

PHENOLIC COMPOUNDS FROM *CALLISTEMON CITRINUS* LEAVES AND STEMS

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ABSTRACT

In the search for bioactive constituents from Vietnamese plants, the leaves and stems of *Callistemon citrinus* (Curtis) Skeels were selected for chemical investigation. Phytochemical analysis of plant led to the isolation of eight phenolic compounds including two flavonoids (eucalyptine (**1**) and 8-demethyleucalyptine (**2**)), two alcohols (blumenol A (**3**), *n*-tetratriacontanol (**4**)), three benzoic acid derivatives (acid gallic (**5**), methyl gallate (**6**), protocatechuic acid (**7**)), one sterol (β -sitosterol (**8**)), and along with one sesquiterpene (2,6,10-bisabolatriene (**9**)). The structures of the natural compounds were determined from the spectroscopic evidences including 1D- and 2D-NMR and ESI-MS.

Keywords: *Callistemon citrinus* (Curtis) Skeels, flavonoid, triterpenoid.

1. INTRODUCTION

The Myrtaceae family in Vietnam comprises about 30 genera, several of which are very common and widely distributed throughout the country such as *Eucalyptus*, *Rhodomyrtus*, *Syzygium*, and *Callistemon* [1]. Species *Callistemon citrinus* (Curtis) Skeels, local name “Tram bong do”, is grown as ornaments in Vietnam for its beautiful form, glossy green foliage and red, bottle-brush like flowers. Phytochemical studies of *Callistemon* species have led to the isolation

and characterization of flavones [2], acylphloroglucinols [3, 4], triterpenoids [5], neolignans [6], essential oil, steroids and saponins [7].

In previous papers, we reported the isolation of acylphloroglucinol derivatives and triterpenoids with soluble epoxide hydrolase inhibitory activity [8] as well as flavonoids with inhibitory effect on NO production in LPS-activated RAW264.7 macrophage [9] from *Callistemon citrinus* leaves and stems. In this study, we describe the isolation and structural elucidation of eight phenolic compounds, including two flavones (eucalyptin (**1**) and 8-demethyleucalyptin (**2**)), two alcohols (blumenol A (**3**), tetratriacontan-1-ol (**4**)), three benzoic acid derivatives (gallic acid (**5**), methyl gallate (**6**) and protocatechuic acid (**7**)), one sterol (β -stigmasterol (**8**)) along with one sesquiterpene (2,6,10-bisabolatriene (**9**)). The structures of the natural compounds were identified by spectroscopic evidences including 1D- and 2D-NMR and ESI-MS.

2. MATERIALS AND METHODS

2.1. General experimental procedures

¹H-NMR (500 MHz), ¹³C NMR (125 MHz) spectra were measured on a Bruker AVANCE 500 spectrometer. The ESI-MS spectra were obtained with a ESI-MicroQ-TOF III (Bruker Daltonics Inc.) and a FT-ESI-MS (Varian Inc.) mass spectrometer. UV and IR spectra were obtained on a JASCO V-630 and an Impact 410 Nicolet FT-IR spectrometer, respectively. Column chromatography (CC) was carried out on silica gel (Si 60 F₂₅₄, 230-400 mesh, Merck). All solvents were distilled before use. Precoated plates of silica gel 60 F₂₅₄ were used for analytical purposes. Compounds were visualized under UV radiation (254, 365 nm) and by spraying plates with 10% H₂SO₄ followed by heating with a heat gun.

2.2. Plant material

The leaves and stems of *Callistemon citrinus* (Curtis) Skeels were collected in Hue province, Vietnam. The plants were identified by the botanist Dr. Tran The Bach (Institute of Ecology and Biological Resources, VAST). A voucher specimen (HCTN-2118) is deposited in the herbarium of the Institute of Natural Products Chemistry, VAST, Hanoi, Vietnam.

2.3. Extraction and isolation

Dried powdered leaves and stems of *C. citrinus* (3.2 kg) were extracted with MeOH over the period of 5 days at room temperature and concentrated under reduced pressure to yield a black crude MeOH extract (190 g). This crude MeOH extract was suspended in hot MeOH-water (1:1, v/v) and successively partitioned with *n*-hexane, dichloromethane (DCM), ethyl acetate (EtOAc) and water. The resulting fractions were concentrated under reduced pressure to give the corresponding solvent-soluble fractions *n*-hexane (27.3 g), DCM (63.0 g), EtOAc (55.4 g), and water.

The *n*-hexane fraction (27.0 g) was chromatographed on a silica gel column, using solvent gradients of *n*-hexane – EtOAc (1:0, 40:1, 20:1, 10:1, and 5:1, v/v, 1.0 L each) to afford 5 subfractions (H1 to H5). The sub-fraction H4 was rechromatographed on silica gel column, eluting with DCM – EtOAc (20:1, v/v) to yield compound **1** (11.0 mg) and sub-fraction H4A, which was further purified on a silica gel column eluting with CHCl₃–EtOAc (15/1, v/v) to get

compound **9** (5.6 mg). A precipitate from the sub-fraction H3 was filtered and washed by *n*-hexane (1 mL × 2) to yield **4** (12.8 mg). The residue was separated by CC on silica gel, eluting with acetone–MeOH (1/4, v/v) to give compound **7** (15.7 mg) and **8** (7.2 mg).

The DCM fraction (63.0 g) was subjected to chromatography on a flash silica gel column (400 – 630 mesh), eluted with gradient DCM – methanol (1:0, 40:1, 20:1, 10:1, 5:1, 2.5:1, 1:1 and 0:1, v/v, 1.5 L each) to afford 6 subfractions (Fr. D1 to D6). The subfraction D1 (10.2 g) was subjected to silica gel CC, eluted with an isocratic solvent mixture of *n*-hexane-DCM-acetone (1:2:0.1, v/v/v), to afford 12 subfractions (D1A to D1L). The subfraction D1C was eluted with isocratic solvent system of *n*-hexane – DCM (1:3, v/v) on a silica gel column (230 – 400 mesh) to yield compound **2** (6.1 mg).

The EtOAc fraction (55.4 g) was chromatographed on a flash silica gel column (400 – 630 mesh) eluting with gradients of CH₂Cl₂-MeOH (1:0 ~ 0:1, v/v) to afford 7 subfractions (E1~E7). The subfraction E3 was subjected to column chromatography on silica gel eluting with CH₂Cl₂-MeOH (15:1) to afford 2 subfractions E3A and E3B. The subfraction E3B was further separated on a silica gel column eluting with a mixture of *n*-hexane - Me₂CO (2:1), to yield compounds **6** (99.0 mg) and **3** (4.9 mg). The subfraction E4 was subjected to column chromatography on silica gel eluting with gradients of CHCl₃-MeOH-H₂O (4:1:0.1 – 3:1:0.1, v/v/v) to obtain 6 subfractions (E4A - E4F). The subfraction E4C was subjected to CC on silica gel eluting with an isocratic mixture of Me₂CO-CHCl₃-H₂O (2:1:0.1, v/v/v), to afford 6 subfractions (E4C1~E4C6). The subfraction E4C1 was chromatographed over a RP-18 column, eluting with MeOH-H₂O (1:1, v/v) to obtain 4 subfractions (C1A~C1D). The subfraction C1A was rechromatographed over a RP-18 column eluting with MeOH-H₂O (1:3) to yield compounds **5** (123.0 mg).

2.4. Spectral and physical data

2.4.1 Eucalyptin (1) pale lemon yellow powder, C₁₉H₁₈O₅ (M=326). ¹H-NMR (500 MHz, CDCl₃), δ(ppm): 6.60 (s, 1H, H-3), 7.85 (d, 2H, *J* = 9.5 Hz, H-2',6'), 7.01 (d, 2H, *J* = 9.5 Hz, H-3',5'), 12.87 (s, 5-OH), 3.80 (s, 3H, 7-OCH₃), 3.89 (s, 3H, 4'-OCH₃), 2.20 (s, 3H, 8-CH₃), 2.38 (s, 3H, 6-CH₃). ¹³C-NMR (125 MHz, CDCl₃), δ(ppm): 163.8 (s, C-2), 104.0 (d, C-3), 183.2 (s, C-4), 157.3 (s, C-5), 114.1 (s, C-6), 162.6 (s, C-7), 107.4 (s, C-8), 152.9 (s, C-9), 108.8 (s, C-10), 123.8 (s, C-11), 127.9 (d, C-2',6'), 114.5 (d, C-3',5'), 159.8 (s, C-4'), 60.4 (q, 7-OCH₃), 55.5 (q, 4'-OCH₃), 8.5 (q, 8-CH₃), 8.3 (q, 6-CH₃). ESI-MS (*m/z*): 327 [M+H]⁺.

2.4.2 8-demethyleucalyptin (2) white powder, C₁₈H₁₆O₅ (M=312). ¹H-NMR (500 MHz, DMSO-*d*₆), δ(ppm): 6.86 (s, 1H, H-3), 6.92 (s, 1H, H-8), 8.05 (d, 2H, *J* = 9.5 Hz, H-2',6'), 7.10 (d, 2H, *J* = 9.0 Hz, H-3',5'), 13.05 (s, 5-OH), 3.86 (s, 3H, 7-OCH₃), 3.91 (s, 3H, 4'-OCH₃), 1.99 (s, 3H, 6-CH₃). ¹³C-NMR (125 MHz, DMSO-*d*₆), δ(ppm): 163.0 (s, C-2), 103.7 (d, C-3), 181.9 (s, C-4), 162.3 (s, C-5), 107.5 (s, C-6), 163.3 (s, C-7), 90.3 (s, C-8), 155.4 (s, C-9), 104.4 (s, C-10), 122.8 (s, C-1'), 128.2 (d, C-2', 6'), 114.5 (d, C-3', 5'), 157.4 (s, C-4'), 56.3 (q, 7-OCH₃), 55.5 (q, 4'-OCH₃), 7.2 (q, 6-CH₃). ESI-MS (*m/z*): 313 [M+H]⁺.

2.4.3 Blumenol A (3) white powder, C₁₃H₂₀O₃ (M = 224). ¹H-NMR (500 MHz, MeOD-*d*₄), δ(ppm): 2.50 (1H, d, *J* = 17.0 Hz, H-6a), 2.19 (1H, dd, *J* = 6.5, 17.0 Hz, H-6b), 5.90 (1H, m, *J* = 1.5 Hz, H-2), 5.80 (1H, m, *J* = 16.0 Hz, H-7), 5.82 (1H, m, *J* = 16.0, 4.5 Hz, H-8), 4.34 (1H, m, *J* = 4.5; 6.5 Hz, H-9), 1.26 (3H, d, *J* = 6.5 Hz, H-10), 1.04 (3H, s, H-11), 1.06 (3H, s, H-12), 1.93 (3H, d, *J* = 1.5 Hz, H-13). ¹³C-NMR (125 MHz, MeOD-*d*₄), δ(ppm): 201.2 (s, C-1),

42.4 (s, C-5), 50.7 (t, C-6), 127.1 (s, C-2), 167.4 (s, C-3), 79.9 (s, C-4), 129.9 (d, C-7), 136.9 (d, C-8), 68.6 (d, C-9), 23.8 (q, C-10), 24.5 (q, C-11), 23.4 (q, C-12), 19.5 (q, C-13).

2.4.4 *Tetratriacontan-1-ol* (**4**) white powder, C₃₄H₇₀O (M=494). ¹H-NMR (500 MHz, CDCl₃): 3.64 (2H, t, *J* = 6.5 Hz, H-1), 1.55 (4H, m, H-2&3), 1.25-1.34 (60H, H-4÷H-33), 0.88 (3H, t, *J* = 6.5 Hz, H-34); ESI-MS (*m/z*): 493 [M-H]⁻.

2.4.5. *Gallic acid* (**5**) white powder, C₇H₆O₅ (M=170). ¹H-NMR (500 MHz, MeOD-*d*₄), δ (ppm): 7.00 (2H, s, H-2, -6). ¹³C-NMR (125 MHz, MeOD-*d*₄), δ (ppm): 170.5 (s, C-7), 110.4 (d, C-2, -6), 146.5 (s, C-3, -5), 139.7 (s, C-4), 122.0 (s, C-1).

2.4.6. *Methyl gallate* (**6**) white powder, C₈H₈O₅ (M=184). ¹H-NMR (500 MHz, MeOD-*d*₄), δ (ppm): 7.00 (2H, s, H-2, -6), 3.77 (3H, s, H-8). ¹³C-NMR: (500 MHz, MeOD-*d*₄), δ (ppm): 169.1 (s, C-7), 52.3 (q, C-8), 110.1 (d, C-2, -6), 146.6 (s, C-3, -5), 139.8 (s, C-4), 121.5 (s, C-1).

2.4.7. *Protocatechuic acid* (**7**) white powder, C₇H₆O₄, (M=154). ¹H-NMR (500 MHz, MeOD-*d*₄), δ (ppm): 7.93 (COOH), 7.45 (1H, s, H-2), 6.81 (1H, d, *J* = 8.0 Hz, H-5), 7.44 (1H, d, *J* = 8.0 Hz, H-6). ¹³C-NMR (125 MHz, MeOD-*d*₄), δ (ppm): 170.4 (s, C-7), 115.8 (d, C-6), 117.8 (d, C-5), 123.4 (s, C-4), 146.1 (s, C-3), 123.9 (d, C-2), 151.5 (s, C-1).

2.4.8. β -*sitosterol* (**8**) white powder, C₂₉H₅₀O (M=414). ¹H-NMR (500 MHz, CDCl₃), δ (ppm): 3.52 (1H, tt, *J* = 6.5, 11.0 Hz, H-3), 5.35 (1H, brd, *J* = 6.5 Hz, H-6), 0.68 (3H, s, H-18), 1.01 (3H, s, H-19), 0.90 (3H, d, *J* = 7.0 Hz, H-21), 0.83 (3H, d, *J* = 6.5 Hz, H-26), 0.81 (3H, d, *J* = 7.0 Hz, H-28), 0.85 (3H, d, *J* = 7.0 Hz, H-29).

2.4.9. *2,6,10-bisabolatriene* (**9**) colourless oil, C₁₅H₂₄ (M=204). ¹H-NMR (500 MHz, CDCl₃), δ (ppm): 2.01 (2H, quint. *J* = 7.5 Hz, H-1); 5.10 (1H, H-2); 2.07 (2H, quint., *J* = 7.5 Hz, H-4); 2.07 (2H, quint., *J* = 7.5 Hz, H-5); 1.99 (2H, quint., *J* = 8.0 Hz, H-8); 1.99 (2H, quint., *J* = 8.0 Hz, H-9); 5.10 (1H, H-10); 1.60 (3H, s, H-12); 1.60 (3H, s, H-13); 1.60 (3H, s, H-14); 1.68 (3H, s, H-15). ¹³C-NMR (125 MHz, CDCl₃), δ (ppm): 26.7 (t, C-1); 124.3 (d, C-2); 135.1 (s, C-3); 28.3 (t, C-4); 26.8 (t, C-5); 124.2 (s, C-6); 134.9 (s, C-7); 39.8 (t, C-8); 39.7 (t, C-9); 124.4 (d, C-10); 131.2 (s, C-11); 16.1 (q, C-12); 16.0 (q, C-13); 25.7 (q, C-14); 17.7 (q, C-15); ESI-MS (*m/z*): *m/z* 205 [M+H]⁺.

3. RESULTS AND DISCUSSION

Compound (**2**) was isolated from the dichloromethane (DCM) fraction of the methanolic extract of *Callistemon citrinus* leaves and stems. Three compounds (**3**, **5** and **6**) were isolated from the EtOAc fraction and five compounds (**1**, **4**, **7**, **8** and **9**) were obtained from the *n*-hexane fraction.

Compounds **1** and **2** are two flavones (Fig. 1). While compound **1** was obtained from *n*-hexane fraction, compound **2** was isolated from EtOAc fraction of the methanolic extract of *C. citrinus* leaves. The 1D-NMR spectra including ¹H, ¹³C and DEPT of two compounds are very similar. The ¹H spectrum of **1** showed a low-field singlet signal (δ_{H} 12.87, 5-OH), two doublet aromatic proton signals characteristic of a *para*-substituted B-ring, two methoxy groups (δ_{H} 3.89, and 3.80) and two singlet methyl substituents (δ_{H} 2.38 and 2.20). ¹³C-NMR analysis of **1** revealed the presence of 19 carbons including one carbonyl (δ_{C} 183.2), four methines of a *para*-

substituted B-ring, one A-ring aromatic methine, two methoxyl, and two C-methyl carbons. Molecular formula of **1** was determined to be $C_{19}H_{18}O_5$ based on the quasi – molecular ion peak observed at m/z 327 $[M+H]^+$ in positive mode electrospray ionization (ESI-MS) mass spectrometry. From the 1D-NMR and ESI-MS spectral data, compound **1** was identified as 5-hydroxy-7,4'-dimethoxy-6,8-dimethylflavone with common name as eucalyptin [10]. Similarly, molecular formula of **2** was $C_{18}H_{16}O_5$, based on the ESI-MS quasi – molecular ion peak at m/z 313 $[M+H]^+$, indicating the absence of one methyl group in comparison with that of eucalyptin (**1**). In the NMR spectra of **2**, the absence of one C-methyl signal, resulting in the shift of C-8 signal to the upfield region (δ_C 90.3 in **2** vs. 107.4 in **1**), and the addition of one proton in the aromatic field (δ_H 6.92, H-8), as well as by comparison with spectral data reported in the literature, confirmed the structure of compound **2** as 5-hydroxy-7,4'-dimethoxy-6-methylflavone or 8-demethyleucalyptin [10].

Compound **3** was isolated as white powder from the EtOAc fraction. The 1H -NMR spectrum of **3** showed characteristic signals of four methyl groups at δ_H 1.26 (3H, d, $J = 6.5$ Hz, H-10), 1.04 (3H, s, H-11), 1.06 (3H, s, H-12), and 1.93 (3H, d, $J=1.5$ Hz, H-13); one methylene at δ_H 2.50 (1H, d, $J=17.0$ Hz, H-6a), 2.19 (1H, d, $J = 17.0$; 6.5 Hz, H-6b) and four methines at δ_H 5.90 (1H, m, H-2), 5.80 (1H, d, $J=16.0$ Hz, H-7), 5.82 (1H, dd, $J = 16.0$, 4.5 Hz, H-8), 4.34 (1H, m, $J = 6.5$, 4.5 Hz, H-9). The strong coupling constant ($J = 16.0$ Hz) of protons H-7 and H-8 indicated the *trans*-configuration of $C_7=C_8$ double bond. ^{13}C -NMR/DEPT spectra of **3** showed 13 carbons (1 x CO, 3 x C_4 , 4 x CH, 4 x CH_3 and 1 x CH_2). The analysis of HMBC spectrum indicated the presence of a 3,5,5-trimethyl-2-cyclohexene-1-one unit and a 3-hydroxy-1-butenyl side chain. Comparison of the spectral data with those in the literature, compound **3** was determined as blumenol A [11].

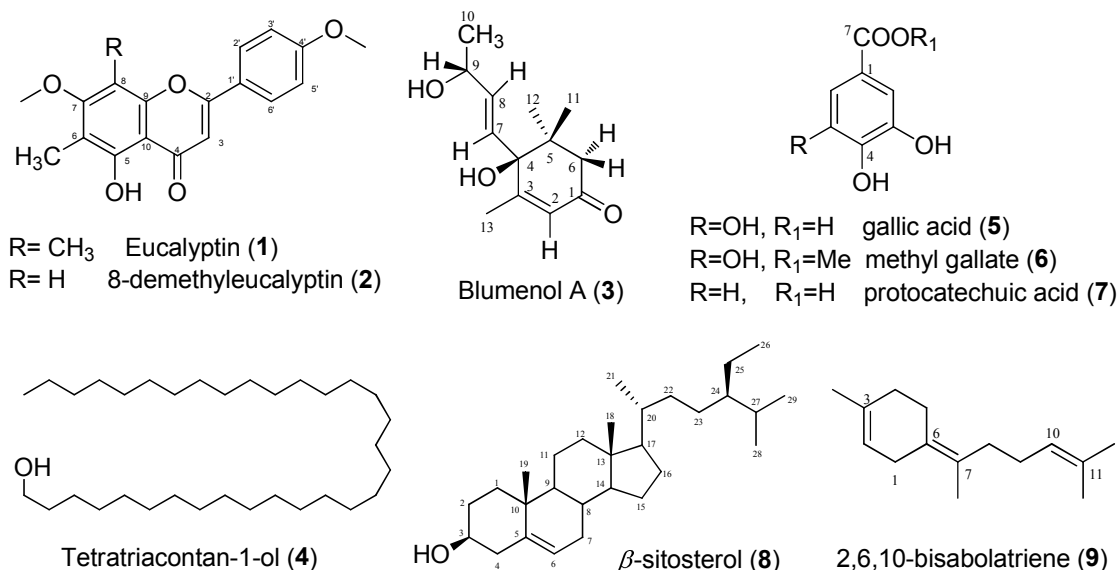


Figure 1. Structures of isolated compounds (**1-9**).

Compound **4** was obtained as white powder from *n*-hexane fraction. 1H -NMR spectrum of **4** presented signals of hydroxymethylene protons at δ_H 3.64 (2H, t, $J = 7.0$ Hz, H-1); two methylenes at δ_H 1.56 (4H, t, $J=7.0$ Hz, H₂-2,3), sixty consecutive methylene protons at δ_H 1.25 – 1.26 (60H, s) and one methyl group (δ_H 0.88, 3H, t, $J = 7.0$ Hz, H-34). These data suggested

that **4** was a long-chain alcohol. The ESI-MS spectrum of **4** in negative mode showed a pseudomolecular ion peak at m/z 493 $[M-H]^-$, indicating the molecular formula of **4** as $C_{34}H_{70}O$. Compound **4** was determined to be *n*-tetratriacontanol, other name sapiol [12].

Compound **5** and **6** were isolated from ethyl acetate fraction. The ^{13}C -NMR and DEPT spectra of compounds **5** and **6** showed signals of a carbonyl group (δ_C 169.1 - 170.5, C-7), two pairs of equivalent carbons at δ_C 110.4 (d, C-2, C-6), 146.5 (s, C-3, C-5) and two quaternary carbons at δ_C 139.7 (s, C-4) / 122.0 (s, C-1), belonging to a 3,4,5-trisubstituted benzoic acid derivative. The 1H - and ^{13}C -NMR spectra also showed the presence of a methyl group in compound **6**. From spectroscopic evidences, compound **5** was identified as 3,4,5-trihydroxy benzoic acid or gallic acid [13] and compound **6** was its derivative as 3,4,5-trihydroxy-methylbenzoate or methyl gallate [14].

Compound **7** was also obtained from *n*-hexane fraction. The 1H - and ^{13}C -NMR spectra of **7** were similar to those of gallic acid **5**, except for the presence of a proton substituted for an hydroxy group at carbon C-5 (δ_C 117.8). In agreement with that, the 1H -NMR spectrum of **7** showed signals of three aromatic methine protons at δ_H 7.45 (s, H-2), 6.81 (d, $J = 8.0$ Hz, H-5) and 7.44 (d, $J = 8.0$ Hz, H-6), belonging to a 3,4-disubstituted benzoic acid. In comparison to literature, compound **7** was identified as 3,4-dihydroxybenzoic acid, trivial name protocatechuic acid.

Compound **8**, obtained as white powder from *n*-hexane fraction, was elucidated as β -sitosterol on the basis of the NMR data and comparison with the data reported in the literature [15].

Compound **9** was obtained as colourless oil from *n*-hexane fraction of methanol extract of *C. citrinus* leaves. From ESI-MS ion peak at m/z 205 $[M+H]^+$, the molecular formula of **9** was identified as $C_{15}H_{24}$ ($M=204$). The 1H NMR spectrum of **9** contained the signals of four quaternary methyls at δ_H 1.68 (3H, s, H₃-15) / 1.60 (9H, s, H₃-12, H₃-13 and H₃-14), five methylene at δ_H 1.99 -2.07; and two methines at δ_H 5.10 (2H, s, H-2, -10). These data, together with the presence of 15 carbon signals in the ^{13}C NMR spectrum (4 x C₄, 2 x CH, 5 x CH₂, 4 x CH₃) suggested that **9** was a sesquiterpene. By comparison of the spectroscopic data with those published in literature, compound **9** was identified as 2,6,10-bisabolatriene [16].

4. CONCLUSION

In the search for bioactive constituents from Vietnamese plants, the leaves and stems of *Callistemon citrinus* (Curtis) Skeels were selected for chemical investigation. From the methanolic extract of this species, solvent-soluble fractions with increased polarity were produced including *n*-hexane, dichloromethane, ethyl acetate and water. Phytochemical analysis of different fractions of the plant led to the isolation of eight phenolic compounds including two flavones (eucalyptin (**1**) and 8-demethyleucalyptin (**2**)), two alcohols (blumenol A (**3**), *n*-tetratriacontanol (**4**)), three benzoic acid derivatives (acid gallic (**5**), methyl gallate (**6**) and protocatechuic acid (**7**)), one sterol (β -sitosterol (**8**)), along with one sesquiterpene (2,6,10-bisabolatriene (**9**)). The structures of the natural compounds were determined from the spectroscopic evidences including 1D- and 2D-NMR and ESI-MS.

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REFERENCES

1. Pham Hoang Ho, ed. *An Illustrated Flora of Vietnam* Vol. II. 2000, Youth Publisher: Hanoi, Vietnam. p. 41.
2. Nazreen S., Kaur G., Alam M. M., Shafi S., Hamid H., Ali M. and Alam M. S. - New flavones with antidiabetic activity from *Callistemon lanceolatus* DC, *Fitoterapia* **83** (8) (2012) 1623-1627.
3. Khambay B. P. S., Beddie D. G., Hooper A. M., Simmonds M. S. J., and Green P. W. C. - New Insecticidal Tetradecehydroxanthenediones from *Callistemon viminalis*, *J. Nat. Prod.* **62** (12) (1999) 1666 -1667.
4. Rattanaburi S., Mahabusarakam W., Phongpaichit S., and Carroll A. R. - Acylphloroglucinols from *Callistemon lanceolatus* DC, *Tetrahedron* **69** (30) (2013) 6070-6075.
5. Jeong W., Hong S. S., Kim N., Yang Y. T., Shin Y. S., Lee C., Hwang B. Y. and Lee D. - Bioactive triterpenoids from *Callistemon lanceolatus*, *Arch. Pharm. Res.* **32** (6) (2009) 845-849.
6. Rattanaburi S., Mahabusarakam W., Phongpaichit S. and Carroll A. R. - Neolignans from *Callistemon lanceolatus*, *Phytochemistry Lett.* **5** (1) (2012) 18-21.
7. Goyal P. K., Jain R., Jain S. and Sharma A. - A Review on biological and phytochemical investigation of plant genus *Callistimon*, *Asian Pac. J. Trop. Biomed.* **2** (3, Supplement) (2012) S1906-S1909.
8. Khanh P.N., Duc H.V., Huong T.T., Son N.T., Ha V.T., Van D.T., Tai B.H., Kim J.E., Kim Y.H. and Cuong N.M. - Acylphloroglucinol derivatives and triterpenoids with soluble epoxide hydrolase inhibitory activity from *Callistemon citrinus*, *Fitoterapia* (submitted) (2015).
9. Khanh P.N., Duc H.V., Huong T.T., Son N.T., Ha V.T., Van D.T., Tai B.H., Kim Y.H. and Cuong N.M. - Flavonoids and triterpenoids with inhibitory effect on NO production in LPS-activated RAW264.7 macrophage from *Callistemon citrinus*, *Bull. Korean Chem. Soc.* (submitted) (2015).
10. Sidana J., Neeradi D., Choudhary, A., Singh S., Foley W. J. and Singh I. P. - Antileishmanial polyphenols from *Corymbia maculata*, *J. Chem. Scie.* **125** (4) (2013) 765-775.
11. Wu D.-K., Zhang C.-P., Zhu C.-Y., Wang Y.-L., Guo L.-L., Zhang K.-Q. and Niu X.-M. - Metabolites from Carnivorous Fungus *Arthrobotrys entomopaga* and Their Functional Roles in Fungal Predatory Ability, *J. Agr. Food Chem.* **61** (17) (2013) 4108-4113.
12. Shukla Y.N. and Thakur R.S. - Heptatriacontan-4-one, tetratriacontanyl octacosanoate and other constituents from *Pedaliium murex*, *Phytochemistry* **22** (4) (1983) 973-974.
13. Liu Y., Pukala T.L., Musgrave I.F., Williams D.M., Dehle F.C. and Carver J.A. - Gallic acid is the major component of grape seed extract that inhibits amyloid fibril formation, *Bioorg. Med. Chem. Lett.* **23** (2013) 6336-6340.
14. Wang C.R., Zhou R., Ng T.B., Wong J.H., Qiao W.T. and Liu F. - First report on isolation of methyl gallate with antioxidant, anti-HIV-1 and HIV-1 enzyme inhibitory activities

- from a mushroom (*Pholiota adiposa*)., *Environ. Toxicol. Pharmacol.* **37** (2) (2014) 626-637.
15. Carle R., Beyer J., Chemina A. and Krempp E. 2H NMR determination of site-specific natural isotope fractionation in (-)- α -bisabolols, *Phytochemistry* **31** (1) (1992) 171-174.
16. Lee T-H., Chiou J-L., Lee C-K. and Kuo, Y-H. - Separation and determination of chemical constituents in roots of *Rhus javanica* L. var. *roxburghiana*, *J. Chinese Chem. Soc.* **52** (2005) 833-841.

TÓM TẮT

CÁC HỢP CHẤT PHENOL PHÂN LẬP TỪ LÁ VÀ CÀNH LOÀI *CALLISTEMON CITRINUS*

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Từ lá và cành loài Tràm bông đỏ *Callistemon citrinus* (Curtis) Skeels tám hợp chất phenol và một hợp chất sesquiterpene đã được phân lập và xác định cấu trúc bao gồm hai flavon (eucalyptine (1) và 8-demethyleucalyptine (2)), hai hợp chất alcol (blumenol A (3), *n*-tetratriacontanol (4)), ba dẫn xuất của acid benzoic (acid gallic (5), methyl gallate (6) và acid protocatechuic (7)), một hợp chất sterol (β -sitosterol (8)) và một sesquiterpene (2,6,10-bisabolatriene (9)). Cấu trúc hóa học của các hợp chất trên được xác định nhờ các phương pháp hóa lý và phương pháp phổ bao gồm phổ cộng hưởng từ nhân 1 chiều, 2 chiều và phổ khối lượng.

Từ khóa: *Callistemon citrinus* (Curtis) Skeels, flavonoid, triterpenoid.