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Research Article

ISOLATION OF SOME COMPOUNDS FROM *PSYCHOTRIA ADENOPHYLLA* WALL.

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ABSTRACT

*Pychotria adenophylla (Wall.) Kuntze has many popular traditional uses in Asian countries, for treatments of hemorrhage, respiratory troubles, analgesia, or resistance. This study was done to investigate the phytochemicals of P. adenophylla grown in Binh Chau, Ba Ria-Vung Tau Province. Their chemical structures were elucidated by using Nuclear Magnetic Resonance spectroscopy (NMR), as well as by the comparison of their NMR data with reported ones. Five compounds consisted of vanillin (**1**), coniferyl aldehyde (**2**), chrysophanol (**3**), α-asarone (**4**), and γ-asarone (**5**).*

***Keywords*:** α-asarone; γ-asarone; coniferyl aldehyde; chrysophanol; pychotria adenophylla; vaniline

1. Introduction

The genus *Psychotria* (*Rubiaceae*) comprises approximately 1,700 species. These plants are distributed popularly in tropical and subtropical areas (Davis et al., 2001). Different parts of different species (leaves, roots, and rhizomes) have been widely used in traditional medicine for treating fever, bronchitis, ulcers, and stomachache and for gynecological hemorrhage in females (Calixto et al., 2016). The plant *Psychotria adenophylla* Wall was used to treat respiratory diseases and bronchial disorders. Pharmacological studies have indicated that the genus *Psychotria* had various biological activities, such as antimicrobial, antiviral, analgesic, hypoglycemic, and strong cytotoxic activities against several cancer cell lines (Benevides et al., 2005; Pimenta et al., 2011). This report aims to the isolation and structural elucidation of five compounds vanillin (**1**), coniferyl aldehyde (**2**), chrysophanol (**3**), α-asarone (**4**), and γ-asarone (**5**) from leaves of

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Psychotria adenophylla collected from Binh Chau, Ba Ria-Vung Tau Province by various spectroscopic methods.

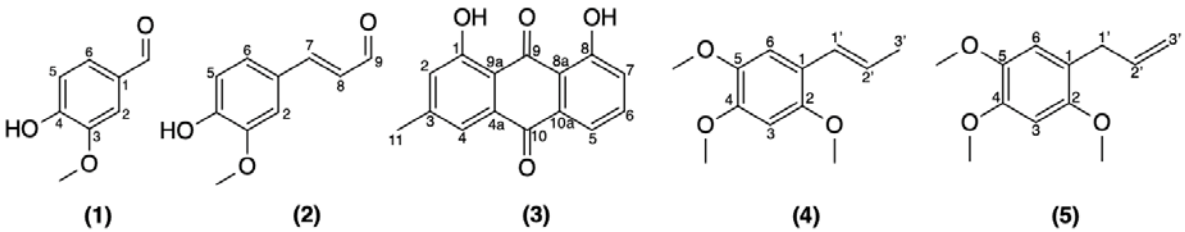

Figure 1. *Chemical structures of isolated compounds* ***1-5***

2. The experiment

2.1. General experimental procedures

The NMR spectra were recorded on a Bruker Avance spectrometer (500 MHz or 400 MHz for ^{1}H–NMR and 125 MHz or 100 MHz for ^{13}C–NMR) with samples dissolved in acetone-d_6 and methanol-d_4. Distilled solvents (Chemsol) were used to prepare extracts and to elute column chromatography and thin-layer chromatography. Thin-layer chromatography was carried out on silica gel 60 (Merck, 40-63 μm) and spots were visualized by spraying with 10% H_2SO_4 solution, followed by heating.

2.2. Plant materials

Leaves of *Psychotria adenophylla* were collected in Binh Chau, Ba Ria-Vung Tau Province in May-July 2022. The scientific name of the material was identified as *Psychotria adenophylla* by Assoc. Prof. Dr. Dang Van Son (Institute of Tropical Biology, Vietnam Academy of Science and Technology). A voucher specimen (No UE-P018) was deposited in the herbarium of the Department of Organic Chemistry, Faculty of Chemistry, Ho Chi Minh University of Education, Ho Chi Minh City, Vietnam.

2.3. Extraction and isolation

Dried leaves of *Pyschotria adenophylla* (3.0 kg) were extracted with methanol three times (3 x 10 L) at room temperature. The filtrated solution was evaporated at reduced pressure to obtain a crude extract (650 g). This extract was separated into *n*-hexane extract (22.2 g, **H**), *n*-hexane: EtOAc extract (67 g, **HEA**), and EtOAc (190.0 g, **EA**) by liquid-liquid partition method. The **H** extract was applied to silica gel column chromatography and eluted with methanol to afford 11 fractions (coded, H1-H11). The fraction H4 (2.1 g) was further subjected to a silica gel column chromatography, eluted with the solvent system of *n*-hexane: ethyl acetate: acetone (9:1:1, v/v/v) to give seven subfractions (H4.1-H4.7). Next, subfraction HEA4.1 (123 mg) was separated into three different fractions (coded, S1-S3) by a silica gel column chromatography, eluted with the same solvent system. Purifying fraction S1 (31 mg) by silical gel CC gave compounds **1** (15.5 mg), **2** (11.5 mg), and **3** (3.5 mg). Subfraction HEA4.5 (67 mg) was subjected to a silica gel column chromatography, using a solvent system of *n*-hexane: chloroform: methanol: water (10:1:0.2:0.01, v/v/v/v) to give

three different fractions (coded, R1-R3). The same manner was applied to the fraction R3 (36 mg) to afford the mixture of **4** and **5** (17.2 mg).

- **Vanillin** (**1**). White amorphous solid. ^{1}H-NMR (500 MHz, acetone-d_6, δ ppm, *J* in Hertz): 9.83 (1H, *s*, H-7), 7.48 (1H, *d*, 2.0, H-2) ,7.46 (1H, *dd*, 8.0, 2.0, H-6), 7.02 (1H, *d*, 8.0, H-5), 3.94 (3H, *s*, 3-OCH_3) (Ito et al., 2001).
- **Coniferyl aldehyde** (**2**). White amorphous solid. ^{1}H-NMR (400 MHz, methanol-d_4, δ ppm, *J* in Hertz): 9.48 (1H, *d*, 8.0, H-9), 7.52 (1H, *d*, 16.0, H-7), 7.16 (1H, *d*, 2.0, H-2), 7.08 (1H, *dd*, 8.0, 2.0, H-6), 6.76 (1H, *d*, 8.0, H-5), 6.58 (1H, *dd*, 16.0, 8.0, H-8), 3.81 (3H, *s*, CH_3-O-3) (Ito et al., 2001).
- **Chrysophanol** (**3**). Yellow amorphous solid. ^{1}H-NMR (500 MHz, methanol-d_4, δ ppm, *J* in Hertz): 12.05 (1H, *s*, 8-OH), 11.95 (1H, *s*, 1-OH), 7.83 (1H, *t*, 8.0, H-6), 7.79 (1H, *dd*, 8.0, 1.0, H-5), 7.63 (1H, *d*, 1.0, H-4), 7.36 (1H, *dd*, 8.0, 1.0, H-7), 7.20 (1H, *brs*, H-2), 2.50 (3H, *s*, H-11). ^{13}C-NMR (125 MHz, methanol-d_4, δ ppm): 190.0 (C-9), 182.4 (C-10), 163.7 (C-8), 163.3 (C-1), 150.7 (C-3), 138.3 (C-6), 134.8 (C-10a), 125.2 (C-7), 124.9 (C-2), 121.7 (C-4), 120.3 (C-5), 116.7 (C-8a), 115.0 (C-4a), 114.7 (C-9a), 20.1 (C-11) (Zhang et al., 2012).
- **α-Asarone (4).** Colorless oil. ^{1}H-NMR (500 MHz, acetone-d_6, δ ppm, *J* in Hertz): 7.04 (1H, *s*, H-6), 6.67 (1H, *s*, H-3), 6.64 (1H, *dd*, 16.0, 2.0, H-1′), 6.13 (1H, *dq*, 16.0, 6.5, H-2′), 3.84 (3H, *s*, 2-OCH_3), 3.82 (3H, *s*, 3-OCH_3), 3.78 (3H, *s*, 5-OCH_3), 1.84 (3H, *dd*, 6.5, 2.0, H-3′). ^{13}C-NMR (125 MHz, acetone-d_6, δ ppm): 151.1 (C-4), 149.6 (C-2), 143.7 (C-5), 125.3 (C-2′), 122.9 (C-1′), 118.3 (C-1), 110.7 (C-6), 98.5 (C-3), 56.1 (5-OCH_3), 55.8 (4-OCH_3), 55.5 (2-OCH_3), 17.8 (C-3′) (Patra & Mitra, 1981).
- **γ-Asarone (5).** Colorless oil. ^{1}H-NMR (500 MHz, acetone-d_6, δ ppm, *J* in Hertz): 6.76 (1H, *s*, H-6), 6.69 (1H, *s*, H-3), 5.95 (1H, *m*, H-2′), 5.02 (1H, *m*, H-3′a), 4.97 (1H, *m*, H-3′b), , 3.81 (3H, *s*, 2-OCH_3), 3.83 (3H, *s*, 3-OCH_3), 3.74 (3H, *s*, 5-OCH_3), 3.29 (2H, *d*, 6.5, H-1′). ^{13}C-NMR (125 MHz, acetone-d_6, δ ppm): 151.5 (C-2), 148.6 (C-4), 143.2 (C-5), 138.1 (C-2′), 119.6 (C-1), 115.4 (C-6), 114.3 (C-3′), 98.7 (C-3), 33.4 (C-1′), 56.3 (5-OCH_3), 55.7 (2-OCH_3), 55.7 (4-OCH_3) (Patra & Mitra, 1981).

3. Results and discussion

Compound **1** was obtained as a white amorphous solid. The ^{1}H NMR spectrum of **1** showed three proton signals characterized for a 1,3,4-trisubstitutedbenzene ring, including δ_H 7.48 (1H, *d*, 2, H-2),7.46 (1H, *dd*, 8.0, 2.0, H-6), and 7.02 (1H, *d*, 8.0, H-5), an aldehyde proton at δ_H 9.83 (1H, *s*, H-7) and a methoxy group at δ_H 3.94 (3H, s, 3-OCH_3). Based on the comparison of its NMR data with the published ones (Ito et al., 2001), **1** was thus determined to be vanillin.

The ^{1}H NMR spectrum of **2** was similar to those of **1**, except for the changes in the side chain at C-1. Particularly, the aldehyde group at C-1 in **1** was replaced by the α,β-unsaturated aldehyde group (-CH=CH-CHO) in **2**. The good compatibility between its NMR

data and those in the literature (Ito et al., 2001) indicated the structure of **2** being coniferyl aldehyde.

Compound **3** was obtained as a yellow amorphous powder. The ^{1}H NMR spectrum indicated the presence of a methyl group [δ_H 2.50 (3H, *s*, H-11)], five aromatic protons [δ_H 7.83 (1H, *t*, 8.0, H-6), 7.79 (1H, *dd*, 8.0, 1.0, H-5), 7.63 (1H, *d*, 1.0, H-4), 7.36 (1H, *dd*, 8.0, 1.0, H-7), 7.20 (1H, *brs*, H-2)], and two hydrogen-bond hydroxy groups [δ_H 12.05 (1H, *s*, 8-OH), 11.95 (1H, *s*, 1-OH)]. The ^{13}C-NMR spectrum of **3** showed signals of 15 carbons, comprising of two ketone carbon signals at δ_C 190.0 (C-9) and 182.4 (C-10), seven quaternary carbons [δ_C 114.7 (C-9a), 115.0 (C-4a), 116.7 (C-8a), 134.8 (C-10a), 150.7 (C-3), 163.3 (C-1), 163.7 (C-8), the two latter oxygenated], five aromatic methines [δ_C 124.9 (C-2), 121.7 (C-4), 120.3 (C-5), 138.3 (C-6) and 125.2 (C-7)], and one methyl at δ_C 20.1 (C-11). Based on the above analysis along with the good compatibility of its NMR data with those of chrysophanol (Zhang et al., 2012), the chemical structure of **3** was identified to be chrysophanol.

The mixture of **4** and **5** was obtained as a colorless oil. Detailed comparison of NMR data of this mixture to those of α-asarone and γ-asarone (Patra & Mitra, 1981), compounds **4** and **5** were identified as shown in Figure 1.

4. Conclusions

From the leaves of *P. adenophylla* collected in Ba Ria – Vung Tau Province, five compounds, vanillin (**1**), coniferyl aldehyde (**2**), chrysophanol (**3**), α-asarone (**4**), and γ-asarone (**5**) were isolated. Their chemical structures were determined by using the NMR spectroscopic method as well as by comparison with the literature. To the best of our knowledge, these five compounds were isolated from *P. adenophylla* for the first time. Further studies on this species are in the progress.

❖ ***Conflict of Interest:*** *Authors have no conflict of interest to declare.*

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PHÂN LẬP MỘT SỐ HỢP CHẤT HỮU CƠ TỪ CÂY LẤU TUYẾN *PSYCOTRIA ADENOPHYLLA* (WALL.) KUNTZE

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TÓM TẮT

*Pychotria adenophylla (Wall.) Kuntze có nhiều ứng dụng phổ biến trong y học cổ truyền ở các nước châu Á như trị xuất huyết, khó thở, giảm đau, chống oxy hóa, miễn dịch… Nghiên cứu này được thực hiện nhằm khảo sát thành phần hoá học của lá cây P. adenophylla được tìm thấy ở Bình Châu, tỉnh Bà Rịa – Vũng Tàu. Cấu trúc hóa học của chúng đã được xác định bằng cách sử dụng quang phổ cộng hưởng từ hạt nhân, cũng như bằng cách so sánh dữ liệu NMR của chúng với dữ liệu đã được báo cáo. Năm hợp chất gồm vanillin **(1)**, coniferyl aldehyde **(2)**, chrysophanol **(3)**, α-asarone **(4)** và γ-asarone **(5)**.*

Từ khóa: α-asarone; γ-asarone; coniferyl aldehyde; chrysophanol; pychotria adenophylla; vaniline