

## **4.0 GENERAL METHODS**

### **4.1 PLANT MATERIAL**

The bark of *Aglaia lanuginose*. King was collected in Peninsula Malaysia where its exact location was located at 127 km along the road from Kota Bharu to Ipoh, Jeli Kelantan. A voucher specimen (KL 4232) has been deposited at the Herbarium of Department of Chemistry, University Malaya, Kuala Lumpur.

### **4.2 INSTRUMENTATION**

UV spectra were recorded on a Shimadzu UV-160A UV-Visible Recording Spectrophotometer using dichloromethane as solvent with mirror UV cell. Mass spectra were carried out on Shimadzu GC-MS Spectrometer (HP 6890 Series Mass Selective Detector and HP 6890 Series GC System). The infrared (IR) spectra were obtained through Perkin Elmer FT-IR Spectrometer RX1 using chloroform as solvent. NMR spectra were obtained using JEOL-JNM-LA400 FT NMR Spectrometer System using deuterated chloroform as solvent.

### **4.3 CHROMATOGRAPHY**

#### **4.3.1 Thin layer chromatography (TLC)**

Aluminium supported silica gel 60 F<sub>254</sub> plates was used to determine the spots of the isolated compounds. UV light Model UVGL-58 Mineralight Lamp 230V~50/60 Hz was used to examine spots or bands on the TLC after spraying with the required reagents.

#### **4.3.2 Column chromatography**

All solvents used in this experiment were industrial grade (distilled). Silica gel 60, 70-230 mesh ASTM (Merck 7734) and silica gel 60, 230-400 Mesh ASTM (9385) Merck were used for column chromatography. A slurry of silica gel 60 (approximately 30:1 silica gel to sample ratio) in hexane solvent system was poured into a glass column of appropriate size with gentle tapping to remove air bubbles. The crude extract was initially dissolved in minimum amount of solvent and loaded on top of the packed column. The extract was eluted with an appropriate solvent system at a certain flow rate. Fractions with similar TLC profiles compounds were combined and evaporated off.

### **4.3.3 Preparative thin layer chromatography**

Plates of size 20 cm x 20 cm were cleaned using soap and rinsed with water, then with acetone and dried in the oven. The slurry was prepared by adding 60 g of silica gel 60F<sub>254</sub> (230-400 Mesh ASTM) or DC-Fertigplatten SIL G-24UV<sub>254</sub> to 120 ml distilled water in a closed container, and was shaken vigorously to obtain a smooth mixture. The slurry was then spread onto the clean and dry plates using the Shandon spreading jig with thickness ranging from 0.25 mm to 1 mm, depending on the amount of samples to be worked upon. Then, the plates were activated in the oven for over an hour at 110°C before used.

### **4.3.4 Detector reagents**

#### **a) Iodine vapour**

The TLC plate was placed in a container or tank containing iodine/ iodine vapour. Brown spots on yellow background indicated the presence of unsaturated compounds.

#### **b) Vanillin-sulphuric acid vapour**

Vanillin solution was prepared by adding vanillin (0.5 g) in concentrated H<sub>2</sub>SO<sub>4</sub> (2ml) was added with cooling to ethanol (8 ml) before spraying onto TLC plate. The plate was then heated at 100°C-105°C until full development of colours had

occurred. Dried chromatography TLC plates were sprayed with vanillin reagent. The occurrence are blue, red, pink, brown, dark green, grey or purple indicated the presence of phenylpropenes and simple terpenes

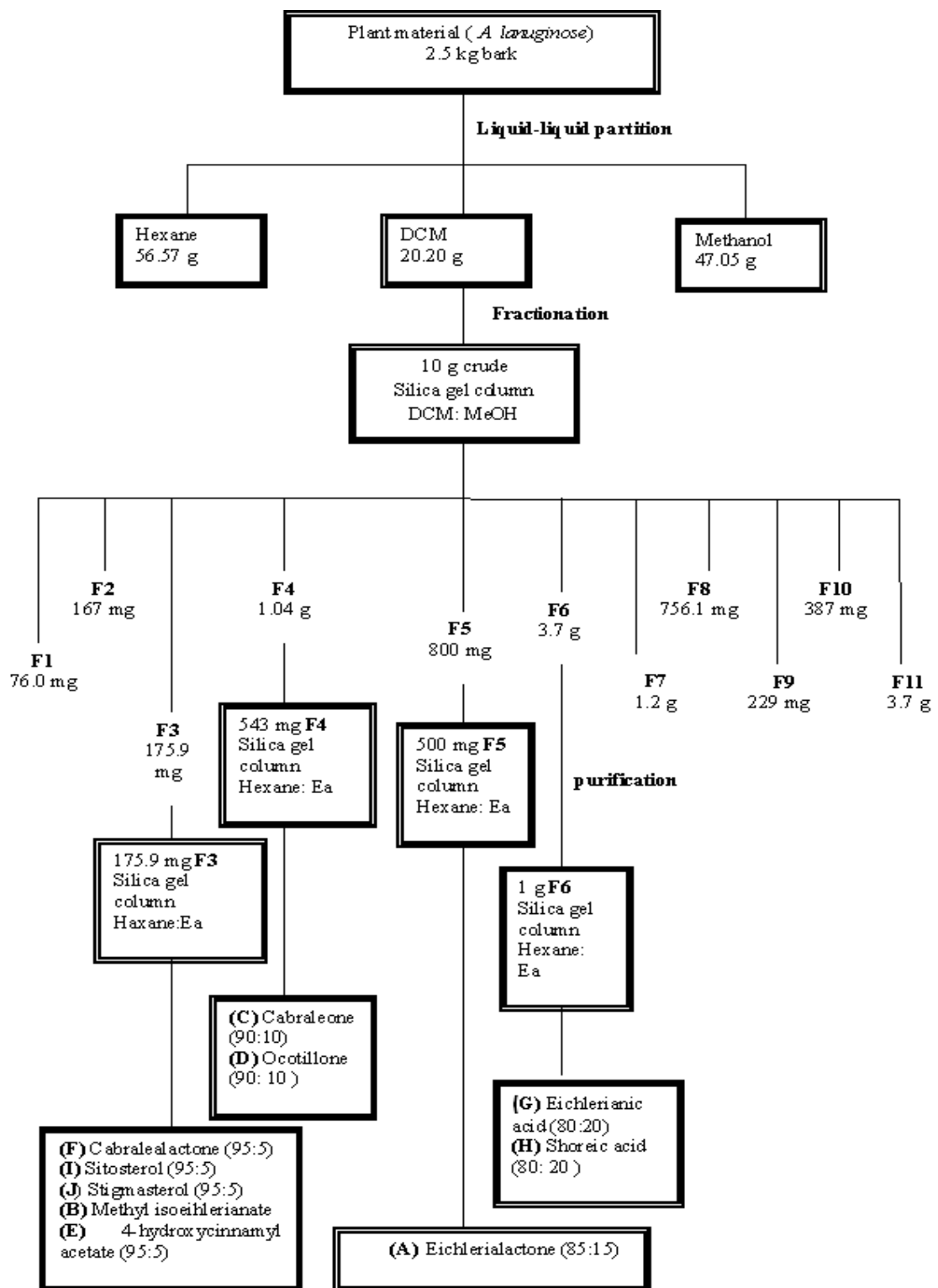
#### **4.4 EXTRACTION OF PLANT MATERIALS.**

Dried ground barks (2.5 kg) were extracted exhaustively with hexane, DCM and MeOH at room temperature. The hexane, DCM and MeOH extracts were evaporated using rotary evaporator to yield crud hexane (58.57 g), DCM (20.2 g) and MeOH (47.05 g) respectively.

The DCM crude extract (10 g) was subjected to a column chromatography over silica gel and eluted with DCM/ MeOH, hexane/ ethyl acetate, hexane/ acetone to give a total of 11 fractions. The column was eluted with mixtures of DCM/ MeOH with 1% increment of each mixture. A total of 11 fractions were collected. Each fraction was subjected to column chromatography over silica gel for purification.

The column was eluted with mixtures of hexane/ ethyl acetate with 5% increment of each mixture. A number of compounds were further purified by multiple development of prep-TLC plate using hexane/ ethyl acetate or hexane/ acetone (90:10, 80:20, 70:30, 60:40 etc) solvent systems depending on the polarity of the compounds. The non-polar fraction, fraction 3 (175 mg) was further purified by a column chromatography (eluent hexane/ ethyl acetate) to give compound **F, I, J, B** and **E**.

The other non-polar fraction, fraction 4 was purified by a small column (eluent hexane/ ethyl acetate) followed by HPLC on reversed phase C-18 column to give compound **C** and **D**. The detector used for the separation of compound **C** and **D** was the Refractive Index (RI) Detector. The purification of fraction 5 with column chromatography gave compound **A**. The moderately polar fraction, fraction 6 was further purified by a small column (eluent hexane/ acetone) to obtain a mixture of compound **G** and **H**. Compound **G** and **H** were obtained in pure forms by using recrystallization technique with methanol solvent. The structure identification was carried out by <sup>1</sup>H-NMR, <sup>13</sup>C-NMR, COSY, HMQC, HMBC, DEPT, IR, UV and Mass- spectroscopy. The purification of compounds isolated from *Aglaia lanuginose* was summarized in Scheme 4.1.



Scheme 4.1: Flow chart showing the isolation and purification of compounds from *Aglaia lanuginosa*.

#### 4.4 GENERAL SPECTRAL DATA

Eichlerialactone **1**: isolation as white crystal, mp 109-110°C. <sup>1</sup>H-NMR (400 MHz, CDCl<sub>3</sub>): δ1.24 (2H, m, H-1α), δ1.60 (2H, m, H-1β), δ1.18(2H, m, H-2α), δ1.50 (2H, m, H-2β), δ1.98 (H, m, 5), δ1.38 (2H, m, H-6), δ1.54 (2H, m, H-7β), δ1.24 (2H, m, H-7α), δ1.50 (1H, m, H-9), δ1.30 (2H, m, H-11α), δ1.44 (2H, m, H-11β), δ1.28 (2H, m, H-12α), δ1.90 (2H, m, H-12β), δ1.68 (H, m, H-13), δ1.24 (2H, m, H-15α), δ1.50 (2H, m, H-15β), δ1.32 (2H, m, H-16α), δ1.82 (2H, m, H-16β), δ1.94 (1H, m, H-17), δ0.90 (3H, s, H-18), δ0.86 (3H, s, H-19), δ 1.34 (3H, s, H-21), δ1.94 (2H, m, H-22α), δ2.16 (2H, m, H-22β), δ2.56 (2H, m, H-23), δ2.0 (2H, brs, H-25), δ2.0 (2H, m, H-22), δ4.67(1H, s, H-25a), δ4.88(1H, s, H-25b), δ1.76 (3H, s, H-26), δ1.0 (3H, s, H-27); <sup>13</sup>C-NMR (100 MHz CDCl<sub>3</sub>) (C-1 to C-27): 34.43 (1), 28.41 (2), 176.90 (3), 147.40 (4), 50.80 (5), 24.56 (6), 33.89 (7), 40.11 (8), 41.07 (9), 39.13 (10), 21.84 (11), 26.41 (12), 42.70 (13), 50.42 (14), 31.07 (15), 25.00 (16), 49.45 (17), 16.14 (18), 20.14 (19), 89.86 (20), 22.47 (21), 32.95 (22), 28.68 (23), 177.00 (24), 147.40 (25), 23.22 (26), 15.33 (27) From LCMS, m/z 453.33 [M+Na] (calcd for C<sub>27</sub>H<sub>42</sub>O<sub>3</sub>Na), 431.32[M+H] (calcd for C<sub>27</sub>H<sub>42</sub>O<sub>4</sub>)

20,24-Epoxy-25-hydroxy-3,4-seco-4(28) dammaren-3-ioc acid methyl ester. (Methyl isoeichlerianate): Isolated as a white crystal. mp 109-110°;  $\nu_{\max}$ , (CHCl<sub>3</sub>) 3460, 3553, 1743, 1635 cm<sup>-1</sup>. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ1.73 (2H, m, H-1β), δ1.53 (2H, m, H-1α), δ0.88 (3H, s, H-18), δ0.84 (3H, s, H-19), δ1.14 (3H, s, H-21), δ3.75 (1H, t, H-24), δ1.18 (3H, s, H-26), δ1.11 (3H, s, H-27), δ4.66 (1H, brs, H-28α), δ4.84 (1H, brs, H-28β), δ1.76 (3H, s, H-29), δ0.99 (3H, s, H-30), δ3.66 (3H, s, H-31); <sup>13</sup>C-NMR data (100 MHz CDCl<sub>3</sub>) (C-1 to C-30): 34.40 (C1), 28.32 (2), 174.59 (3), 147.54 (4), 50.7 (5), 24.58 (6), 33.87 (7), 39.99 (8), 40.97 (9), 37.48 (10), 21.68 (11), 26.91 (12), 42.90 (13), 50.62 (14), 31.18 (15), 25.75 (16), 50.62 (14), 31.18 (15), 25.75 (16), 50.02 (17), 15.97 (18), 20.13 (19), 86.03 (20), 21.93 (21), 37.48 (22), 29.63 (23), 86.4 (24), 71.1 (25), 27.6 (26), 24.2 (27), 113.2 (28), 23.2 (29), 15.3 (30), 51.6 (31). EIMS m/z (rel. int.): 143 [M-C<sub>8</sub>H<sub>15</sub>O<sub>2</sub>]<sup>+</sup> (100), 429 [M-59]<sup>+</sup>, 473 [M-15]<sup>+</sup> (8). 489 [M+1].

Compound **C**: 20S-2SR-epoxy-25-hydroxydammarane-3-one (cabraleone). Isolated as white amorphous powder; mp 163-166.5°, [ $\alpha$ ]<sub>D</sub>+77° (c, 0.1),  $\nu_{\max}$  (CHCl<sub>3</sub>) 3520, 1690 (C=O), 1085, 1064 cm<sup>-1</sup>. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$ 1.45 (2H, m, H-1 $\alpha$ ),  $\delta$ 1.72 (2H, m, H-1 $\beta$ ),  $\delta$ 2.46 (2H, m, H-2 $\alpha$ ),  $\delta$ 2.54 (2H, m, H-2 $\beta$ ),  $\delta$ 1.38 (1H, m, H-5),  $\delta$ 1.45 (2H, m, H-6 $\alpha$ ),  $\delta$ 1.55 (2H, m, H-6 $\beta$ ),  $\delta$ 1.30 (2H, m, H-7 $\alpha$ ),  $\delta$ 1.58 (2H, m, H-7 $\beta$ ),  $\delta$ 1.88 (1H, m, H-9),  $\delta$ 1.28 (2H, m, H-11 $\alpha$ ),  $\delta$ 1.50 (2H, m, H-11 $\beta$ ),  $\delta$ 1.25 (2H, m, H-12),  $\delta$ 1.58 (1H, m, H-13),  $\delta$ 1.10 (2H, m, H-15 $\alpha$ ),  $\delta$ 1.48 (2H, m, H-16 $\beta$ ),  $\delta$ 1.32 (2H, m, H-16 $\alpha$ ),  $\delta$ 1.44 (1H, s, H-17),  $\delta$ 0.95 (3H, s, H-18),  $\delta$ 0.89 (3H, s, H-19),  $\delta$ 1.11 (3H, s, H-21),  $\delta$ 1.7 (2H, m, H-22 $\alpha$ ),  $\delta$ 1.85 (2H, m, H-22 $\beta$ ),  $\delta$ 3.64 (1H, t, H-24),  $\delta$ 1.19 (3H, s, H-26),  $\delta$ 1.15 (3H, s, H-27),  $\delta$ 1.08 (3H, s, H-28),  $\delta$ 1.04 (3H, s, H-29),  $\delta$ 1.01 (3H, s, H-30). <sup>13</sup>C-NMR (100 MHz CDCl<sub>3</sub>) (C-1 to C-30): 39.94 (C1), 34.13 (C2), 218.14 (C3), 47.43 (4), 55.35 (5), 19.68 (6), 34.80 (7), 40.32 (8), 49.80 (9), 36.88 (10), 22.34 (11), 27.02 (12), 43.00 (13), 50.02 (14), 31.44 (15), 25.84 (16), 50.20 (17), 16.32 (18), 16.09 (19), 86.52 (20), 27.20 (21), 34.62 (22), 26.38 (23), 86.38 (24), 70.27 (25), 24.08 (26), 27.83 (27), 26.37 (28), 21.01 (29), 15.21 (30). EIMS *m/z* (rel. int.): 143 [M-C<sub>8</sub>H<sub>15</sub>O<sub>2</sub>]<sup>+</sup> (100), 443 [M-15]<sup>+</sup> (8), 399 [M-59]<sup>+</sup>. LCMS found *m/z* 481.3605(M+Na)<sup>+</sup> (calcd for C<sub>30</sub>H<sub>50</sub>O<sub>3</sub>Na, 481.3002).

Compound **D**: 20S-2R-epoxy-25-hydroxydammarane -3-one (ocotillone). Mp 161-164° Isolated as a white amorphous powder.  $\nu_{\max}$  (CHCl<sub>3</sub>) 3520, 1690 (C=O), 1085, 1064 cm<sup>-1</sup> <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$ 1.46 (2H, m, H-1 $\alpha$ ),  $\delta$ 1.94 (2H, m, H-1 $\beta$ ),  $\delta$ 1.38 (1H, m, H-5),  $\delta$ 1.40 (2H, m, H-6 $\alpha$ ),  $\delta$ 1.55 (2H, m, H-6 $\beta$ ),  $\delta$ 1.66 (2H, m, H-7 $\alpha$ ),  $\delta$ 1.70 (2H, m, H-7 $\beta$ ),  $\delta$ 1.80 (1H, m, H-9),  $\delta$ 1.25 (2H, m, H-11 $\alpha$ ),  $\delta$ 1.50 (2H, m, H-11 $\beta$ ),  $\delta$ 2.00 (2H, m, H-12 $\alpha$ ),  $\delta$ 1.70 (2H, m, H-13),  $\delta$ 1.1 (1H, m, H-13),  $\delta$ 0.90 (3H, s, H-15 $\alpha$ ),  $\delta$ 1.48 (2H, s, H-15 $\beta$ ),  $\delta$ 1.32 (2H, m, H-16),  $\delta$ 1.44 (1H, m, H-17),  $\delta$ 0.88 (3H, s, H-18),  $\delta$ 0.94 (3H, s, H-19),  $\delta$ 1.11 (3H, s, H-21),  $\delta$ 1.70 (2H, m, H-22 $\alpha$ ),  $\delta$ 1.85 (2H, m, H-22 $\beta$ ),  $\delta$ 1.80 (2H, m, H-23),  $\delta$ 3.76 (1H, t, H-24),  $\delta$ 1.15 (3H, m, H-26),  $\delta$ 1.20 (3H, m, H-27),  $\delta$ 1.08 (3H, m, H-28),  $\delta$ 1.04 (3H, m, H-29),  $\delta$ 1.00 (3H, m, H-30). <sup>13</sup>C-NMR (100 MHz CDCl<sub>3</sub>): 39.90 (1), 34.13 (2), 218.13 (3), 47.41(4), 55.33 (5), 19.67 (6), 37.57(7), 40.31(8), 50.09(9), 36.87(10), 21.99(11), 27.04 (12), 43.02 (13), 49.94 (14), 31.18 (15), 25.85 (16), 50.09 (17), 16.05 (18), 16.05 (19), 86.45 (20), 26.76 (21), 34.63 (22), 34.63 (23), 84.49 (24), 71.14 (25),



21.73 (26), 27.69 (27), 24.31(28), 21.03(29), 15.20 (30). EIMS  $m/z$  (rel. int.): 143 [M-C<sub>8</sub>H<sub>15</sub>O<sub>2</sub>]<sup>+</sup> (100), 443 [M-15]<sup>+</sup> (8), 399 [M-59]<sup>+</sup>. LCMS found  $m/z$  481.3605(M+Na)<sup>+</sup> (calcd for C<sub>30</sub>H<sub>50</sub>O<sub>3</sub>Na, 481.3002).

Compound **E**: 4-Hydroxycinnamyl acetate. Isolated as yellow oil; UV  $\lambda_{\max}$  314, IR  $\nu_{\max}$  3383, 1733, 1652, 1602, 802 cm<sup>-1</sup>. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$ 7.36 (1H, d, J=8.6 Hz, H-2),  $\delta$ 7.02 (1H, d, J=8.6 Hz, H-3),  $\delta$ 7.02 (1H, d, J=8.6 Hz, H-5),  $\delta$ 7.36 (1H, d, J=8.6 Hz, H-6),  $\delta$ 6.57 (1H, d, J=16.12 Hz, H-7),  $\delta$ 6.29 (1H, d, J= 5.6, 5.6 Hz, H-8),  $\delta$ 4.30 (2H, d, J= 6.6 Hz, H-9),  $\delta$ 2.28 (3H, s, H-11). <sup>13</sup>C-NMR data (100 MHz CDCl<sub>3</sub>): 134.48 (1), 121.68 (2), 127.39 (3), 150.63 (4), 127.39 (5), 121.68 (6), 130.05 (7), 128.7 (8), 63.59 (9), 169.55 (10), and 21.12 (11), EIMS  $m/z$  192(calcd for C<sub>11</sub>H<sub>12</sub>O<sub>3</sub>).

Compound **F**: Cabralealactone. Isolated as a white amorphous powder. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$ 1.84 (2H, m, H-1),  $\delta$ 2.5 (2H, m, H-2),  $\delta$ 1.49 (2H, m, H-7),  $\delta$ 1.31 (1H, m, H-9),  $\delta$ 1.56 (2H, m, H-12 $\alpha$ ),  $\delta$ 1.21 (2H, m, H-12 $\beta$ ),  $\delta$ 1.52 (H, m, H-13),  $\delta$ 1.31 (2H, s, H-14),  $\delta$ 1.85 (H, s, H-17),  $\delta$ 0.83 (3H, s, H-18),  $\delta$ 0.92 (3H, s, H-19),  $\delta$ 1.32 (3H, s, H-21),  $\delta$ 2.00 (2H, m, H-22),  $\delta$ 1.85 (2H, m, H-23),  $\delta$ 1.06 (3H, m, H-25),  $\delta$ 1.01 (3H, m, H-26),  $\delta$ 0.98 (3H, m, H-27). <sup>13</sup>C-NMR (100 MHz CDCl<sub>3</sub>): 39.82 (1), 34.54 (2), 218.04 (3), 47.38 (4), 55.28 (5), 19.59 (6), 34.09 (7), 42.73 (8), 49.97 (9), 36.82 (10), 21.76 (11), 24.95 (12), 42.97 (13), 49.43 (14), 30.98 (15), 28.62 (16), 49.43 (17), 16.07 (18), 15.17 (19), 89.86 (20), 22.08 (21), 33.15 (22), 26.40 (23), 176.93 (24), 26.69 (25), 21.01 (26), 15.99 (27). EIMS  $m/z$  (rel. int.): 414 [M]<sup>+</sup>, 399 [M-15]<sup>+</sup> (8), 315 [M-99]<sup>+</sup>. LCMS found  $m/z$  437.30 (M+Na)<sup>+</sup>.

Compound **G**: 20S, 24S-epoxy-25-hydrogen-3-4-secodammarane -4(28)-en -3-oic acid (eichlerianic acid). Isolated as a white amorphous powder; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): 1.75 (2H, m, H-1 $\alpha$ ),  $\delta$ 1.53 (2H, m, H-1 $\beta$ ),  $\delta$ 2.14 (2H, m, H-2 $\alpha$ ),  $\delta$ 2.32 (2H, m, H-2 $\beta$ ),  $\delta$ 1.15 (2H, m, H-7),  $\delta$ 1.43 (1H, m, H-9),  $\delta$ 1.35 (2H, m, H-11),  $\delta$ 1.40 (3H, s, H-15),  $\delta$ 1.75 (2H, m,

H-16),  $\delta$ 1.80 (1H, m, H-17),  $\delta$ 0.82 (3H, s, H-18),  $\delta$ 0.79 (3H, s, H-19),  $\delta$ 1.08 (3H, s, H-21),  $\delta$ 1.57 (2H, m, H-22),  $\delta$ 1.80 (2H, m, H-23),  $\delta$ 3.57 (1H, t, H-24),  $\delta$ 1.13 (3H, m, H-26),  $\delta$ 1.05 (3H, m, H-27),  $\delta$ 4.60 (3H, m, H-28 $\alpha$ ),  $\delta$ 4.78 (3H, m, H-28 $\beta$ ),  $\delta$ 1.67 (3H, m, H-29),  $\delta$ 0.99 (3H, m, H-30).  $^{13}\text{C}$ -NMR (100 MHz  $\text{CDCl}_3$ ) (C-1 to C-30): 34.26 (1), 28.46 (2), 179.52 (3), 147.46 (4), 50.77 (5), 24.58 (6), 33.87 (7), 40.01 (8), 41.15 (9), 39.03 (10), 22.29 (11), 26.86 (12), 42.88 (13), 50.34 (14), 31.43 (15), 25.78 (16), 49.71 (17), 16.28 (18), 20.15 (19), 86.56 (20), 27.09 (21), 34.72 (22), 26.32 (23), 86.31 (24), 70.32 (25), 27.77 (26), 23.19 (27), 113.41 (28), 24.00 (29), 15.30 (30). EIMS  $m/z$  (rel. int.): 143 [ $\text{M}-\text{C}_8\text{H}_{15}\text{O}_2$ ] $^+$  (100), 473 [ $\text{M}-1$ ].

Compound **H**: 20S, 24R-epoxy-25-hydrogen-3-4-secodammarane -4(28)-en -3-oic acid (shoreic acid). Isolated as white crystal;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$ 1.75 (H, m, H-1 $\alpha$ ),  $\delta$ 1.53 (H, m, H-1 $\beta$ ),  $\delta$ 2.14 (H, m, H-2 $\alpha$ ),  $\delta$ 2.32 (H, m, H-2 $\beta$ ),  $\delta$ 1.15 (2H, m, H-7),  $\delta$ 1.43 (1H, m, H-9),  $\delta$ 1.35 (2H, m, H-11),  $\delta$ 1.40 (3H, s, H-15),  $\delta$ 1.75 (2H, m, H-16),  $\delta$ 1.80 (1H, m, H-17),  $\delta$ 0.82 (3H, s, H-18),  $\delta$ 0.79 (3H, s, H-19),  $\delta$ 1.08 (3H, s, H-21),  $\delta$ 1.57 (2H, m, H-22),  $\delta$ 1.80 (2H, m, H-23),  $\delta$ 3.57 (1H, t, H-24),  $\delta$ 1.13 (3H, m, H-26),  $\delta$ 1.05 (3H, m, H-27),  $\delta$ 4.60 (3H, m, H-28 $\alpha$ ),  $\delta$ 4.78 (3H, m, H-28 $\beta$ ),  $\delta$ 1.67 (3H, m, H-29),  $\delta$ 0.99 (3H, m, H-30).  $^{13}\text{C}$ -NMR (100 MHz  $\text{CDCl}_3$ ): 34.26 (1), 28.46 (2), 179.52 (3), 147.46 (4), 50.77 (5), 24.58 (6), 33.87 (7), 40.01 (8), 41.15 (9), 39.03 (10), 22.29 (11), 26.86 (12), 42.88 (13), 50.34 (14), 31.43 (15), 25.78 (16), 49.71 (17), 16.28 (18), 20.15 (19), 86.56 (20), 27.09 (21), 34.72 (22), 26.32 (23), 84.36 (24), 70.32 (25), 27.77 (26), 23.19 (27), 113.41 (28), 24.00 (29), 15.30 (30). EIMS  $m/z$  (rel. int.): 143 [ $\text{M}-\text{C}_8\text{H}_{15}\text{O}_2$ ] $^+$  (100), 473 [ $\text{M}-1$ ].

Sitosterol and Stigmasterol( isolated as mixtures). Isolated as white solid; UV  $\lambda_{\text{max}}$  302, 504(mixture),  $\nu_{\text{max}}$  ( $\text{CHCl}_3$ ) 3430 (mixture);  $^1\text{H}$ -NMR sitosterol (400 MHz,  $\text{CDCl}_3$ ):  $\delta$ 1.84 (H, m, H-1 $\beta$ ),  $\delta$ 1.08 (H, m, H-1 $\alpha$ ),  $\delta$ 1.80 (H, m, H-2 $\beta$ ),  $\delta$ 1.50 (H, m, H-2 $\alpha$ ),  $\delta$ 3.51 (2H, m, H-3 $\alpha$ ),  $\delta$ 2.24 (H, m, H-4 $\beta$ ),  $\delta$ 2.26 (H, m, H-4 $\alpha$ ),  $\delta$ 5.33 (H, d J=5.12Hz, H-6)  $\delta$ 1.96 (H, m, H-7 $\beta$ ),  $\delta$ 1.50 (H, m, H-7 $\alpha$ ),  $\delta$ 0.65 (3H, s, H-18)  $\delta$ 1.00 (3H, s, H-19)  $\delta$ 0.95 (3H, d, H-21)  $\delta$ 0.80 (3H, d, H-26),  $\delta$ 0.77 (3H, d, H-27),  $\delta$ 0.77 (3H, t, H-29);  $^1\text{H}$ -NMR stigmasterol (400

MHz, CDCl<sub>3</sub>):  $\delta$ 1.84 (H, m, H-1 $\beta$ ),  $\delta$ 1.08 (H, m, H-1 $\alpha$ ),  $\delta$ 1.80 (H, m, H-2 $\beta$ ),  $\delta$ 1.50 (H, m, H-2 $\alpha$ ),  $\delta$ 3.51 (2H, m, H-3 $\alpha$ ),  $\delta$ 2.24 (H, m, H-4 $\beta$ ),  $\delta$ 2.26 (H, m, H-4 $\alpha$ ),  $\delta$ 5.33 (H, d, J=5.12Hz, H-6)  $\delta$ 1.96 (H, m, H-7 $\beta$ ),  $\delta$ 1.50 (H, m, H-7 $\alpha$ ),  $\delta$ 0.65 (3H, s, H-18)  $\delta$ 1.00 (3H, s, H-19)  $\delta$ 0.95 (3H, d, H-21),  $\delta$ 5.15 (H, dd, J<sub>22,23</sub>=15.1Hz, H-22),  $\delta$ 5.00 (H, dd, J<sub>23,24</sub>=8.5 Hz, H-23),  $\delta$ 1.54 (H, m, H-24),  $\delta$ 0.80 (3H, d, H-26),  $\delta$ 0.77 (3H, d, H-27),  $\delta$ 0.77 (3H, t, H-29);

<sup>13</sup>C-NMR sitosterol (100 MHz CDCl<sub>3</sub>):

37.20 (1), 31.60 (2), 71.70 (3), 42.30 (4), 140.70 (5), 121.70 (6), 31.80 (7), 31.80 (8), 50.10 (9), 36.50 (10), 21.10 (11), 39.80 (12), 42.30 (13), 56.70 (14), 24.30 (15), 28.20 (16), 56.03 (17), 11.84 (18), 19.40 (19), 36.13 (20), 18.76 (21), 33.90 (22), 25.38 (23), 45.81 (24), 29.1 (25), 19.79 (26), 19.01 (27), 23.05 (28), 11.96 (29),

<sup>13</sup>C-NMR stigmasterol (100 MHz CDCl<sub>3</sub>):

37.20 (1), 31.60 (2), 71.70 (3), 42.30 (4), 140.70 (5), 121.69 (6), 31.80 (7), 31.80 (8), 50.10 (9), 36.50 (10), 21.10 (11), 39.80 (12), 42.30 (13), 56.70 (14), 24.30 (15), 28.20 (16), 56.03 (17), 12.00 (18), 19.40 (19), 40.4 (20), 21.1 (21), 138.3 (22), 129.3 (23), 51.20 (24), 31.80 (25), 21.2 (26), 19.01 (27), 25.40 (28), 12.20 (29),

Mass spectrum (mixture): [414 M<sup>+</sup>], 396, 329, 303, 213, 145, 105, 55 (fragments identified for sitosterol. 412 [M<sup>+</sup>], 394, 351 (fragments identified for stigmasterol)

## 5.1 CONCLUSIONS

The chemical study on dichloromethane extract of the leaves has led to the isolation of ten compounds. Structural elucidation was established through spectroscopic methods; NMR (nuclear magnetic resonance), UV (ultra violet), IR (infra-red) and MS (mass spectroscopy).

The triterpenoids isolated from the bark of *Aglaia lanuginosa* were the dammarane triterpenoids identified as eichleriatone **85**, methyl eichlerianate **102**, cabraleone **80**, ocotillone **81**, cabralealactone **82**, eichlerianic acid **84**, shoreic acid **83** and two sterols identified as sitosterol **104**, sigmasterol **105**. One aromatic compound, 4-hydroxycinnamyl-acetate **106** was isolated from this plant.

This particular *Aglaia* species (*Aglaia lanuginosa* King) has not been studied before. However there were chemical studies done on other *Aglaia* species; *A. argentia*, *Aglaia lawii*, *Aglaia elliptica*, *Aglaia tomentosa* and *Aglaia foveolata*. As for the triterpenoids isolated from the bark, their occurrences are common to Meliaceae species. As can be observed from table 2.2, there are other Meliaceae species having the similar type of compound with *Aglaia* species such as *Cabralea politricha*, *Cabralea eichleriana*, *Dysoxylum richii*, *Dysoxylum muelleri* and *Dysoxylum cauliflorum*. From this study, we found that compound **106**, 4-hydroxycinnamyl-acetate has never been isolated from any *Aglaia* species.

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