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Research Article

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New pyranoxanthenes from the stem bark of *Calophyllum soulattri* Burm. F.

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Abstract: Five new pyranoxanthenes (**1-5**), together with eight known xanthenes were isolated from hexane extract of *C. soulattri* stem bark. Among the new compounds, xanthenes **1-3** are structural isomers. The structures of compounds **1-13** were determined by 1D and 2D NMR analysis.

Keywords: Calophyllaceae, *Calophyllum soulattri*, hexane extract, xanthone.

1. Introduction

One of the largest genera from the family Calophyllaceae is *Calophyllum*. The scientific studies of the genus *Calophyllum* revealed that it is a rich source of bioactive secondary metabolites (Gupta & Gupta, 2020). In this response, we will focus on *C. soulattri*, a flowering plant species which is native to Vietnam, Thailand, Singapore, Philippines, Papua New Guinea, Malaysia, Indonesia, India, Cambodia, Brunei, and Australia (Northern Territory). The plant of this species has been traditionally used as a tonic for horses and treatment for rheumatic pain (Khan et al., 2002). The stem bark of *C. soulattri* has been reported to contain several phytochemicals such as pyranoxanthone, furanoxanthone, and coumarin (Ee et al., 2011). In addition, the stem bark of *C. soulattri* has been studied for its potential biological activities such as cytotoxic, anti-inflammatory, and antimicrobial activities (Chinthu et al., 2023). Moreover, *C. soulattri* species may be a promising source of new bioactive compounds, considering the rising number of naturally occurring compounds being discovered. For example, a new pyranocoumarin, soulamarin (Ee et al., 2011); a new diprenylated xanthone, phylattrin (Mah et al., 2012); a new pyranoxanthone, soulattrin (Mah et al., 2011); and two new isoprenylated benzofuran-3-ones, airlanggins A and B (Tanjung et al., 2018).

The present work on the stem bark of *C. soulattri* Burm. F. collected from Sarawak, Malaysia focused on the phytochemical study which has led to the discovery of five new pyranoxanthenes, **1-5** along with eight previously known xanthenes **6-13** (Figure 1). The structural

characterization of compounds **1-13** was achieved by 1D and 2D NMR analyses.

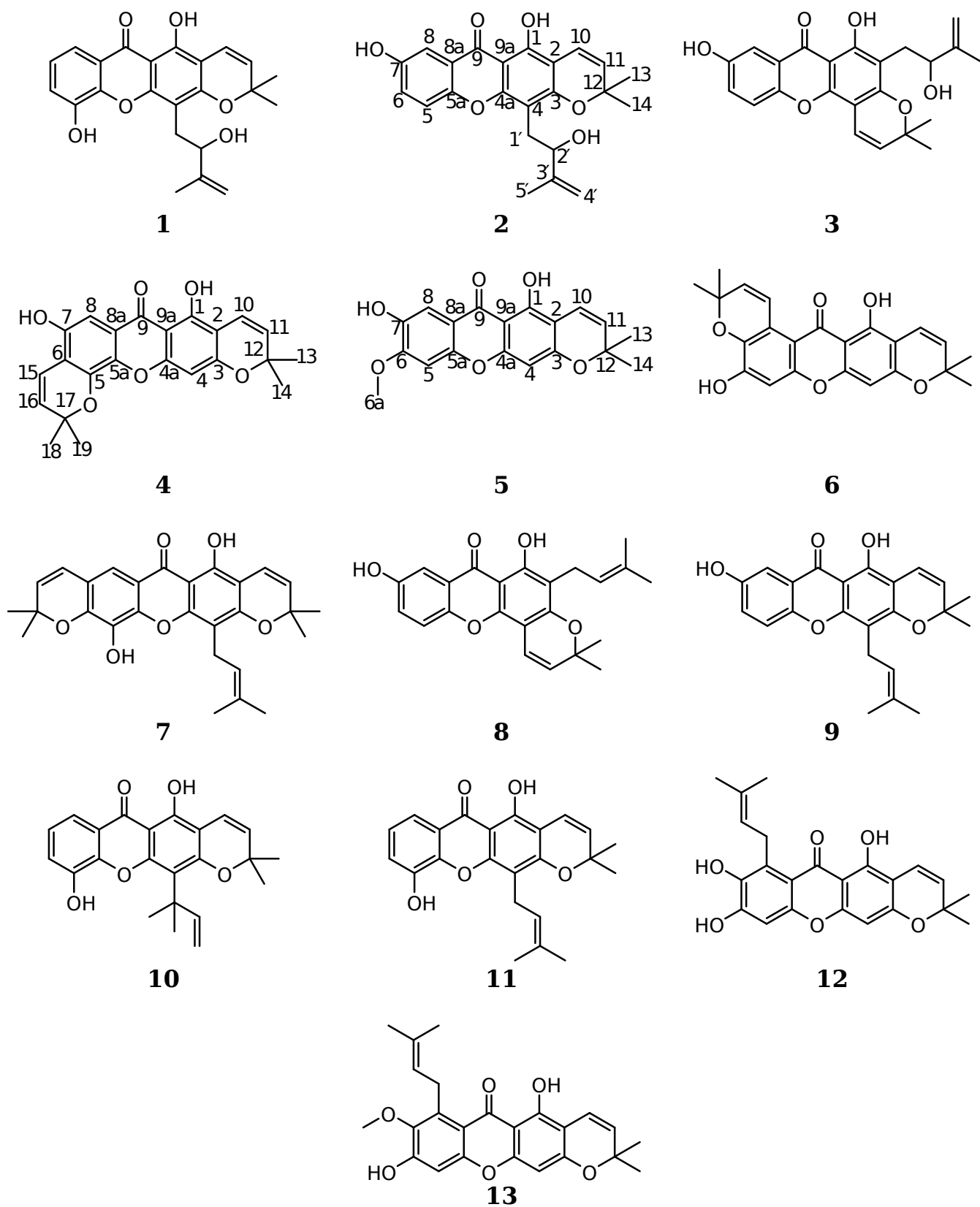


Figure 1. Structure of compounds 1-13.

2. Result and Discussion

Soulaxanthone B (**2**) was isolated as a yellow needle crystal. The proton NMR spectrum exhibited two deshielded singlet peaks resonating at δ H 13.37 and δ H 8.93 indicating the presence of two hydroxyl groups. Doublet resonances observed at δ H 5.77 (10.0 Hz, 1H) and δ H 6.73 (10.0 Hz, 1H) along with two set of singlets integrating for three protons each (δ H 1.53 and 1.54) formed the 2,2-dimethylpyrano system. The signals of methyl group (δ H 1.90, *s*, 3H), olefinic protons of terminal double bond (δ H 4.64, and 4.72), hydroxy methine proton (δ H 4.43, *dd*, 6.5, 7.0) and a methylene proton (δ H 3.05, *dd*, 7.0, 13.1, 1H; δ H 3.09, *dd*, 6.5, 13.1, 1H) established a 2-hydroxy-3-methylbut-3-en-1-yl substituent. The presence of the aromatic protons at δ H 7.39 (H-6), 7.53 (H-5) and 7.58 (H-8) were indicative of the xanthone scaffold. The COSY experiment further confirms the presence of benzene ring when it displayed the *ortho*-coupling of H-6 with H-5, and *meta*-coupling of H-6 with H-8 which gave the *J* coupling constant of 9.0 Hz and 3.0 Hz respectively. The carbon NMR spectrum exhibited 23 carbon peaks which established C₂₃H₂₂O₆, and DEPT (45, 90, 135) experiments have concluded three methyls, two methylene, six methines, and twelve quaternary carbons. The most shifted carbon resonance at δ C 181.9 was identified as the carbonyl. The assignment of the structure was supported with the 2D NMR (HSQC, HMBC and COSY). HMBC correlations between OH-1 (δ H 13.37) to C-1, C-2 and C-9a determined the position of hydroxyl group at C-1. The COSY spectrum demonstrated the *ortho*-coupling of H-10 with H-11 which evident the presence of a pyrano ring in the system. The H-10 proton, resonating at δ H 6.73 showed heteronuclear connectivity with C-1, C-2 and C-3 which established the pyrano ring was fused at C-2 and C-3 of the xanthone scaffold. The absence ¹*J* correlation of a doublet resonating at δ H 3.86 in HSQC spectrum suggested a hydroxyl group which must be positioned at C-2' (δ C 75.6). This was further supported by DEPT experiments showing C-2' is a methine carbon. The cross-peaks of methylene protons H-1' resonating at δ H 3.05 and 3.09, with C-3, C-4 and C-4a concluded the 2-hydroxy-3-methylbut-3-en-1-yl was substituted at quaternary carbon C-4 (δ C 105.7). The OH-7 (δ H 8.93) exhibited the HMBC interactions with C-6, C-7 and C-8 indicated the positioning of a hydroxyl group at aromatic carbon C-7.

Soulaxanthone A (**1**) and soulaxanthone C (**3**) are structural isomers to compound **2** and obtained as a yellow solid with a molecular formula C₂₃H₂₂O₆. They exhibited similar proton and carbon resonances with significantly shifted chemical shifts. In comparison to compound **2**, the 5-OH in compound **1** was confirmed to be bonded to C-5 by comparison of the xanthone skeleton at C-5, C-6, C-7, and C-8 with that of trapezifolixanthone skeleton (Lizazman et al., 2022). On the other hand, compound **3** gave the cross-peaks of H-10 with C-3 and H-11 with C-4 in

the HMBC spectrum indicating the pyrano ring was fused to the xanthone skeleton at C-4 and C-3 through oxygen. The 2-hydroxy-3-methylbut-3-en-1-yl substituent of compound **3** was assigned to position C-2 based on HMBC correlations between the methylene proton H-1' (δ H 2.88 and 2.96) with C-1 (δ C 161.9), C-2 (δ C 109.5), and C-3 (δ C 159.7).

7-Hydroxyvenuloxanthone (**4**) was isolated as a yellow needle crystal. The 1 H NMR spectrum showed the presence of the proton signals of two singlets in the aromatic region at δ H 6.42 and 7.45 (each 1H) which suggests that compound **4** is a typical for a xanthone with six substituents. The presence of a chelated hydroxyl group at δ H 13.52 and a lone hydroxyl group at δ H 8.34 are assignable to the signals of 1-OH and 7-OH respectively. The proton NMR also revealed the presence of two sets of 2,2-dimethylpyrano group, of which one of the groups was observed at δ H 6.95 (*d*, 10.1 Hz, 1H), δ H 5.95 (*d*, 10.1 Hz, 1H), and δ H 1.53 (*s*, 6H). Another ring was indicated by two doublets at δ H 6.69 and 5.74 with $J = 10.2$ Hz, along with a singlet resonance integrating for six protons (δ H 1.47). This suggestion was further confirmed by COSY which exhibited the couplings of the doublets' protons with each other (H-10/H-11 and H-15/H-16). The 13 C NMR spectrum of **4** showed 23 carbon signals in accordance with molecular formula C₂₃H₂₀O₆. Meanwhile, the DEPT experiments (DEPT 45, 90, 135) gave four methyls, six methines and thirteen quaternary carbons including a carbonyl carbon resonating at δ C 180.8. The HMBC correlations of 1-OH with three quaternary carbons (δ C 103.7, 105.1, 158.4) showing that the chelated hydroxyl group was bonded to C-1. The carbons resonating at δ C 110.5, 148.3, and 146.9 showed cross-peaks with a proton (δ H 6.69) belongs to one of the pyrano ring showed that the group was fused at C-5 and C-6. Moreover, the doublets at δ H 5.95 and 6.95 gave HMBC correlations with C-2 which indicates the attachment of another set of pyrano ring to C-2 and C-3.

Soulaxanthone D (**5**) was obtained in the form of pale-yellow needle crystal. Analysis of the 1 H NMR spectrum revealed a chelated hydroxyl group (δ H 13.57) and three aromatic protons (δ H 6.33, 6.95, and 7.55). The presence of two *ortho*-coupled doublets at δ H 5.74 and 6.68, along with a singlet integrating for six protons at δ H 1.47, indicated the existence of 2,2-dimethyl-2*H*-pyrano ring. Additionally, a singlet at δ H 3.99 integrating for three protons confirmed the presence of methoxyl group in compound. Further analysis using 13 C NMR and DEPT 45 experiments revealed 23 carbon signals comprising five methines, three methyls, and eleven quaternary carbons, including a carbonyl that resonates at δ C 180.6, consistent with molecular formula C₁₉H₁₆O₆. In the HMBC spectrum, cross peaks between the chelated hydroxyl group and three aromatic carbons at δ C 103.7, 105.1, and 158.3 indicated its position at C-1 (δ C 158.9). Correlations between δ H 3.99 (H-6a) with δ C 146.8 (C-6) confirmed the methoxyl moiety at carbon 6. The aromatic proton H-4 resonating at δ H 6.33 showed correlations with δ C 103.7 (C-9a), δ C 158.0

(C-4a), δ C 160.9 (C-3), and δ C 105.1 (C-2). Meanwhile, aromatic proton H-5 (δ H 6.95) gave cross peaks with δ C 155.4 (C-7), δ C 146.8 (C-6), δ C 153.4 (C-5a), δ C 113.4 (C-8a), confirming protons H-4 and H-5 belong to different parts of the ring system. On the other hand, the remaining aromatic proton resonating δ H 7.55 (H-8) appears as a singlet suggesting it is in *para*-position relative to proton H-5, which also appears as a singlet. This was confirmed by the 2J - 3J HMBC correlations of H-8 (δ H 7.55) with δ C 153.4 (C-5a), 155.4 (C-7), and 180.6 (C-9). Moreover, the olefinic protons δ H 6.68 (H-10) was correlated with C-1 and C-3, and δ H 5.74 (H-11) gave cross peak with δ C 105.1 (C-2) indicating the pyrano ring was fused with the xanthone skeleton at C-2 and C-3.

The COSY and long range HMBC correlations are consistent with compounds **1-5** (Figure 2). The 1 H and 13 C NMR data for compounds **1-5** are reported herein for the first time (Table 1).

Table 1. ¹H (400 MHz) and ¹³C (100 MHz) NMR data of compounds **1-5** in acetone-*d*₆, δ in ppm

No	1		2		3		4		5	
	¹ H (Mult, <i>J</i> Hz, int)	¹³ C	¹ H (Mult, <i>J</i> Hz, int)	¹³ C	¹ H (Mult, <i>J</i> Hz, int)	¹³ C	¹ H (Mult, <i>J</i> Hz, int)	¹³ C	¹ H (Mult, <i>J</i> Hz, int)	¹³ C
1	13.33 (<i>s</i> , OH)	156.9	13.37 (<i>s</i> , OH)	156.8	13.47 (<i>s</i> , OH)	161.9	13.52 (<i>s</i> , OH)	158.4	13.57 (<i>s</i> , -OH)	158.3
2		104.9		104.6		109.5		105.1		105.1
3		159.6		159.4		159.7		161.0		160.9
4		106.3		105.7		101.2	6.42 (<i>s</i> , 1H)	95.5	6.33 (<i>s</i> , 1H)	95.4
4a		155.9		156.2		151.3		157.8		158.0
5	9.25 (<i>s</i> , OH)	147.2	7.53 (<i>d</i> , 9.0, 1H)	119.9	7.55 (<i>d</i> , 9.0, 1H)	119.9		146.9	6.95 (<i>s</i> , 1H)	103.7
5a		146.3		150.8		150.7		144.4		153.4
6	7.37 (<i>dd</i> , 1.5, 7.9, 1H)	121.5	7.39 (<i>dd</i> , 3.0, 9.0, 1H)	125.3	7.38 (<i>dd</i> , 3.0, 9.0, 1H)	125.3		110.5		146.8
6a									3.99 (<i>s</i> , -OCH ₃)	56.6
7	7.27 (<i>t</i> , 7.9, 1H)	124.9	8.93 (<i>s</i> , OH)	154.9	8.98 (<i>s</i> , OH)	154.9	8.34 (<i>s</i> , OH)	148.3		155.4
8	7.68 (<i>dd</i> , 1.3, 7.9, 1H)	116.3	7.58 (<i>d</i> , 3.0, 1H)	109.2	7.59 (<i>d</i> , 3.0, 1H)	109.3	7.45 (<i>s</i> , 1H)	109.3	7.55 (<i>s</i> , 1H)	105.5
8a		122.1		121.6		121.8		114.4		113.4
9		182.2		181.9		181.6		180.8		180.6
9a		103.9		103.7		103.5		103.7		103.7
10	6.72 (<i>d</i> , 10.0, 1H)	116.0	6.73 (<i>d</i> , 10.0, 1H)	116.1	6.90 (<i>d</i> , 10.0, 1H)	115.8	6.95 (<i>d</i> , 10.1, 1H)	115.7	6.68 (<i>d</i> , 10.2, 1H)	115.8
11	5.77 (<i>d</i> , 10.0, 1H)	128.6	5.77 (<i>d</i> , 10.0, 1H)	128.4	5.76 (<i>d</i> , 10.0, 1H)	127.8	5.95 (<i>d</i> , 10.1, 1H)	131.6	5.74 (<i>d</i> , 10.2, 1H)	128.8
12		79.3		79.2		79.3		79.4		78.9
13	1.52 (<i>s</i> , 3H)	28.7	1.54 (<i>s</i> , 3H)	28.6	1.52 (<i>s</i> , 3H)	28.5	1.53 (<i>s</i> , 3H)	28.1	1.47 (<i>s</i> , 3H)	28.5
14	1.53 (<i>s</i> , 3H)	28.7	1.53 (<i>s</i> , 3H)	28.6	1.52 (<i>s</i> , 3H)	28.5	1.53 (<i>s</i> , 3H)	28.1	1.47 (<i>s</i> , 3H)	28.5

1'	3.10 (<i>m</i> , 1H)	29.9	3.05 (<i>m</i> , 1H)	30.0	2.88 (<i>m</i> , 1H)	29.7		
	3.17 (<i>m</i> , 1H)		3.09 (<i>m</i> , 1H)		2.96 (<i>m</i> , 1H)			
2'	4.02 (<i>d</i> , OH)	75.6	3.86 (<i>d</i> , OH)	75.6	3.68 (<i>d</i> , OH)	75.3		
	4.52 (<i>m</i> , 1H)		4.43 (<i>m</i> , 1H)		4.42 (<i>m</i> , 1H)			
3'		149.		149.		149.		
		2		0		2		
4'	4.65 (<i>s</i> , 1H)	110.	4.64 (<i>s</i> , 1H)	110.	4.66 (<i>s</i> , 1H)	110.		
	4.78 (<i>s</i> , 1H)	2	4.72 (<i>s</i> , 1H)	4	4.75 (<i>s</i> , 1H)	3		
5'	1.88 (<i>s</i> , 3H)	18.0	1.90 (<i>s</i> , 3H)	17.8	1.85 (<i>s</i> , 3H)	17.8		
15							6.69 (<i>d</i> , 10.2,	115.
							1H)	8
16							5.74 (<i>d</i> , 10.2,	128.
							1H)	7
17								78.9
18							1.47 (<i>s</i> , 3H)	28.5
19							1.47 (<i>s</i> , 3H)	28.5

Brasilixanthone B (6): yellow needle crystal. $^1\text{H NMR}$ (400 MHz, acetone- d_6) δH : 13.81 (*s*, 1-OH), 9.31 (*s*, 6-OH), 8.02 (*d*, $J=10.2$ Hz, 1H, H-15), 6.83 (*s*, 1H, H-5), 6.68 (*d*, $J=10.1$ Hz, 1H, H-10), 6.27 (*s*, 1H, H-4), 5.95 (*d*, $J=10.2$ Hz, 1H, H-16), 5.72 (*d*, $J=10.1$ Hz, 1H, H-11), 1.47 (*s*, 6H, H-13 & H-14), 1.47 (*s*, 6H, H-18 & H-19). $^{13}\text{C NMR}$ (100 MHz, acetone- d_6) δC : 183.3 (C-9), 160.8 (C-3), 158.7 (C-1), 157.4 (C-4a), 154.1 (C-5a), 154.0 (C-6), 139.1 (C-7), 133.7 (C-16), 128.5 (C-11), 121.4 (C-15), 120.9 (C-8), 115.9 (C-10), 108.4 (C-8a), 105.0 (C-2), 104.4 (C-9a), 103.5 (C-5), 94.8 (C-4), 78.9 (C-12), 76.8 (C-17), 28.5 (C-13 & C-14), 27.2 (C-18 & C-19). The $^1\text{H NMR}$ and $^{13}\text{C NMR}$ data are similar with literature (Cheng et al., 2004).

Caloxanthone I (7): yellow needle crystal. $^1\text{H NMR}$ (400 MHz, acetone- d_6) δH : 13.47 (*s*, 1-OH), 8.54 (*s*, 1H, 5-OH), 7.43 (*s*, 1H, H-8), 6.69 (*d*, $J=10.0$ Hz, 1H, H-10), 6.58 (*d*, $J=10.0$ Hz, 1H, H-15), 5.90 (*d*, $J=10.0$ Hz, 1H, H-16), 5.74 (*d*, $J=10.0$ Hz, 1H, H-11), 5.33 (*t*, 7.4, 1H, H-2'), 3.53 (*d*, $J=7.4$ Hz, 2H, H-1'), 1.87 (*s*, 3H, H-4'), 1.66 (*s*, 3H, H-5'), 1.51 (*s*, 6H, H-18 & H-19), 1.49 (*s*, 6H, H-13 & H-14). $^{13}\text{C NMR}$ (100 MHz, acetone- d_6) δC : 181.5 (C-9), 158.6 (C-3), 156.7 (C-1), 154.9 (C-4a), 146.9 (C-6), 146.7 (C-5a), 134.5 (C-5), 132.5 (C-16), 131.9 (C-3'), 128.5 (C-11), 123.2 (C-2'), 122.1 (C-15), 119.3 (C-7), 116.2 (C-10), 115.2 (C-8a), 113.2 (C-8), 108.4 (C-4), 105.1 (C-2), 103.5 (C-9a), 79.0 (C-17), 78.9 (C-12), 28.5 (C-18 & C-19), 28.3 (C-13 & C-14), 25.9 (C-5'), 22.0 (C-1'), 18.2 (C-4'). The $^1\text{H NMR}$ and $^{13}\text{C NMR}$ data are similar with literature (Zamakshshari et al., 2019).

Bracteaxanthone XII (8): yellow needle crystal. $^1\text{H NMR}$ (400 MHz, acetone- d_6) δH : 13.41 (*s*, 1-OH), 9.02 (*s*, 7-OH), 7.59 (*d*, $J=3.1$ Hz, 1H, H-8), 7.55 (*d*, $J=9.0$ Hz, 1H, H-5), 7.38 (*dd*, $J=9.0$ & 3.1 Hz, 1H, H-6), 6.90 (*d*, $J=10.0$ Hz, 1H, H-10), 5.77 (*d*, $J=10.0$ Hz, 1H, H-11), 5.25 (*t*, $J=7.4$ Hz, 1H, H-2'), 3.34 (*d*, $J=7.4$ Hz, 2H, H-1'), 1.81 (*s*, 3H, H-4'), 1.66 (*s*, 3H, H-5'), 1.51 (*s*, 6H, H-13 & H-14). $^{13}\text{C NMR}$ (100 MHz, acetone- d_6) δC : 180.0 (C-9), 158.8 (C-1), 157.3 (C-3), 154.1 (C-7), 149.1 (C-4a), 148.4 (C-5a), 130.4 (C-3), 127.1 (C-11), 124.3 (C-6), 122.1 (C-2), 119.6 (C-8a), 119.0 (C-5), 114.1 (C-10), 110.1 (C-2), 108.4 (C-8), 101.8 (C-9a), 101.2 (C-4), 78.4 (C-12), 27.4 (C-13 & C-14), 25.0 (C-5), 20.8 (C-1'), 17.1 (C-4). The $^1\text{H NMR}$ and $^{13}\text{C NMR}$ data are similar with literature (Zhang et al., 2019).

Brasixanthone B (9): yellow needle crystal. $^1\text{H NMR}$ (400 MHz, acetone- d_6) δH : 13.30 (*s*, 1-OH), 8.97 (*s*, 7-OH), 7.57 (*d*, $J=3.0$ Hz, 1H, H-8), 7.52 (*d*, $J=9.0$ Hz, 1H, H-5), 7.37 (*dd*, $J=9.0$ & 3.0 Hz, 1H, H-6), 6.71 (*d*, $J=10.0$ Hz, 1H, H-10), 5.76 (*d*, $J=10.0$ Hz, 1H, H-11), 3.49 (*d*, $J=7.4$ Hz, 2H, H-1'), 5.26 (*t*, $J=7.4$ Hz, 1H, H-2'), 1.89 (*s*, 3H, H-4'), 1.66 (*s*, 3H, H-5'), 1.50 (*s*, 6H, H-13 & H-14). $^{13}\text{C NMR}$ (100 MHz, acetone- d_6) δC : 181.8 (C-9), 158.9 (C-3), 156.5 (C-1), 155.3 (C-4a), 154.9 (C-7), 150.9 (C-5a), 131.8 (C-3'), 125.3 (C-6), 128.6 (C-11), 123.1 (C-2'), 121.6 (C-8a), 119.9 (C-5), 116.1 (C-10), 109.2 (C-8), 108.0 (C-4), 104.8 (C-2), 103.8 (C-9a), 79.0

(C-12), 28.5 (C-13 & C-14), 25.9 (C-5'), 21.9 (C-1'), 18.1 (C-4'). The ^1H NMR and ^{13}C NMR data are similar with literature (Mah et al., 2012).

Caloxanthone C (10): yellow needle crystal. ^1H NMR (400 MHz, acetone- d_6) δH : 13.72 (*s*, 1-OH), 8.82 (*s*, 5-OH), 7.67 (*dd*, $J=7.9$ & 1.5 Hz, 1H, H-8), 7.38 (*dd*, $J=7.9$ & 1.5 Hz, 1H, H-6), 7.24 (*t*, $J=7.9$ Hz, 1H, H-7), 6.73 (*d*, $J=9.9$ Hz, 1H, H-1'), 6.52 (*dd*, $J=17.5$ & 10.6 Hz, 1H, H-2''), 5.76 (*d*, $J=9.9$ Hz, 1H, H-2'), 5.05 (*dd*, $J=17.5$ & 1.2 Hz, 1H, H-3''), 4.90 (*dd*, $J=10.6$ & 1.2 Hz, 1H, H-3'), 1.76 (*s*, 6H, H-4' & H-5'), 1.52 (*s*, 6H, H-4' & H-5'). ^{13}C NMR (100 MHz, acetone- d_6) δC : 182.4 (C-9), 160.2 (C-3), 157.2 (C-1), 155.9 (C-4a), 152.6 (C-2''), 147.3 (C-5), 146.0 (C-5a), 128.6 (C-2'), 124.9 (C-7), 121.8 (C-8a), 120.9 (C-6), 116.3 (C-1'), 116.0 (C-8), 114.4 (C-4), 107.6 (C-3''), 105.8 (C-2), 104.4 (C-9a), 79.3 (C-3'), 41.9 (C-1''), 30.0 (C-4' & C-5''), 28.1 (C-4' & C-5'). The ^1H NMR and ^{13}C NMR data are consistent with literature (Lizazman et al., 2022).

Trapezifolixanthone (11): yellow needle crystal. ^1H NMR (400 MHz, acetone- d_6) δH : 13.33 (*s*, 1-OH), 9.24 (*s*, 5-OH), 7.69 (*dd*, $J=7.9$ & 1.5 Hz, 1H, H-8), 7.39 (*dd*, $J=7.9$ & 1.5 Hz, 1H, H-6), 7.28 (*t*, $J=7.9$ Hz, 1H, H-7), 6.71 (*d*, $J=10.0$ Hz, 1H, H-10), 5.77 (*d*, $J=10.0$ Hz, 1H, H-11), 5.31 (*t*, $J=7.3$ Hz, 1H, H-2'), 3.57 (*d*, $J=7.3$ Hz, 2H, H-1'), 1.87 (*s*, 3H, H-5'), 1.66 (*s*, 3H, H-4'), 1.51 (*s*, 6H, H-13 & H-14). ^{13}C NMR (100 MHz, acetone- d_6) δC : 182.2 (C-9), 159.2 (C-3), 156.6 (C-1), 154.9 (C-4a), 147.2 (C-5), 146.4 (C-5a), 131.8 (C-3'), 128.7 (C-11), 124.8 (C-7), 123.3 (C-2'), 122.1 (C-8a), 121.5 (C-6), 116.3 (C-8), 116.0 (C-10), 108.6 (C-4), 105.1 (C-9a), 104.0 (C-2), 79.1 (C-12), 28.5 (C-13 & C-14), 26.0 (C-4'), 22.0 (C-1'), 18.1 (C-5'). The ^1H NMR and ^{13}C NMR data are consistent with literature (Lizazman et al., 2022).

7-O-Demethylmangostanin (12): yellow solid. ^1H NMR (400 MHz, acetone- d_6) δH : 14.10 (*s*, 1-OH), 6.85 (*s*, 1H, H-5), 6.68 (*d*, $J=10.1$ Hz, 1H, H-10), 6.23 (*s*, 1H, H-4), 5.71 (*d*, $J=10.1$ Hz, 1H, H-11), 5.31 (*t*, $J=7.0$ Hz, 1H, H-2'), 4.18 (*d*, $J=7.0$ Hz, 2H, H-1'), 1.84 (*s*, 3H, H-4'), 1.64 (*s*, 3H, H-4'), 1.50 (*s*, 6H, H-13 & H-14). ^{13}C NMR (100 MHz, acetone- d_6) δC : 183.3 (C-9), 160.5 (C-3), 158.9 (C-1), 157.2 (C-4a), 153.4 (C-6), 152.9 (C-5a), 141.9 (C-7), 131.5 (C-3'), 128.3 (C-11), 127.9 (C-8), 124.3 (C-2'), 116.1 (C-10), 111.9 (C-8a), 104.9 (C-2), 104.3 (C-9a), 101.3 (C-5), 94.5 (C-4), 78.7 (C-12), 28.5 (C-13 & C-14), 26.4 (C-1'), 26.0 (C-5'), 18.3 (C-4'). The ^1H NMR and ^{13}C NMR data are similar with literature (Yang et al., 2017).

9-Hydroxycalabaxanthone (13): yellow needle crystal. ^1H NMR (400 MHz, acetone- d_6) δH : 13.92 (*s*, 1-OH), 6.87 (*s*, 1H, H-5), 6.68 (*d*, $J=10.0$ Hz, 1H, H-10), 6.26 (*s*, 1H, H-4), 3.81 (*s*, 3H, 7a-OCH₃), 5.72 (*d*, $J=10.0$ Hz, 1H, H-11), 5.27 (*t*, $J=6.8$ Hz, 1H, H-2'), 4.12 (*d*, $J=6.8$ Hz, 2H, H-1'), 1.83 (*s*, 3H, H-4'), 1.66 (*s*, 3H, H-5'), 1.47 (*s*, 6H, H-13 & H-14). ^{13}C NMR (100 MHz, acetone- d_6) δC : 182.9 (C-9), 160.8 (C-3), 158.8 (C-1), 157.8 (C-5a), 157.2 (C-4a), 156.3 (C-6), 144.8 (C-7), 138.2 (C-8), 131.6 (C-3'), 128.5 (C-11), 124.6 (C-2'), 116.0 (C-10), 111.9 (C-8a), 105.1 (C-2), 104.2 (C-9a),

102.9 (C-5), 94.7 (C-4), 61.3 (C-7a), 78.8 (C-12), 28.5 (C-13 & C-14), 26.9 (C-1'), 25.9 (C-5'), 18.3 (C-4').

3. Experiment Section

3.1. General

The NMR spectra were recorded on BRUKER spectrophotometer in acetone-*d*₆ at 400 (¹H), 100 (¹³C) MHz with TMS as the internal standard. The column chromatography and radial chromatography (chromatotron) were carried out using silica gel 60 Cat. No. 1093851000 (Merck) and silica gel 60 Cat. No. 1077491000 (Merck). Thin layer chromatography was carried out using TLC silica gel 60 F254 aluminium sheet Cat. No. 1055540001 (Merck). The TLC plate was visualized under UV light at 254 nm and 366 nm.

3.2. Plant Material

The stem bark of *C. soulattri* Burm. F. was collected in 2021 from Semengok, Sarawak, Malaysia. The plant was identified by Mr. Tinjan anak Kuda, a botanist from the Forest Department, Sarawak.

3.3. Extraction and Isolation

Approximately 1.5 kg of dried and ground *C. soulattri* stem bark material was extracted thrice, each time with 6 L of *n*-hexane at room temperature for 72 h. The combined *n*-hexane extract was concentrated under reduced pressure by using BUCHI rotavapor R-215 to afford 28.3 g of dried crude. The crude was subjected to silica gel CC packed with *n*-hexane and eluted with *n*-hexane-CHCl₃ mixtures (90:10 - 0:100) followed by CHCl₃-EtOAc mixtures (90:10 - 0:100) to give 20 fractions (CSH1-20). Fraction CSH7 was isolated with Sephadex LH-20 CC packed and eluted with MeOH 100% to give 14 fractions (CSH7A-N). The purification of subfractions CSH7J-N using chromatotron eluted with *n*-hexane-EtOAc (8:2) yielded 4.1 mg of **2** as yellow solid. Fraction CSH9 was subjected to Sephadex LH-20 with MeOH 100% as mobile phase to give 16 fractions (CSH9A-P). Subfractions CSH9I-N was further purified using chromatotron with *n*-hexane-EtOAc (8:2) eluent to afford **1** (CSH9I6, 6.7 mg) and **3** (CSH9I9, 3.3 mg). Fraction CSH10 was isolated with Sephadex LH-20 CC packed and eluted with MeOH 100% to give 15 fractions. Subfractions CSH10K-O was subjected to purification using chromatotron eluted with *n*-hexane-EtOAc (9:1) and yielded 10.2 mg of **4** (CSH10K1) and 4.3 mg of **5** (CSH10K5).

4. Conclusion

The isolation of the stem bark of *C. soulattri* has afforded four new xanthones **1-5**, and eight known xanthones **6-13**. To the best of our knowledge, this is the first report of brasilixanthone B (**6**), caloxanthone I (**7**), bracteaxanthone XII (**8**), 7-*O*-demethylmangostanin (**12**), and 9-hydroxycalabaxanthone (**13**) from *C. soulattri*.

Supplementary Materials: ¹H NMR, ¹³C NMR, COSY, HSQC, and HMBC spectra of compounds **1-5** are reported in supplementary materials as Figures S1-S25.

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Author Contributions: MAL has contributed to writing - original draft preparation resources, and VJYM conceptualization writing - review, supervision, and funding acquisition.

Conflict of Interest: The authors declare no conflict of interest.

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Figures

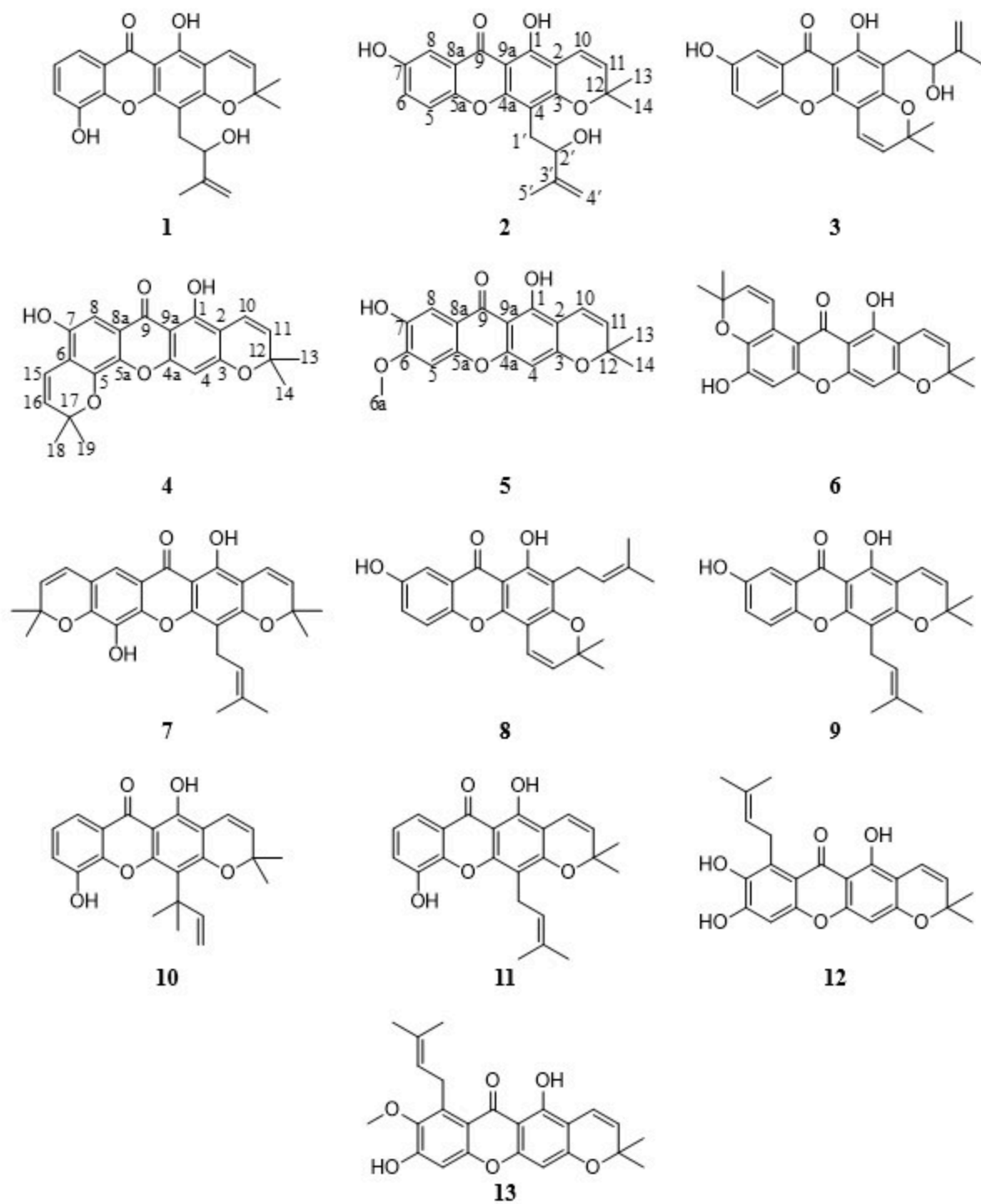


Figure 1

Figure 1. Structure of compounds 1-13.

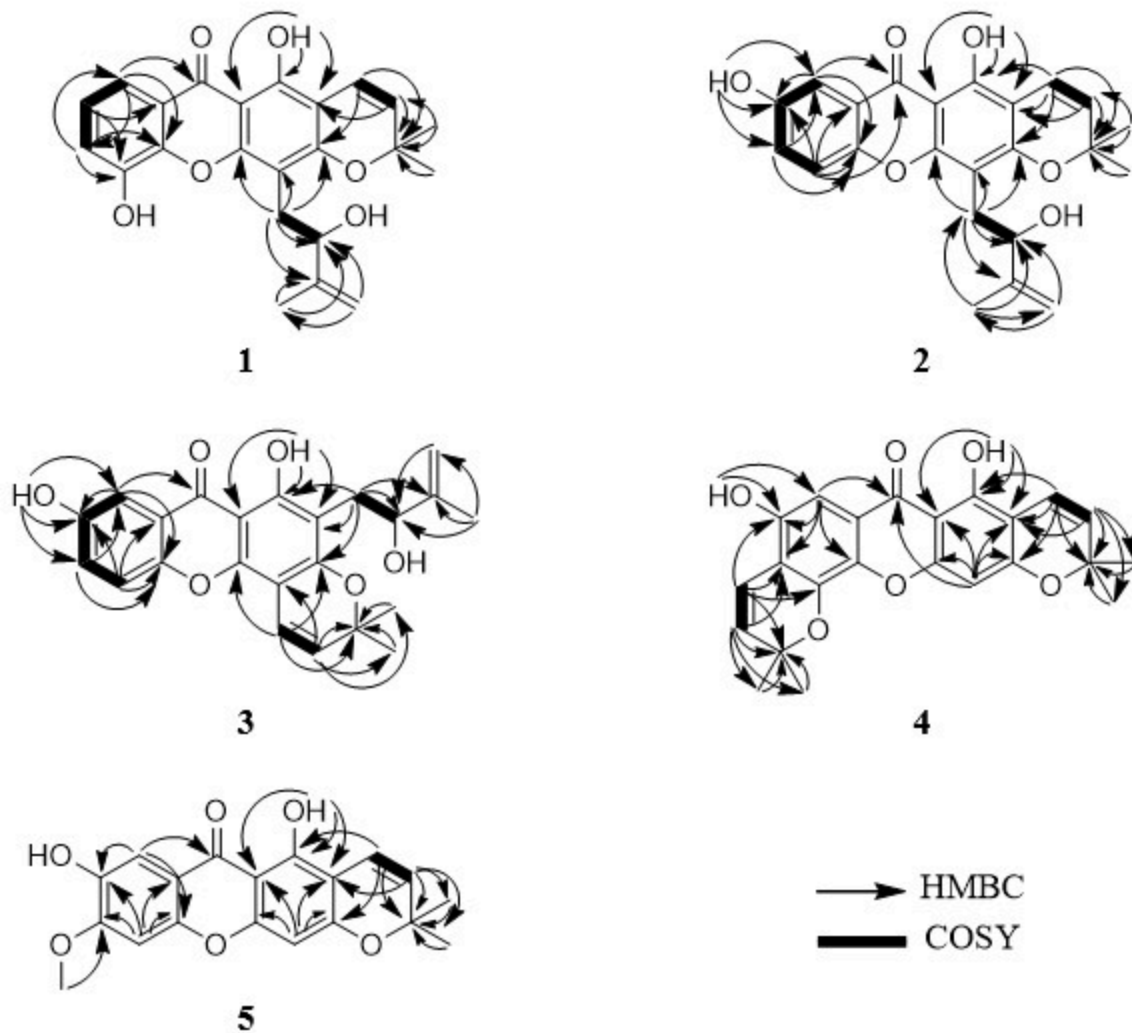


Figure 2

Figure 2. COSY and HMBC correlations for compounds **1-5**.

Supplementary Files

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