

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].

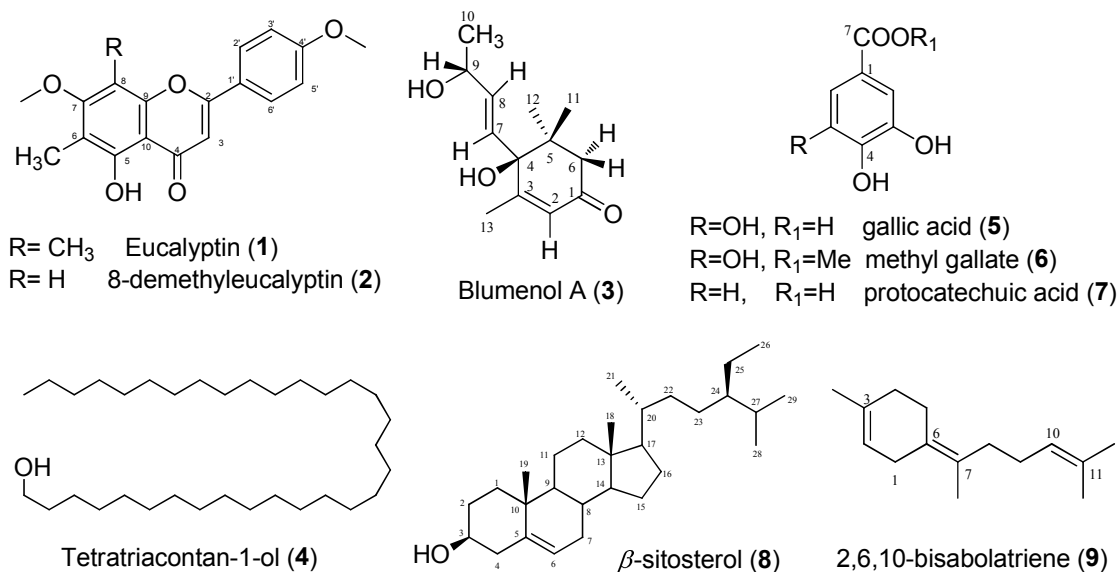


Figure 1. Structures of isolated compounds (**1-9**).

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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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.