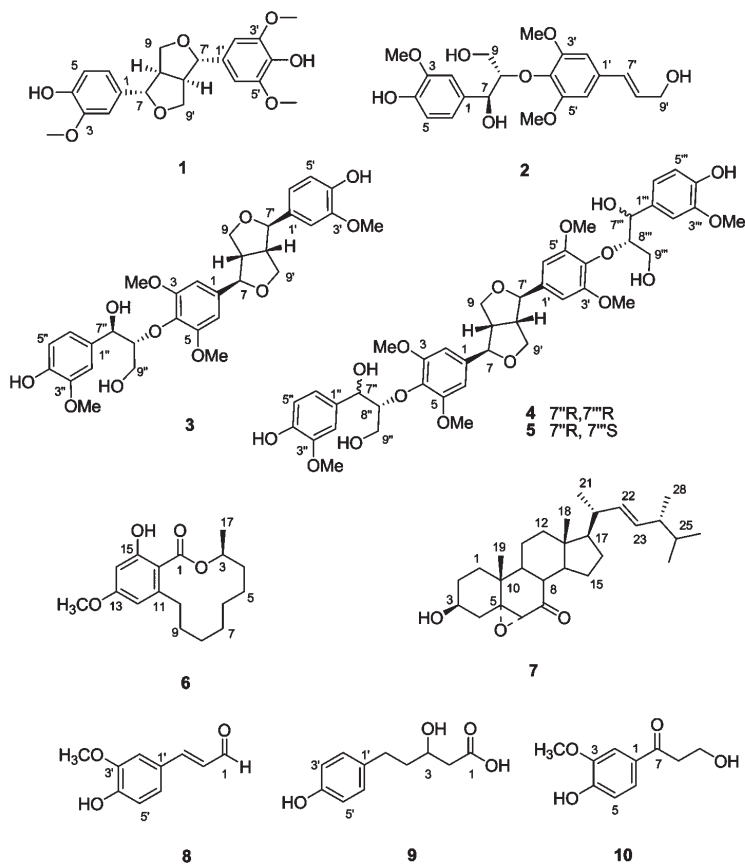


Fig. 1 Chemical structures of compounds 1-10

(C-6), 115.8 (C-5), 111.4 (C-2), 105.0 (C-2', 6'), 87.7 (C-8), 74.1 (C-7), 63.8 (C-9'), 61.6 (C-9), 56.7 (OCH₃-3, 3'), 56.4 (OCH₃-5'). It was identified as *erythro*-guaiacylglycerol- β -O-4'-sinapyl ether by comparison of the physical and spectral data with the reported data^[6].

Compound 3 White powder, C₃₁H₃₆O₁₁, ESI-MS m/z : 607 [M + Na]⁺, ¹H NMR (CD₃OD, 400 MHz) δ : 6.95 (2H, brs, H-2', 2''), 6.82 (1H, d, J = 8.0 Hz, H-5'), 6.81 (1H, d, J = 8.0 Hz, H-5''), 6.77 (1H, d, J = 8.0 Hz, H-6'), 6.73 (1H, d, J = 8.0 Hz, H-6''), 6.67 (2H, brs, H-2, 6), 4.75 (1H, d, J = 2.3 Hz, H-7''), 4.70 (2H, d, J = 3.8 Hz, H-7, 7'), 4.27 (1H, m, H-9a), 4.25 (1H, m, H-9'a), 4.11 (1H, m, H-8''), 3.91 (2H, m, H-9b, 9'b), 3.82 (6H, s, OCH₃-3, 5), 3.81 (6H, s, OCH₃-3', 3''), 3.87 (2H, m, H-9'a), 3.40 (2H, m, H-9'b), 3.13 (2H, m, H-8, 8'); ¹³C NMR (CD₃OD, 100 MHz) δ : 154.9 (C-3, 5), 149.5 (C-3''), 149.0 (C-3'), 147.7 (C-4'), 147.3 (C-4''), 139.3 (C-1), 136.5 (C-4), 134.2 (C-1'),

134.1 (C-1''), 121.1 (C-6'), 120.5 (C-6''), 116.5 (C-5'), 116.0 (C-5''), 111.7 (C-2'), 111.4 (C-2''), 104.6 (C-2, 6), 87.9 (C-8''), 87.7 (C-7), 87.6 (C-7'), 74.4 (C-7''), 73.3 (C-9), 73.1 (C-9'), 62.1 (C-9''), 57.1 (OCH₃-3, 5), 56.8 (OCH₃-3'), 56.7 (OCH₃-3''), 56.2 (C-8'), 55.9 (C-8). It was identified as hedytol C by comparison of the physical and spectral data with the reported data^[7-8].

Compound 4 White powder, C₄₂H₅₀O₁₆, ESI-MS m/z : 833 [M + Na]⁺, ¹H NMR (CDCl₃, 400 MHz) δ : 6.95 (2H, brs, H-2'', 2'''), 6.87 (2H, dd, J = 8.1, 1.6 Hz, H-6'', 6'''), 6.72 (2H, d, J = 8.1 Hz, H-5'', 5'''), 6.63 (4H, brs, H-2, 6, 2', 6'), 4.99 (2H, m, H-7'', 7'''), 4.78 (2H, d, J = 3.0 Hz, H-7, 7'), 4.27 (2H, m, H-9a, 9'a), 4.11 (2H, m, H-8'', 8'''), 3.91 (2H, m, H-9b, 9'b), 3.88 (6H, s, OCH₃-3', 5'), 3.87 (6H, s, OCH₃-3, 5), 3.81 (6H, s, OCH₃-3'', 3'''), 3.81 (2H, m, H-9'a, 9''a), 3.40 (2H, m, H-9'b, 9''b), 3.13 (2H, m, H-8, 8'); ¹³C NMR (CDCl₃, 100 MHz) δ : 153.4 (C-3, 5, 3', 5'), 146.6 (C-3'', 3'''),

144.8 (C-4'', 4'''), 137.5 (C-1, 1'), 134.2 (C-4, 4'), 131.2 (C-1'', 1'''), 118.7 (C-6'', 6'''), 114.2 (C-5'', 5'''), 108.3 (C-2'', 2'''), 102.8 (C-2, 6, 2', 6'), 87.0 (C-8'', 8'''), 85.8 (C-7, 7'), 72.4 (C-7'', 7'''), 72.0 (C-9, 9'), 60.5 (C-9'', 9'''), 56.1 (OCH₃-3, 5, 3', 5'), 54.4 (OCH₃-3'', 3'''), 54.4 (C-8, 8'). It was identified as hedyotisol A^by comparison of the physical and spectral data with the reported data^[8-10].

Compound 5 White powder, C₄₂H₅₀O₁₆, ESI-MS *m/z*: 833 [M + Na]⁺, ¹H NMR (CDCl₃, 400 MHz) δ: 6.95 (2H, brs, H-2'', 2'''), 6.87 (2H, d, *J* = 8.1 Hz, H-6'', 6'''), 6.72 (2H, d, *J* = 8.1 Hz, H-5'', 5'''), 6.62 (4H, brs, H-2, 6, 2', 6'), 5.00 (2H, m, H-7'', 7'''), 4.77 (2H, d, *J* = 3.0 Hz, H-7, 7'), 4.33 (2H, m, H-9a, 9'a), 4.11 (2H, m, H-8'', 8'''), 3.96 (1H, m, H-9b), 3.94 (1H, m, H-9b'), 3.91 (6H, s, OCH₃-3', 5'), 3.88 (6H, s, OCH₃-3, 5), 3.82 (3H, s, OCH₃-3''), 3.81 (3H, s, OCH₃-3'''), 3.81 (1H, m, H-9''a), 3.54 (1H, m, H-9''''a), 3.47 (1H, m, H-9''b), 3.33 (1H, m, H-9''''b), 3.10 (2H, m, H-8, 8'); ¹³C NMR (CDCl₃, 100 MHz) δ: 153.4 (C-3, 5), 153.1 (C-3', 5'), 146.6 (C-3'''), 146.5 (C-3''), 145.4 (C-4'''), 144.8 (C-4''), 137.6 (C-1'), 137.5 (C-1), 134.6 (C-4), 134.4 (C-4'), 131.3 (C-1'', 1'''), 120.3 (C-6'''), 118.7 (C-6''), 114.2 (C-5'', 5'''), 109.8 (C-2'''), 108.3 (C-2''), 102.7 (C-2, 6), 106.6 (C-2', 6'), 89.0 (C-8'''), 87.0 (C-8''), 85.8 (C-7'), 85.7 (C-7), 74.0 (C-7'''), 72.3 (C-7''), 72.0 (C-9, 9'), 60.5 (C-9'', 9'''), 56.1 (OCH₃-3, 5, 3', 5'), 54.4 (OCH₃-3'', 3'''), 54.4 (C-8, 8'). It was identified as hedyotisol B by comparison of the physical and spectral data with the reported data^[8,9].

Compound 6 White needles, C₁₇H₂₄O₄, ESI-MS *m/z*: 315 [M + Na]⁺, ¹H NMR (CD₃OD, 400 MHz) δ: 6.28 (1H, d, *J* = 2.0 Hz, H-12), 6.23 (1H, d, *J* = 2.0 Hz, H-14), 5.16 (1H, m, H-3), 3.75 (3H, s, OCH₃), 2.65 (1H, ddd, *J* = 13.4, 7.8, 7.8 Hz, H-10a), 2.49 (1H, ddd, *J* = 13.4, 7.8, 7.8 Hz, H-10b); ¹³C NMR (CD₃OD, 100 MHz) δ: 170.1 (C-1), 160.7 (C-13), 159.4 (C-15), 143.8 (C-11), 117.6 (C-16), 109.2 (C-12), 97.8 (C-14), 73.4 (C-3), 56.2 (OCH₃), 33.6 (C-4), 31.3 (C-10),

31.1 (C-9), 27.7 (C-8), 26.3 (C-7), 25.6 (C-6), 22.3 (C-5), 19.9 (C-17). It was identified as ozorolide by comparison of the physical and spectral data with the reported data^[11].

Compound 7 White needles, C₂₈H₄₄O₃, ¹H NMR (CDCl₃, 400 MHz) δ: 5.19 (1H, m, H-23), 5.13 (1H, m, H-22), 3.90 (1H, m, H-3), 3.05 (1H, s, H-6), 1.02 (3H, s, H-19), 0.98 (3H, d, *J* = 6.6 Hz, H-21), 0.89 (3H, d, *J* = 6.8 Hz, H-28), 0.82 (3H, d, *J* = 6.6 Hz, H-27), 0.80 (3H, d, *J* = 6.6 Hz, H-26), 0.67 (3H, s, H-18); ¹³C NMR (CDCl₃, 100 MHz) δ: 207.7 (C-7), 135.4 (C-22), 132.0 (C-23), 68.7 (C-3), 68.1 (C-5), 63.0 (C-6), 55.1 (C-17), 52.0 (C-14), 46.7 (C-8), 43.9 (C-13), 43.3 (C-9), 42.8 (C-24), 40.1 (C-20), 39.5 (C-12), 38.7 (C-4), 35.0 (C-10), 33.0 (C-25), 32.8 (C-1), 30.8 (C-2), 28.4 (C-16), 24.8 (C-11), 21.1 (C-15), 21.0 (C-21), 19.9 (C-27), 19.6 (C-26), 17.6 (C-28), 15.5 (C-19), 12.3 (C-18). It was identified as 5α, 6α-epoxy-3β-hydroxyergosta-22-ene-7-one by comparison of the physical and spectral data with the reported data^[12].

Compound 8 Yellow needles, C₁₀H₁₀O₃, ¹H NMR (CD₃OD, 600 MHz) δ: 9.57 (1H, d, *J* = 7.8 Hz, H-1), 7.59 (1H, d, *J* = 15.6 Hz, H-3), 7.26 (1H, d, *J* = 1.8 Hz, H-2'), 7.17 (1H, dd, *J* = 8.4, 1.8 Hz, H-6'), 6.85 (1H, d, *J* = 8.4 Hz, H-5'), 6.66 (1H, dd, *J* = 15.6, 7.8 Hz, H-2), 3.91 (3H, s, OCH₃); ¹³C NMR (CD₃OD, 150 MHz) δ: 196.4 (CHO-1), 156.5 (C-3), 151.9 (C-4'), 149.6 (C-3'), 127.0 (C-1'), 126.8 (C-2), 125.3 (C-6'), 116.5 (C-5'), 111.9 (C-2''), 56.5 (OCH₃). It was identified as coniferyl aldehyde by comparison of the physical and spectral data with the reported data^[13].

Compound 9 Colorless gum, C₁₁H₁₄O₄, ¹H NMR (CD₃OD, 500 MHz) δ: 6.98 (2H, d, *J* = 8.4 Hz, H-2', 6'), 6.66 (2H, d, *J* = 8.4 Hz, H-3', 5'), 4.97 (1H, brs, OH-4'), 3.96 (1H, m, H-3), 2.63 (2H, m, H-5), 2.53 (2H, m, H-2), 1.70 (2H, m, H-4); ¹³C NMR (CD₃OD, 125 MHz) δ: 175.7 (C-1), 156.4 (C-4'), 134.2 (C-1'), 130.4 (C-2', 6'), 116.2 (C-3', 5'), 68.8 (C-3), 43.3 (C-4), 40.3 (C-2), 32.0 (C-5). It was identified as 3-hydroxy-5-(p-hydroxy-

phenyl) pentanoic acid by comparison of the physical and spectral data with the reported data^[14].

Compound 10 Colorless gum, C₁₀H₁₂O₄, ¹H NMR (CD₃OD, 500 MHz) δ: 7.56 (1H, dd, J = 8.0, 1.4 Hz, H-6), 7.53 (1H, d, J = 1.4 Hz, H-2), 6.84 (1H, d, J = 8.0, H-5), 3.92 (2H, m, H-9), 3.89 (3H, s, OCH₃-3), 3.14 (2H, m, H-9); ¹³C NMR (CD₃OD, 125 MHz) δ: 199.6 (C-7), 153.3 (C-4), 149.0 (C-3), 130.6 (C-1), 124.7 (C-6), 115.8 (C-2), 111.8 (C-5), 58.9 (C-9), 56.3 (OCH₃-3), 41.6 (C-8). It was identified as β-hydroxypropiovanillone by comparison of the physical and spectral data with the reported data^[15,16].

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