

Oligostilbenoids and Phenolic Compounds from the Stem Bark of *Shorea materialis* RIDL. (Dipterocarpaceae)

(Oligostilbenoid dan Sebatian Fenol daripada Kulit Batang *Shorea materialis* RIDL. (Dipterocarpaceae))

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Received: 25 April 2024/Accepted: 24 February 2025

ABSTRACT

The *Shorea* genus is known for its abundance of sources of oligostilbenoids, which exhibit diverse biological functions. Nevertheless, despite its considerable potential, there remains a dearth of phytochemical investigations including *Shorea materialis*. Consequently, this study aims to isolate the chemical constituents from the stem bark of this species. The crude extract of *S. materialis* was obtained from the maceration technique and fractionation using vacuum liquid chromatography. The resulting fractions were further purified via column chromatography. The molecular structures of the isolated molecules were determined using various spectroscopic techniques. Two dimer stilbenoids, ampelopsins A (**1**) and D (**2**), three trimers, namely α -viniferin (**3**), hopeanolin (**4**) and davidiol A (**5**), one tetramer, vaticanol B (**6**), together with two phenolic compounds, *p*-hydroxybenzaldehyde and (**7**) 2,3,5-trihydroxy benzoic acid (**8**) were effectively isolated. The isolation of these compounds was the first report from this species.

Keywords: Dipterocarpaceae; oligostilbenoids; phenolic compounds; *Shorea materialis*

ABSTRAK

Genus *Shorea* terkenal dengan kekayaan sumber oligostilbenoid yang menunjukkan pelbagai fungsi biologi. Walaupun genus ini mempunyai potensi yang tinggi, kajian fitokimia berkaitan spesies daripada genus ini masih sedikit termasuklah *Shorea materialis*. Oleh itu, kajian ini dijalankan bertujuan untuk mengasingkan sebatian kimia daripada kulit batang spesies ini. Ekstrak kasar *S. materialis* diperolehi daripada kaedah rendaman dan fraksinasi menggunakan kromatografi cecair vakum. Fraksi yang diperolehi seterusnya ditulenkan lagi menggunakan kromatografi turus. Struktur molekul sebatian yang telah dipencilkan ditentukan menggunakan pelbagai teknik spektroskopi. Dua sebatian stilbenoid dimer, ampelopsin A (**1**) dan D (**2**), tiga stilbenoid trimer, iaitu α -viniferin (**3**), hopeanolin (**4**) dan davidiol A (**5**), satu stilbenoid tetramer, vaticanol B (**6**), bersama dengan dua sebatian fenol, *p*-hidroksibenzaldehid dan (**7**) asid benzoik 2,3,5-trihidroksi (**8**) telah berjaya dipencilkan. Penulenan sebatian ini merupakan pertama kali dilaporkan daripada spesies ini.

Kata kunci: Dipterocarpaceae; oligostilbenoid; sebatian fenol; *Shorea materialis*

INTRODUCTION

The *Shorea* genus is a member of the Dipterocarpaceae family, encompassing 196 species, primarily comprising rainforest trees (Mabberley 2017). *Shorea* is indigenous to Southeast Asia, from Northern India to Malaysia, Indonesia, and the Philippines. The genus in question exhibits dominance in the tropical forest canopy of western Malaysia and the Philippines (Larjavaara & Muller-Landau 2013). Ashton (2004) and Larjavaara (2013) reported that *Shorea faguetiana* was the tallest tropical angiosperm, reaching a height of 96.9 m, as observed in Tawau Hills National Park, Sabah. Many of these species are

characterised by their vast size, namely as forest-emergent organisms, with average heights ranging from 40 to 70 m. Additionally, there are instances where certain species surpass even greater heights, exceeding 80 m. The *Shorea* genus encompasses several species, namely *S. curtisii*, *S. lepidota*, *S. leprosula*, *S. roxburghii*, *S. acuminata*, *S. robusta*, *S. smithiana*, and *S. sumatrana* (Corner 1988). Within the Dipterocarps family, *Shorea* holds significant economic importance in Southeast Asia, emerging as the primary exporter in the building and furniture sectors (Jong 1976; Mufarhatun, Hilwan & Rachmat 2021). Certain *Shorea* species, such as *S. robusta*, exhibit various

pharmacological properties, including anti-inflammatory, anti-obesity, antibacterial, wound healing, antipyretic, and analgesic effects (Soni et al. 2013).

Over the years, extensive research has been conducted on the phytochemical investigation of *Shorea* species, uncovering its distinctive and intricate composition. The genus *Shorea* is a plant that contains a diverse array of oligostilbenoids, which exhibit different levels of polymerisation, such as monomers (Patcharamun et al. 2011), dimers (Kamarozaman et al. 2019), trimers (Guo et al. 2020; Ito, Nishiguchi & Inuma 2019; Ito et al. 2022), and tetramers (Kamarozaman et al. 2019). The oligostilbenoids have been associated with various biological activities, such as antibacterial, antiviral (Ito et al. 2018), antidiabetic (Morikawa et al. 2012), anticholinesterase (Kamarozaman et al. 2019), immunotherapy (Chhabra et al. 2021) and chemopreventive agent (Espinoza & Inaoka 2017; Kamarozaman et al. 2013; Moriyama et al. 2016; Zhang, Henry & Zhang 2020). *Shorea materialis*, or known as *Balau Pasir* among the Peninsular Malaysians, is a tree predominantly found in marshy environments within Pahang, Perak, Terengganu, and Johor (Yong et al. 2011). So far, no comprehensive investigation has been conducted on the phytochemical composition of *S. materialis*. In order to further explore the therapeutic potential of this species, we undertook a phytochemical analysis of the stem bark of *S. materialis*, leading to the identification of the plant's oligostilbenoids.

MATERIALS AND METHODS

CHROMATOGRAPHIC METHODS

The profiling procedure uses Aluminium-supported silica gel 60 F₂₅₄ for thin-layer chromatography (TLC). The TLC plates are spotted using a fine glass capillary tube and developed in the chromatographic chamber with various solvent systems at room temperature. The spots were visualised under UV light (254 and 356 nm). The premix sample was coated with Silica gel 60 (0.2-0.5 mm) for column chromatography (Merck, Germany). The vacuum liquid chromatography (VLC) technique used Silica gel 60 G for thin-layer chromatography (Merck, Germany) for fractionation, while the column chromatography (CC) method used Silica gel 60 (0.040-0.062 mm) 230 – 400 mesh ASTM (Merck, Germany) for isolation and purification processes.

PLANT COLLECTION

The stem bark of *S. materialis* was collected from the Reserve Forest, Universiti Teknologi MARA (UiTM), Jengka, Pahang. The plant material was identified by Siti Munirah Mat Yunoh, a botanist affiliated with the Forest Research Institute Malaysia (FRIM). The voucher specimen, identified as FSG2, was appropriately stored and catalogued at the FRIM herbarium.

EXTRACTION PROCEDURE

The stem bark of *S. materialis* was dried, cut into small pieces and ground into granules (1 kg). The granules were macerated in acetone (1 L) at room temperature for 24 h and repeated thrice. The acetone extract was filtered using No 1 Whatman filter paper (32 cm) and evaporated under reduced pressure at 20 mbar and temperature of 50 °C using a rotary evaporator to yield concentrated crude acetone extract (117 g). The crude extract was stored at 5° C for further use.

ISOLATION AND PURIFICATION OF CONSTITUENT

The crude acetone extract was subjected to vacuum liquid chromatography (VLC) eluted with a mixture of *n*-hexane: ethyl acetate in increasing polarity to obtain eight fractions, SM 1 (*n*-hex, 100%), SM 2 (*n*-hex:EtOAc, 9:1), SM 3 (*n*-hex:EtOAc, 8:2), SM 4 (*n*-hex:EtOAc, 7:3), SM 5 (*n*-hex:EtOAc, 6:4), SM 6 (*n*-hex:EtOAc, 5:5), SM 7 (EtOAc, 100%), SM 8 (MeOH, 100%). SM 5 (26.4 g) was re-fractionated by VLC (*n*-hex: EtOAc), yielding seven subfractions SM 5.1 – 5.7. Subfraction SM 5.5 (2.32 g) was further purified using a 2-cm diameter column chromatography (CC) with the eluent chloroform: methanol (CHCl₃:MeOH from 10:0 to 8:2, v/v) yielding compounds **3** (8 mg) and **4** (4 mg). Another CC was also performed on subfraction SM 5.6 (2.13 g) using CHCl₃: MeOH in ascending polarity, attaining compounds **1** (5 mg) and **2** (4 mg), followed by preparative thin layer chromatography (p-TLC) technique with a solvent system of CHCl₃:MeOH (8:2) to acquire compound **6** (4 mg). Compound **5** (5 mg) was obtained from SM 6 (568.4 mg) by purification with CC. Fractions 3 and 4 were combined, giving a total mass of 1.0413 g and proceeded to a 2 cm-diameter CC with the eluent *n*-hexane:ethyl acetate (*n*-hex:EtOAc from 10:0 to 5:5, v/v) affording compounds **7** (3 mg) and **8** (2 mg).

STRUCTURAL ELUCIDATION

The ¹H and ¹³C NMR spectra were acquired in deuterated acetone-*d*₆ using Bruker 500 and 600 Ultrashield NMR spectrometers at 500/600 and 125 MHz, respectively. The UV absorptions spectra were obtained in methanol solution, employing Biochrome WPA Biowave II UV/Vis Spectrophotometer and IR spectra were attained by using Bruker FT-IR Spectrometer ALPHA I model.

RESULTS AND DISCUSSION

Compound **1** was obtained as a brown amorphous powder. The UV absorption (MeOH) at λ_{max} 220 and 283 nm indicated the presence of a substituted aromatic system in the structure which was supported by the IR absorption at ν_{max} 3339 (OH), 2922 (C–H aliphatic), 1609, 1513, and 1453 (C=C aromatic), 1235 and 1174 (C–O oxyaryl), and 833 (*p*-disubstituted benzene) cm⁻¹. The ¹H NMR spectral data of **1** displayed characteristic of a

dimerstilbenoid, which showed the existence of two groups of *ortho*-coupled aromatic protons that can be attributed to two 4-hydroxyphenyl groups [ring A1: δ_{H} 7.10 (2H, d, $J = 8.4$ Hz, H-2a and 6a), 6.77 (2H, d, $J = 8.4$ Hz, H-3a and 5a), and ring B1: δ_{H} 6.88 (2H, d, $J = 8.4$ Hz, H-2b and 6b), 6.62 (2H, d, $J = 8.4$ Hz, H-3b and 5b)], two pairs of *meta*-coupled aromatic protons on two 1,2,3,5-tetrasubstituted benzene rings [ring A2: δ_{H} 6.22 (1H, br s, H-14a), 6.42 (1H, d, $J = 1.8$ Hz, H-12a), and ring B2: δ_{H} 6.59 (1H, d, $J = 1.8$ Hz, H-14b), 6.14 (1H, d, $J = 1.8$ Hz, H-12b)], five phenolic hydroxyl groups (δ_{H} 8.05, 8.22, 8.23, 8.54, and 8.57). The ^1H NMR spectrum additionally displayed two sets of coupled aliphatic methine protons in sequence: H-7a (δ_{H} 5.76, d, $J = 11.4$) and H-8a (δ_{H} 4.15, d, $J = 11.4$); H-7b (δ_{H} 5.44, d, $J = 4.8$) and H-8b (δ_{H} 5.41, d, $J = 4.8$), along with a single aliphatic hydroxyl group (δ_{H} 3.46) as demonstrated in Table 1.

The ^{13}C NMR spectrum (Table 2) of **1** exhibited 22 signals representing 28 carbons. The signals were assigned to four sp^3 carbons which consist of two aliphatic carbons at δ_{C} 43.5 and 70.9, the latter being deshielded due to attachment with hydroxyl substituent, one aliphatic methine carbon at δ_{C} 49.2 and one aliphatic oxymethine carbon at δ_{C} 88.1. Another 24 signals belong to sp^2 carbons, in which five signals resonated at δ_{C} 156.1 - 159.9 were found to be oxyaryl carbons, six signals representing aromatic quaternary carbons (δ_{C} 42.5 - 118.2) and 13 aromatic methine carbons (δ_{C} 96.7 - 129.6).

The significant coupling constant of H-7a and H-8a (11.4 Hz) in compound **1** demonstrated that these protons were in *trans*-configuration (Karplus 1963), which is shown by (+)-ampelopsin A (11.7 Hz) (Oshima et al. 1990). Meanwhile, *trans*-configuration of H-7b and H-8b in (+)-ampelopsin A, appeared as a doublet, producing a coupling constant of 5.0 Hz for both H-7b and H-8b, which data is similar to compound **1** [H-7b and H-8b (4.8 Hz)]. Thus, the structure of **1** (Figure 1) was determined as ampelopsin A.

Compound **2** appeared as brown amorphous powder. The UV absorption (MeOH) at λ_{max} 298 and 325 nm indicated the presence of a substituted aromatic system in the structure. The ^1H NMR (Table 1) spectral data of compound **2** exhibited four sets of *ortho*-coupled aromatic proton signals in an AABB spin system, belonging to two units of *p*-hydroxybenzene ring resonated at δ_{H} 7.17/6.65 and 7.11/6.74. Four sets of *meta*-coupled aromatic proton signals in the AX spin system at δ_{H} 6.79/6.29 and 6.11/6.10, assignable to one unit of tetrasubstituted benzene rings and one unit of 1,3,5-trisubstituted benzene ring were also identified, respectively. These data which coexists with compound **1**, suggested that **2** is an oxidative dimer stilbenoid. However, the difference in the structure was observed as the ^1H spectrum of **2** showed two sets of mutually coupled aliphatic methine proton signals at δ_{H} 4.43 and 4.14 (1H each) and one free olefinic proton signal at δ_{H} 7.04, supporting the absence of a benzofuran ring in the structure of **2**.

The analysis of its ^{13}C NMR (Table 2) data showed the resonance of six aromatic oxyaryl carbons (δ_{C} 156 - 159.7), six quaternary carbons (δ_{C} 123.8 - 149.9), 13 aromatic methine carbons (δ_{C} 98.4 - 131.0), one olefinic carbon (δ_{C} 122.6) and two aliphatic methine carbons (δ_{C} 58.7 and 59.5). The unique structure in **2** as observed in its NMR data, was possible due to its biogenetic pathway. In this case, ϵ -viniferin is considered to be a biogenetically important precursor as the acid protonates the oxygen atom on the dihydrofuran ring, which then makes a nucleophilic attack on the double bond, forming a five-membered ring intermediate. The intermediate is then deprotonated, resulting in structure **2** (Takaya et al. 2002). Consequently, compound **2** (Figure 1) is determined as ampelopsin D, which was previously reported from *Ampelopsis brevipedunculata* var. *Hancei* (Oshima & Ueno 1993).

Compound **3** (Figure 1) was isolated as a brownish amorphous powder. The IR spectrum indicated the presence of a substituted aromatic system in the structure at ν_{max} 3281 (OH), 1613, 1515, and 1439 (C=C aromatic), 1225 and 1168 (C-O oxyaryl), and 827 (*p*-disubstituted benzene) cm^{-1} . The ^1H NMR spectrum showed six sets of doublet representing *ortho*-coupled aromatic protons at δ_{H} 7.03 ($J = 9.0$, H-2a/6a), 6.71 ($J = 9.0$, H-3a/5a), 7.22 ($J = 8.5$, H-2b/6b), 6.78 ($J = 8.5$, H-3b/5b), 7.05 ($J = 9.0$, H-2c/6c), and 6.79 ($J = 9.0$, H-3c/5c) indicating the presence of 1,4-disubstituted aromatic rings of A1, B1 and C1. Additionally, six sets of *meta*-coupled aromatic protons were seen at δ_{H} 6.24 ($J = 2.0$, H-12a), 6.71 ($J = 2.0$, H-14a), 6.24 ($J = 2.0$, H-12b), 6.60 ($J = 2.0$, H-14b), 6.23 ($J = 2.0$, H-12c), and 6.06 ($J = 2.0$, H-14c) which represents the 1,2,3,5-tetrasubstituted aromatic ring of A2, B2 and C2. A mutually coupled four doublet resonances and two singlet resonances at the aliphatic region displayed a methine group (C-H) at δ_{H} 5.95 ($J = 10.0$, H-7b), 4.71 ($J = 10.0$, H-8b), 4.91 ($J = 6.5$, H-7c), 4.62 ($J = 6.5$, H-8c), 6.07 (H-7a), and 3.95 (H-8a) which confirmed the occurrence of three units of a 1,2-dihydrobenzofuran ring in the structure.

The ^{13}C NMR-APT showed the resonance of 26 peaks representing 26 carbons in the compound. The downfield region of the NMR spectrum displayed an aliphatic carbon atom ranging from 46.9 - 95.6 ppm for six doublet resonances at C-7a/8a, C-7b/8b, and C-7c/8c. The chemical shifts of C-7a/7b/7c were more deshielded than C-8a/8b/8c due to the attachment with oxygen. The chemical shifts of a tertiary carbon C-8a/8b/bc and an olefinic carbon C-10a/10b/10c were used to deduce the presence of three units of 1,2-dihydrobenzofuran ring, which also led to the formation of a cyclononane ring. Also, the 12 doublet aromatic carbon atoms were shown, ranging from 96.2 - 128.7 ppm at C-2a/6a/3a/5a, C-2b/6b/3b/5b, and C-2c/6c/3c/5c. The upfield region shows 18 quaternary carbon atoms ranging from 118.8 - 181.8 ppm. Nine singlets of quaternary aromatic resonance are shown at C-1a/9a/10a, C-1b/9b/10b, and C-1c/9c/10c. Another

TABLE 1. ¹H NMR spectral data for **1-6** in acetone-*d*₆ measured at 500 and 600 MHz

Position	1	2	3	4	5	6
	δ_{H} (m, <i>J</i>)	δ_{H} (m, <i>J</i>)	δ_{H} (m, <i>J</i>)	δ_{H} (m, <i>J</i>)	δ_{H} (m, <i>J</i>)	δ_{H} (m, <i>J</i>)
1a	-	-	-	-	-	-
2a/6a	7.10 (<i>d</i> , 8.4)	7.11 (<i>d</i> , 8.4)	7.03 (<i>d</i> , 9.0)	7.32 (<i>d</i> , 8.5)	7.21 (<i>d</i> , 8.5)	7.22 (<i>d</i> , 8.4)
3a/5a	6.77 (<i>d</i> , 8.4)	6.76 (<i>d</i> , 8.4)	6.71 (<i>d</i> , 9.0)	6.75 (<i>d</i> , 8.5)	6.78 (<i>d</i> , 8.5)	6.80 (<i>d</i> , 8.4)
4a	-	-	-	-	-	-
7a	5.76 (<i>d</i> , 11.4)	4.26 (<i>br s</i>)	6.06 (<i>s</i>)	5.99 (<i>d</i> , 12.0)	6.06 (<i>d</i> , 3.0)	5.79 (<i>d</i> , 11.4)
8a	4.15 (<i>d</i> , 11.4)	4.13 (<i>br s</i>)	3.95 (<i>s</i>)	4.76 (<i>d</i> , 12.0)	4.39 (<i>d</i> , 3.0)	4.66 (<i>d</i> , 11.4)
9a	-	-	-	-	-	-
10a	-	6.11 (<i>d</i> , 2.4)	-	-	-	-
11a	-	-	-	-	-	-
12a	6.42 (<i>d</i> , 1.8)	6.13 (<i>t</i> , 2.4)	6.24 (<i>d</i> , 2.0)	6.29 (<i>d</i> , 2)	6.44 (<i>d</i> , 2.0)	6.29 (<i>d</i> , 2.4)
13a	-	-	-	-	-	-
14a	6.22 (<i>d</i> , 1.8)	6.11 (<i>d</i> , 2.4)	6.71 (<i>d</i> , 2.0)	6.69 (<i>d</i> , 2)	6.55 (<i>d</i> , 2.0)	6.12 (<i>d</i> , 2.4)
1b	-	-	-	-	-	-
2b/6b	6.88 (<i>d</i> , 8.4)	7.17 (<i>d</i> , 8.4)	7.22 (<i>d</i> , 8.5)	7.19 (<i>d</i> , 8.5)	7.04 (<i>d</i> , 8.5)	7.16 (<i>d</i> , 8.4)
3b/5b	6.62 (<i>d</i> , 8.4)	6.66 (<i>d</i> , 8.4)	6.78 (<i>d</i> , 8.5)	6.82 (<i>d</i> , 8.5)	6.60 (<i>d</i> , 8.5)	6.69 (<i>d</i> , 8.4)
4b	-	-	-	-	-	-
7b	5.43 (<i>d</i> , 4.8)	7.03 (<i>d</i> , 1.2)	5.95 (<i>d</i> , 10.0)	6.20 (<i>s</i>)	5.26 (<i>s</i>)	5.22 (<i>d</i> , 3.6)
8b	5.41 (<i>d</i> , 4.8)	-	4.71 (<i>d</i> , 10.0)	3.96 (<i>s</i>)	4.22 (<i>d</i> , 11.5)	3.14 (<i>m</i>)
9b	-	-	-	-	-	-
10b	-	-	-	-	-	-
11b	-	-	-	-	-	-
12b	6.14 (<i>d</i> , 1.8)	6.30 (<i>d</i> , 2.4)	6.24 (<i>d</i> , 2.0)	6.31 (<i>d</i> , 2.0)	6.01 (<i>s</i>)	6.06 (<i>s</i>)
13b	-	-	-	-	-	-
14b	6.59 (<i>d</i> , 1.8)	6.79 (<i>d</i> , 2.4)	6.60 (<i>d</i> , 2.0)	6.54 (<i>d</i> , 2.0)	-	-
1c	-	-	-	-	-	-
2c/6c	-	-	7.05 (<i>d</i> , 9.0)	7.27 (<i>d</i> , 8.5)	6.76 (<i>d</i> , 8.5)	6.42 (<i>d</i> , 8.4)
3c/5c	-	-	6.79 (<i>d</i> , 9.0)	6.77 (<i>d</i> , 8.5)	6.60 (<i>d</i> , 8.5)	6.50 (<i>d</i> , 8.4)
4c	-	-	-	-	-	-
7c	-	-	4.91 (<i>d</i> , 6.5)	5.56 (<i>d</i> , 2.0)	4.37 (<i>s</i>)	4.11 (<i>t</i> , 10.8)
8c	-	-	4.62 (<i>d</i> , 6.5)	4.68 (<i>d</i> , 2.0)	2.97 (overlap moisture peak)	4.57 (<i>d</i> , 10.8)
9c	-	-	-	-	-	-
10c	-	-	-	-	6.41 (<i>d</i> , 2.0)	-
11c	-	-	-	-	-	-
12c	-	-	6.23 (<i>d</i> , 2.0)	5.80 (<i>s</i>)	6.17 (<i>t</i> , 2.0)	6.20 (<i>d</i> , 1.8)
13c	-	-	-	-	-	-
14c	-	-	6.06 (<i>d</i> , 2.0)	-	6.41 (<i>d</i> , 2.0)	6.49 (<i>d</i> , 1.8)
1d	-	-	-	-	-	-
2d/6d	-	-	-	-	-	7.21 (<i>d</i> , 8.4)
3d/5d	-	-	-	-	-	6.79 (<i>d</i> , 8.4)
4d	-	-	-	-	-	-

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7d		5.39 (<i>d</i> , 5.4)
8d		4.69 (<i>d</i> , 5.4)
9d		-
10d		6.12 (<i>d</i> , 2.4)
11d		-
12d		6.30 (<i>t</i> , 2.4)
13d		-
14d		6.12 (<i>d</i> , 2.4)
OH-4a	8.52 (<i>br s</i>)	8.31 (<i>br s</i>)
OH-11a	-	8.30 (<i>br s</i>)
OH-13a	8.78 (<i>br s</i>)	8.07 (<i>br s</i>)
OH-4b	8.71 (<i>br s</i>)	7.93 (<i>br s</i>)
OH-13b	8.62 (<i>br s</i>)	6.56 (<i>br s</i>)
OH-4c	8.44 (<i>br s</i>)	7.99 (<i>br s</i>)
OH-11c	-	7.96 (<i>br s</i>)
OH-13c	-	7.96 (<i>br s</i>)

nine singlets of quaternary aromatic bearing oxygen resonance were shown at C-4a/11a/13a, C-4b/11b/13b and C-4c/11c/13c.

The spectroscopic data (Tables 1 and 2) obtained are identical to those reported by Ito et al. (2000) in the literature, elucidated as α -viniferin (**3**), a trimer stilbenoid. Numerous *Shorea* species have been reported on the occurrence of this compound (Moriyama et al. 2016; Zawawi et al. 2013). Interestingly, two isomers, (+)- α -viniferin and (-)- α -viniferin, were discovered to differ only in their optical rotation. Ito et al. in 2000 reported on the first (+)- α -viniferin bearing sugar moiety (glucopyranoside) from the bark of *S. hemsleyana* (Muharini et al. 2001).

Compound **4** (Figure 1) was isolated as a yellow amorphous powder. The ^1H NMR spectrum showed six *ortho*-coupled doublet resonances at δ_{H} 7.32 ($J = 8.5$, H-2a/6a), 6.75 ($J = 8.5$, H-3a/5a), 7.19 ($J = 8.5$, H-2b/6b), 6.82 ($J = 8.5$, H-3b/5b), 7.27 ($J = 8.5$, H-2c/6c), 6.77 ($J = 8.5$, H-3c/5c) representing 1,4-disubstituted aromatic rings for A1, B1 and C1. The proton spectra also show four doublet *meta* resonances at δ_{H} 6.29 ($J = 2.0$, H-12a), 6.69 ($J = 2.0$, H-14a), 6.31 ($J = 2.0$, H-12b) and 6.54 ($J = 2.0$, H-14b). The presence of mutually coupled aliphatic protons, four doublet resonances at δ_{H} 5.99 ($J = 12.0$, H-7a), 4.76 ($J = 12.0$, H-8a), 5.56 ($J = 2.0$, H-7c), 4.68 ($J = 2.0$, H-8c), and two singlet resonances at δ_{H} 6.20 (H-7b), 3.96 (H-8b) suggested there are three units of 1,2-dihydrobenzofuran ring in the structure. Compounds **3** and **4** demonstrated a similar pattern of protons spectra except for the chemical shift in **4** that was more in the deshielded region, the disappearance of two doublet *meta* resonances and one extra singlet resonance appeared at the aliphatic region, δ_{H} 5.80 (H-12c), suggesting a difference splitting pattern in

compound **4** that does not disturb the furan connected ring. The HMBC correlation shows that resonance at δ_{H} 5.80 (s, H-12c) is supposed to be an olefinic proton, a doublet *meta* resonance correlated with the quaternary-ketone resonance at δ_{C} 180.1 (C-14c) and 177.4 (C-13c). This observation demonstrated that the 1,2,3,5-tetrasubstituted phenyl group (ring C2) is oxidised to an unusual 3,4,5-trisubstituted *ortho*-quinone moiety.

The ^{13}C NMR-APT showed the same resonance distribution as **3**, which consists of three set stilbenes radicals connected through three units of the 1,2-dihydrobenzofuran ring with cyclononane ring. A vast difference is shown at the ring C2 of **4**, where the chemical shift is displayed at the most deshielded region for two ketone resonances of C-14c and C-13c supporting the *ortho*-quinone moiety.

The spectroscopic data (Tables 1 and 2) are similar to the previous report of the compound isolated from the bark of *Hopea exalata* (Ge et al. 2006). Hence, compound **4** is elucidated as hopeanolin, another trimer resveratrol. The study by Ge et al. in 2006 also suggested that biosynthetically, hopeanolin may originate from a precursor, miyabenol C, a metabolite of *Dipterocarpus grandiflorus* (Dipterocarpaceae), via a set of sequential bioreactions. Later, in 2014, this compound was first isolated from *Shorea* species, which is the twigs of *S. acuminata*, by Muhammad et al. (2014).

Compound **5** (Figure 1) was isolated as a colourless powder. The UV absorption (MeOH) at λ_{max} 232, 285 and 330 nm indicated the presence of a substituted aromatic system in the structure and was supported by the IR absorption at ν_{max} 3330 (OH), 2922 (C-H aliphatic), 1613, 1513, and 1448 (C=C aromatic), 1227 and 1169 (C-O oxyaryl) and 835 (*p*-disubstituted benzene) cm^{-1} . The

TABLE 2. ^{13}C -APT NMR spectral data for **1-6** in acetone- d_6 measured at 125 MHz

Position	1	2	3	4	5	6
	δ_{C}	δ_{C}	δ_{C}	δ_{C}	δ_{C}	δ_{C}
1a	132.3	137.3	131.5	130.6	134.4	130.8
2a/6a	129.6	128.8	127.4	128.9	128.1	130.2
3a/5a	115.8	116.3	111.5	116.1	116.0	115.4
4a	157.0	156.7	157.3	158.6	155.8	158.6
7a	88.1	59.5	85.9	90.0	85.8	90.5
8a	49.2	58.7	45.9	52.8	50.4	48.9
9a	142.5	149.3	139.2	137.6	147.1	141.8
10a	118.3	106.4	120.4	118.0	118.1	124.5
11a	155.6	159.3	158.8	162.8	157.9	155.7
12a	101.3	101.3	96.0	97.6	101.3	101.6
13a	159.6	159.3	158.9	161.6	156.8	156.8
14a	105.2	106.4	105.7	106.2	104.0	105.8
1b	130.6	129.7	132.0	134.6	137.5	133.5
2b/6b	128.4	131.0	127.7	129.2	129.6	130.7
3b/5b	115.2	116.0	115.5	116.4	115.4	115.5
4b	158.3	157.3	157.7	159.0	158.1	155.9
7b	43.5	122.6	89.5	88.9	36.5	37.1
8b	70.9	143.1	52.3	44.7	51.4	53.2
9b	140.3	147.5	138.2	139.8	143.1	143.3
10b	118.2	123.8	119.2	122.1	119.2	115.9
11b	159.6	156.1	161.2	161.0	159.4	158.8
12b	96.7	103.8	96.4	97.8	96.0	96.5
13b	158.7	159.7	160.3	159.9	154.7	155.0
14b	110.3	98.4	105.3	106.5	122.2	122.2
1c			131.8	133.8	134.1	131.4
2c/6c			128.1	128.0	129.9	129.3
3c/5c			115.5	115.9	115.6	116.0
4c			157.8	158.2	156.6	156.4
7c			95.1	91.2	56.2	57.6
8c			55.2	50.2	67.5	49.2
9c			140.7	129.5	143.9	141.7
10c			118.3	148.1	108.3	123.5
11c			161.1	171.4	158.9	161.7
12c			97.5	101.7	102.0	95.6
13c			160.2	177.4	158.9	159.4
14c			108.0	180.4	108.3	107.0
1d						134.7
2d/6d						128.3
3d/5d						116.1
4d						158.0

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7d	94.7
8d	57.6
9d	148.1
10/14d	107.6
11/13d	159.9
12d	102.2

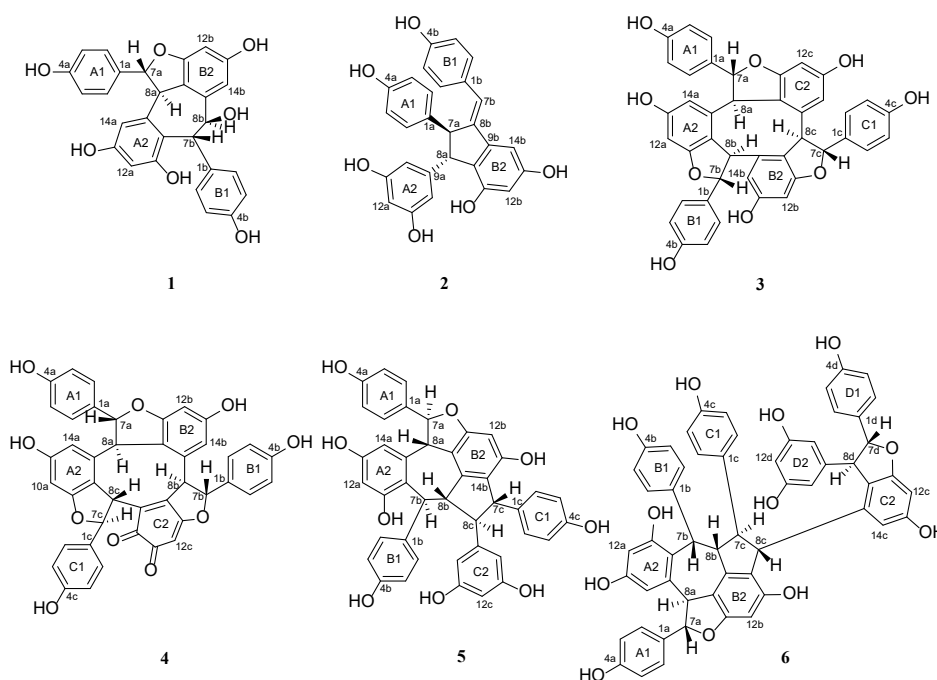


FIGURE 1. Structures of compounds 1 – 6

^1H NMR spectrum showed six sets of *ortho*-coupled doublet signals at δ_{H} 7.21 ($J = 8.5$, H-2a/6a), 6.78 ($J = 8.5$, H-3a/5a), 7.04 ($J = 8.5$, H-2b/6b), 6.60 ($J = 8.5$, H-3b/5b), 6.76 ($J = 8.5$, H-2c/6c) and 6.60 ($J = 8.5$, H-3c/5c) represent the 1,4-disubstituted phenyl rings for A1, B1, C1. The splitting pattern for doublet *meta* position is different in compound **5** whereby one singlet resonance at aromatic region δ_{H} 6.01 (H-12b) representing proton at ring B2, four sets of doublet *meta* resonances are shown with two doublet resonances at δ_{H} 6.44 ($J = 2.0$, H-12a), 6.55 ($J = 2.0$, H-14a) represent ring A2. The spectra displayed an overlapping resonance with the integration of two was shown at δ_{H} 6.41 ($J = 2.0$, H-10c and H-14c) representing aromatic proton at *meta* position for ring C2. Other triplet resonance at δ_{H} 6.17 ($J = 2.0$, H-12c) suggested that the protons (H-10c, H-14c, H-12c) are in a symmetrical *meta* position. The methine protons at H-7 and H-8 also differ from compounds **3** and **4**, with a doublet resonance at δ_{H} 6.06 ($J = 3.0$, H-7a) and 4.39 ($J = 3.0$, H-8a), respectively, suggesting only one unit of 1,2-dihydrobenzofuran connected in the structure. The

aliphatic region shows shielded resonance at δ_{H} 5.26 (H-7b), 4.22 ($J = 11.5$, H-8b), 4.37 (H-7c), 2.97 (overlapped with moisture peak, H-8c), proposing the trimer are connected through oxidative coupling between radicals at C-8b and C-8c. The position of rings C1 and C2 can be connected to the dibenzobicyclo[5.3.0]decadiene ring system between the C-8b and C-8c.

As for the ^{13}C NMR-APT of compound **5**, the carbon resonance distribution differs from **3** and **4**. A shielded resonance was shown at the downfield region of the NMR spectrum for an aliphatic carbon atom ranging from 36.5 – 85.8 ppm for six signals at C-7a/8a, C-7b/8b, and C-7c/8c. For compound **4**, only resonance C-7a is shown to be more deshielded due to the attachment with oxygen. This suggested that the chemical shifts of a tertiary carbon C-8a/7b/8b and an olefinic carbon C-10a/10b comprehend the presence of one unit of the 1,2-dihydrobenzofuran ring, which also led to the formation of a cycloheptane ring. As for the tertiary carbon C-7c/8c, the chemical shift suggested that ring C was connected through a

cyclopentane structure. Also, there were 18 aromatic carbon atoms shown, ranging from 96.0 – 129.9 ppm which represent C-2a/6a/3a/5a/12a/14a, C-2b/6b/3b/5b/12b and C-2c/6c/3c/5c/10c/12c/14c. The upfield region shows 16 quaternary carbon atoms ranging from 108.3 – 158.9 ppm. Eight quaternary aromatic resonances are shown at C-1a/9a/10a, C-1b/9b/10b, and C-1c/9c. Another eight signals of quaternary aromatic bearing oxygen resonance were shown at C-4a/11a/13a, C-4b/11b and C-4c/11c/13c.

From the spectroscopic data (Tables 1 and 2), compound **5** is elucidated as a trimer stilbenoid with one unit of 1,2-dihydrobenzofuran ring. However, four other compounds have been found to have the same splitting pattern as compound **5**: the isomer of davidiol A, vaticanol A, pauciflorol A and B with differences in α and β relative configuration at H-7 and H-8. By comparing the α and β relative configuration, the data is identical to davidiol A from *Sophora davidii* by Tanaka et al. in 2000. Interestingly, these compounds have been isolated a few times from *Shorea* species, such as *S. parviflora* (Rosyidah et al. 2005), *S. rugosa* (Haryoto et al. 2008), *S. macroptera* (Nazri et al. 2012), and *S. acuminatissima* (Haryoto et al. 2014).

Compound **6** was obtained as a brown amorphous powder. The UV absorption (MeOH) at λ_{\max} 230 and 285 nm indicated the presence of a substituted aromatic system in the structure and was supported by the IR absorption at ν_{\max} 3285 (OH), 2925 (C–H aliphatic), 2855 (C–H aldehyde), 1712 (C=O aldehyde) and 1420 (C=C aromatic) cm^{-1} . ^1H NMR spectral data showed eight sets of *ortho*-coupled aromatic proton signals at δ_{H} 7.22 ($J = 8.4$, H-2/6a)/6.78 ($J = 8.4$, H-3/5a), 7.16 ($J = 8.4$, H-2/6b)/6.69 ($J = 8.4$, H-3/5b), 6.50 ($J = 8.4$, H-3/5c)/6.42 ($J = 8.4$, H-2/6c), and 7.18 ($J = 8.4$, H-2/6d)/6.77 ($J = 8.4$, H-3/5d), which represent four units of *p*-hydroxybenzene rings. Two sets of aromatic proton signals at δ_{H} 6.13/6.30, for one unit of 1,3,5-trisubstituted benzene (ring D2), four sets of aromatic proton signals at δ_{H} 6.12/6.29 and 6.20/6.49, for two units of tetrasubstituted benzene (ring A2 and C2), and a singlet aromatic proton signal at

δ_{H} 6.06 for a unit of trisubstituted-3,5-dihydroxybenzene rings (B2) are shown, respectively. Four sets of aliphatic protons were detected at δ_{H} 4.46/5.79 and 4.69/5.39, which correspond to two units of 1,2-dihydrobenzofuran rings. The remaining signals at δ_{H} 3.14 (m), 4.11 (t, $J = 10.8$ Hz), 4.57 (d, $J = 10.8$ Hz), and 5.22 (d, $J = 3.6$ Hz) are aliphatic protons of the adjacent CH corresponds to a CH-CH-CH-CH unit. The unit structures above validated the structure of **6**, which is a tetramer stilbenoid with an extra cyclic besides the benzofuran ring.

The ^{13}C NMR-APT showed a resonance of 46 peaks, signifying the compound's 56 carbons. The signals resonated at δ_{C} 130.2, 115.4, 130.7, 115.5, 129.3, 16.0, 128.3, 116.1, 96.5, 105.8, 96.5, 95.6, 107.0, 107.6, and 102.2 belong to aromatic C-H of ring A1, A2, B1, B2, C1, C2, D1 and D2. On the other hand, the aliphatic C-H of furan's signals appeared at δ_{C} 90.5 (C-7a), 48.9 (C-8a), 94.7 (C-7d), 57.6 (C-8d). The chemical shifts for the rest of aliphatic carbon were seen at δ_{C} 37.1 (C-7b), 53.2 (C-8b), 57.6 (C-7c) and 49.2 (C-8c). The aromatic quaternary carbon showed signals in the upfield region ranging from 115.9 to 148.1 ppm. Meanwhile, 11 signals recorded in the most deshielded area are the signals of oxyaryl carbons ranging from 155.0 to 161.7 ppm.

The combination of a unit of 1,2-dihydrobenzofuran ring, mono- and disubstituted-3,5-dihydroxybenzene rings were comparable to that of the trimer vaticanol A (Tanaka et al. 2000) for bicyclo ring (A4/C3) that made from the arrangement of CH-CH-CH-CH unit, additionally from the substitution of A3 and C3-B1/C1 rings. However, the presence of an extra stilbene unit D was determined to be connected to the C2 ring via the dihydrofuran ring, resulting in compound **6** being a tetramer stilbenoid. The comparison of the ^1H and ^{13}C NMR spectrum data showed that only the spectral data of vaticanol B (Aoki-Utsubo et al. 2023) were highly comparable to the spectral data of **6** (Tables 1 & 2). Thus, chemical **6** was identified as the tetramer stilbenoid vaticanol B (Figure 1).

As for the phenolic compounds, compound **7** (Figure 2) was isolated as a white amorphous powder. The UV absorption (MeOH) at λ_{\max} 230 and 282 nm indicated

TABLE 3. ^1H and ^{13}C -APT NMR spectral data for **7-8** in acetone- d_6 measured at 500 MHz and 125 MHz, respectively

Position	7		Position	8	
	δ_{H} (multiplicity, J)	δ_{C}		δ_{H} (multiplicity, J)	δ_{C}
1	-	163.6	1	7.30 (<i>d</i> , 2.5)	101.4
2	7.01 (<i>dd</i> , 8.5, 1.5)	116.0	2	-	139.7
3	7.80 (<i>dd</i> , 8.5, 1.5)	132.5	3	6.66 (<i>d</i> , 2.5)	110.4
4	-	-	4	-	119.3
5	7.80 (<i>dd</i> , 8.5, 1.5)	128.7	5	-	151.7
6	7.01 (<i>dd</i> , 8.5, 1.5)	116.0	6	-	142.9
7-CHO	9.85	191.2	7-COOH	12.82	168.0

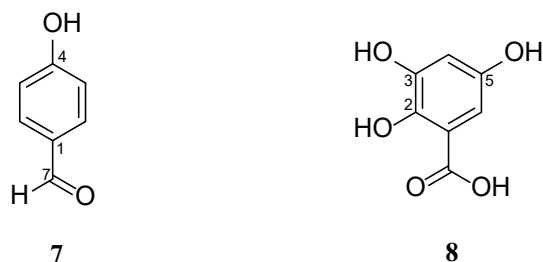


FIGURE 2. Structures of compounds 7–8

the presence of a substituted aromatic system in the structure. The IR absorption at ν_{\max} 3366 (OH), 2922 (C–H aliphatic), 1602, 1516 and 1449 (C=C aromatic), 1249 and 1175 (C–O oxyaryl), and 835 (*p*-disubstituted benzene) cm^{-1} . The ^1H NMR spectrum shows two doublets of doublet resonance at δ_{H} 7.80 (H-3/H-5) and δ_{H} 7.01 (H-2/H-6) and one aldehyde resonance at a deshielded region δ_{H} 9.85. The ^{13}C NMR-APT displayed the aldehyde resonance at δ_{C} 191.2, and the C–OH were shown at δ_{C} 163.6. The aromatic carbon resonated at δ_{C} 132.5 for C-3 and δ_{C} 128.7 for C-5, while C-2 and C-6 were shown at δ_{C} 116.0. The data (Table 3) is identical to *p*-hydroxybenzaldehyde as reported from *Sorghum bicolor* seed by Woodhead, Galeffi and Bettolo in 1982 (Jiang et al. 2022; Woodhead, Galeffi & Bettolo 1982).

Lastly, compound **8** (Figure 2) was also isolated as a white amorphous powder. The ^1H NMR spectrum shows two doublet *meta* resonances at δ_{H} 7.32 (H-1) and 6.66 (H-3). The resonance at δ_{H} 12.41 suggested a carboxylic acid substituent attachment in the structure. The ^{13}C NMR-APT shows a resonance at the deshielded region δ_{C} 168 (C-7), suggesting the carboxylic acid attachment. One quaternary carbon is shown at δ_{C} 119.3 (C-4) at the upfield region on the spectrum. Three C–O resonances are shown at C-2/C-5 and C-6, and the aromatic carbon is displayed at C-1 and C-3. The data (Table 3) is identical to 2,3,5-trihydroxybenzoic acid isolated from *Iris pseudacorus* roots in 2015 (Tarbeeva et al. 2015).

CONCLUSION

Six known oligostilbenoids consist of two dimers, ampelopsins A (**1**) and D (**2**), three trimer with distinct characteristics namely, α -viniferin (**3**), hopeanolin (**4**), and davidiol A (**5**), and one tetramer known as vaticanol B (**6**), as well as two phenolic compounds elucidated as *p*-hydroxybenzaldehyde (**7**) and 2,3,5-trihydroxybenzoic acid (**8**), were successfully isolated from *S. materialis* stem bark. It is essential to highlight that these constituents are the first to be discovered in this species. To the best of our knowledge, this is the first report of the occurrence of compounds **7** and **8** in the family of Dipterocarpaceae and compound **2** in the genus of *Shorea*.

ACKNOWLEDGEMENTS

We are deeply grateful to Universiti Teknologi MARA, Malaysia, for the financial support provided by the *Geran Penyelidikan Fakulti UiTM Cawangan Selangor (DUCS-FAKULTI)* [600-UiTMSEL (PI. 5/4) (127/2022)], Faculty of Applied Sciences and Atta-ur-Rahman Institute for Natural Product Discovery, Universiti Teknologi MARA for providing the research facilities. Additionally, we extend our gratitude to Mr. Nik Hazlan Nik Hashim, Mr. Khairilnuar Zainol and Mr. Akso Rosli for their assistance in the plant collection.

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