

A Qualitative Analysis of *Litsea fulva* Essential Oils Using Comprehensive Two-dimensional Gas Chromatography Coupled with Time-of-Flight Mass Spectrometry

(Penganalisan Kualitatif dalam Minyak Pati *Litsea fulva* Melalui Gas Kromatografi Dua Dimensi

Berganding dengan Spektrometer Jisim Masa-Terbang (TOF/MS))

KHONG HENG YEN*, LAILY B DIN, ZURIATI ZAKARIA, NOR HADIANI ISMAIL,
NORZAMZURINA ISMAIL & MOHD AMBAR YARMO

ABSTRACT

A qualitative analysis of the individual compounds in *Litsea fulva* (locally known as 'Medang') essential oils was performed by comprehensive two-dimensional gas chromatography (GC × GC) coupled with time-of-flight mass spectrometer (TOF/MS) for the identification of the resolved peaks. *Litsea fulva* essential oil was found to contain 98 identifiable peaks with 32 compounds were identified with good matches. These compounds identified included 30 hydrocarbons, 22 alcohols, five acids, 16 ketones, five aldehydes, 12 esters, six ethers and two other compounds. The *L. fulva* leaf oil contained alcohols and ethers, with 34.09% and 24.38%, respectively. The major components of these oils were *cis*-Z- α -bisabolene epoxide (9.51%), *trans*-Z- α -bisabolene epoxide (8.36%), $C_{13}H_{20}O_2$ (7.39%), longipinocarvone (5.68%), τ -Cadinol (4.24%), $C_{15}H_{24}O$ (4.98%) and α -cadinol (3.95%). The study also showed that the comprehensive two-dimensional gas chromatography (GC × GC) is a better and more powerful separation tool in GC and an identification tool for analyzing complex volatile oils compared with the one-dimensional GC.

Keywords: Comprehensive two-dimensional gas chromatography; essential oils; *Litsea fulva*; time-of-flight mass spectrometer

ABSTRAK

Analisis kualitatif setiap sebatian dalam minyak pati *Litsea fulva* (dikenali sebagai 'Medang') dilakukan melalui kromatografi gas dua dimensi berganding dengan spektrometer jisim masa-terbang (TOF/MS) untuk pengesanan puncak-puncak sebatian yang terlerai. Minyak pati *Litsea fulva* didapati mengandungi 98 puncak dengan 32 daripadanya dapat dicamkan sebatianannya. Sebatian yang telah dicamkan termasuklah 30 hidrokarbon, 22 alkohol, lima asid, 16 keton, lima aldehid, 12 ester, enam eter dan dua sebatian lain. Kelimpahan yang paling tinggi dalam minyak pati daun *Litsea fulva* ialah alkohol dan ester, masing-masing 34.09% dan 24.38%. Komponen utama minyak ini ialah *cis*-Z- α -bisabolena epoksida (9.51%), *trans*-Z- α -bisabolena epoksida (8.36%), $C_{13}H_{20}O_2$ (7.39%), longipinokarvon (5.68%), τ -kadinol (4.24%), $C_{15}H_{24}O$ (4.98%) dan α -kadinol (3.95%). Kajian ini juga menunjukkan kromatografi gas dua dimensi komprehensif adalah alat yang lebih baik untuk pemisahan dalam GC dan alat pengesanan untuk analisis minyak meruap yang kompleks berbanding GC satu dimensi.

Kata kunci: Kromatografi gas dua dimensi komprehensif; *Litsea fulva*; minyak pati; spektrometer jisim masa-terbang

INTRODUCTION

Aromatic forest trees and plants from the Lauraceae family were examined as one of the sources of new essential oils and aroma compounds for possible commercial exploitation (Karim & Adirukmi 1991). *Litsea fulva* belongs to the Lauraceae family, which comprises about 50 genus and 2500-3500 species (Argent et al. 1997). *Litsea* genus has about 400 species, which are distributed throughout tropical and subtropical Asia (except Africa), the Pacific, Australia and New Zealand. *Litsea* species as well as any other Lauraceae are locally known as 'Medang' or 'Tejur'.

Some species of *Litsea* have been studied and recognised for their essential oils chemical composition (Ahmad et al. 2005; Bighelli et al. 2005; Choi & Hwang 2004; Lyth & Charles 1998; Valverde et al. 2005) which

included 1,8-cineole, linalool, sabinene, geraniol, neral, geranial, citral and citronellal. A study on *Litsea cubeba* showed that a lot of citral was found in the fruits and also a large amount of geranial and neral was found in the leaves and stem. All these components are important especially as natural pharmaceutical products for medicinal purposes and chemotaxonomy. These essential oils have been used as the basic raw materials in flavouring, perfumes, preparation of beverages, medicines, cosmetics and cleaning preparations. Due to the economic importance of these essential oils from the *Litsea* species and the lack of detailed studies on these essential oils in Malaysia, this study focused on the detailed comprehensive two-dimensional gas chromatography (GC × GC) coupled with a time-of-flight mass spectrometry (ToFMS) analysis of essential oils from *Litsea fulva*.

MATERIALS AND METHODS

EXTRACTION OF ESSENTIAL OILS

About 100 g of ground samples of *Litsea fulva* were hydrodistilled using the Clevenger-type apparatus for 6 h. The oils were extracted and dried over anhydrous sodium sulphate and then they were kept in a dark cold place (4–5°C) or refrigerated before being analysed.

INSTRUMENTAL ANALYSIS OF ESSENTIAL OILS

The chemical compounds in *Litsea fulva* leaf oils were determined by the comprehensive two-dimensional gas chromatography, which consisted of the HP6890 gas chromatography (Agilent Technologies, USA) and a mass spectrometer type LECO with thermal modulator (Zoex Corporation, USA) to enhance the peak capacity for a chromatographic run. This allowed better separation in the complex sample analysis. The cold-jet modulator consisted of two cold and two hot-jets with nozzles which provided the cold-jets mounted orthogonally to the hot-jets. Nitrogen gas was cooled using a heat exchanger through copper tubing which was immersed in liquid nitrogen outside the GC system and delivered through a vacuum-insulated tubing to the cold-jets. This provided two continuous jets of cold nitrogen gas. The GC oven contained two capillary columns which were connected serially via the cold-jet modulator. The column set used a primary column of dimensions 30 m × 0.25 mm i.d × 0.10 µm film thickness Rxi-5MS (coated with 5% diphenyl and 95% dimethyl polysiloxane) phase serially coupled to a second column with dimensions 1.10 m × 0.10 mm i.d × 0.10 µm film thickness Rxi-17 (Crossbond@ 50% diphenyl/50% dimethyl polysiloxane) phase. Both columns were housed in two different ovens, which had its temperature programmed from 55°C (held for 3 min) to 265°C (held for 5 min) at a rate of 8.0°C min⁻¹ for the first column while second column was set 15°C higher than the first column. The injector temperature was 200°C and an injection volume of 1 µL was employed in the splitless mode. Helium was used as the carrier gas with a constant flow rate of 1 mL min⁻¹.

A time-of-flight mass spectrometer (Pegasus, Leco Corporation, USA) was coupled with GC × GC under 70 eV electron impact ionization for identifying the resolved peaks. The ToFMS was operated at an acquisition rate of 200 spectra/s (200 Hz), with an ion-source temperature of 200°C and a transfer-line temperature of 250°C. The scanned mass range was from 40 to 700 m/z, with a modulation period of 5 s for GC × GC studies.

One dimensional GC-ToFMS was also performed using the same column set as above and with similar conditions as described for the GC × GC TOFMS analysis above. The data acquisition rate of 20 Hz was used.

DATA CONVERSION AND PEAK TABLE GENERATION

For data transformation and visualization, the ToFMS data were first exported in ASCII format before being converted

into a two-dimensional array using an in-house programme. The GC × GC-ToF-MS software was used to find all the peaks in the raw GC × GC chromatogram with a signal-to-noise ratio that was higher than 100.

A library search was carried out for all the peaks using the NIST version 2.0 and the results were combined in a single peak table. Normalization of peak area was employed to estimate the percentage of all the individual components in the analysis of essential oils.

RESULTS AND DISCUSSION

Hydrodistillation of *Litsea fulva* leaves yielded pale yellow oil. Preliminary analysis on one dimensional technique tentatively identified 74 compounds (Table 1). Further analysis using two-dimensional technique resulted in identification of 98 compounds (Table 1). Based on the peak table, 60 components with similarities over 800 were identified. The mass spectral match factors included similarity, reverse factors and probability. The similarity and reverse factors that were above 800 and 900, respectively, indicated an acquired mass spectrum which usually showed a good match with the library spectrum. On the other hand, a probability value of more than 9000 mean that the mass spectrum was highly unique and provisional identification based on mass spectra was possible (Dalluge et al. 2002; Marriot & Shellie 2002; Wu et al. 2004). There were 32 components with a good match (Table 1).

The 98 compounds identified included 30 hydrocarbons, 22 alcohols, five acids, 16 ketones, five aldehydes, 12 esters, six ethers and two other components. It was found that a lot of alcohols (34.09%) and ethers (24.38%) components constituted in the *Litsea fulva* leaf oil. The major components of these oils were cis-Z- α -bisabolene epoxide (9.51%), trans-Z- α -bisabolene epoxide (8.36%), C₁₃H₂₀O₂ (7.39%), longipinocarvone (5.68%), τ -Cadinol (4.24%), C₁₅H₂₄O (4.98%) and α -cadinol (3.95%).

There are 30 hydrocarbons from C₉ to C₂₂ including four saturated and five unsaturated linear hydrocarbons and 21 saturated or partly unsaturated cyclic hydrocarbons consisting of four monoterpenes, 10 sesquiterpenes, four phenyl cyclic compounds and three naphthyl compounds. In addition, sesquiterpene hydrocarbons (6.40%) with molecular weights of 204 were found with predominance among the identification of the 30 hydrocarbons. The main components of the hydrocarbons were γ -muurolene (2.04%), C₁₂H₂₀ (2.03%), cadalene (1.67%) and β -elemene (1.16%).

Among the 22 alcohols from C₆ to C₃₇ there were five saturated and five unsaturated linear alcohols and 12 saturated or partly unsaturated cyclic components, with predominance of molecular weights of 222 and which contained of 19.45% of leaf oil. In addition, C₁₅H₂₄O (4.98%), τ -cadinol (4.24%), α -cadinol (3.95%), C₁₉H₃₆O (3.47%), elemol (3.44%), caryophyllenyl alcohol (3.21%), spathulenol (2.41%) and ledol (2.21%) were identified as the major components of alcohol. There were five aldehydes from C₅ to C₂₃ consisting of two saturated and

TABLE 1. Chemical constituents of *L. fulva* leaf oil based on GC × GC - ToF/MS analysis

Peak	¹ R.T. (s)	² R.T. (s)	Name	Formula	Similarity	Reverse	CAS	Area (%)
Hydrocarbons								9.73
1	465	0.99	α-Pinene *	C ₁₀ H ₁₆	956	963	2437-95-8	0.35
2	820	1.10	δ-Elmene *	C ₁₅ H ₂₄	951	966	20307-84-0	0.19
3	1150	1.18	Cyclopentadecane *	C ₁₅ H ₃₀	949	957	295-48-7	0.09
4	860	0.99	Tetradecane	C ₁₄ H ₃₀	936	942	629-59-4	0.02
5	1380	1.17	1-Docosene	C ₂₂ H ₄₄	931	944	1599673	0.04
6	555	1.05	Limonene	C ₁₀ H ₁₆	926	938	5989-54-8	0.05
7	860	1.18	β-Elmene *	C ₁₅ H ₂₄	922	930	515-13-9	1.16
8	355	0.80	Heptane, 2,4-dimethyl-	C ₉ H ₂₀	921	937	2213-23-2	0.02
9	505	1.04	β-Pinene	C ₁₀ H ₁₆	915	916	127-91-3	0.05
10	995	1.18	Cyclotetradecane	C ₁₄ H ₂₈	914	917	295-17-0	0.02
11	925	1.22	γ-Muurolene *	C ₁₅ H ₂₄	914	926	30021-74-0	2.04
12	915	1.22	Aromadendrene *	C ₁₅ H ₂₄	905	929	109119-91-7	0.63
13	1055	1.04	8-Heptadecene	C ₁₇ H ₃₄	899	904	54290-12-9	0.03
14	900	1.20	(+)-Aromadendrene	C ₁₅ H ₂₄	898	908	489-39-4	0.74
15	920	1.01	3-Dodecene, (E)-	C ₁₂ H ₂₄	888	911	7239-23-8	0.02
16	575	0.89	Undecane	C ₁₁ H ₂₄	877	888	1120-21-4	0.02
17	850	1.12	Copaene *	C ₁₅ H ₂₄	877	881	3856-25-5	0.15
18	840	1.15	Clovene	C ₁₅ H ₂₄	877	886	469-92-1	0.13
19	930	1.00	Pentadecane *	C ₁₅ H ₃₂	874	936	629-62-9	0.15
20	880	1.17	α-Himachalene	C ₁₅ H ₂₄	815	850	3853-83-6	0.43
21	900	1.24	α-Bulnesene	C ₁₅ H ₂₄	796	817	3691-11-0	0.52
22	1045	1.88	Megastigma-4,6(Z),8(Z)-triene	C ₁₃ H ₂₀	772	831	71186-25-9	0.44
23	980	1.34	1H-Indene, 1-ethylideneoctahydro-7a-methyl-, cis-	C ₁₂ H ₂₀	768	797	56362-87-9	2.03
24	940	1.23	α-Muurolene	C ₁₅ H ₂₄	765	777	31983-22-9	0.41
Aromatic Hydrocarbons								3.58
25	550	1.13	o-Cymene	C ₁₀ H ₁₄	913	947	527-84-4	0.02
26	960	1.33	Calamenene *	C ₁₅ H ₂₂	848	886	483-77-2	0.80
27	1065	1.56	Cadalene *	C ₁₅ H ₁₈	826	831	483-78-3	1.67
28	890	1.51	Naphthalene, 2,6-dimethyl-	C ₁₂ H ₁₂	796	911	581-42-0	0.06
29	1025	1.58	Cadina-1(10),6,8-triene	C ₁₅ H ₂₂	778	789	1460-96-4	0.40
30	975	1.39	α-Calacorene	C ₁₅ H ₂₀	777	950	0-00-0	0.63
Alcohols								34.09
31	1120	1.22	1-Hexadecanol *	C ₁₆ H ₃₄ O	943	958	36653-82-4	0.24
32	975	1.33	Elemol *	C ₁₅ H ₂₆ O	942	951	639-99-6	3.44
33	1060	1.19	1-Tridecanol	C ₁₃ H ₂₈ O	931	944	1599-67-3	0.02
34	1215	1.15	1-Hexadecen-3-ol, 3,5,11,15-tetramethyl-	C ₂₀ H ₄₀ O	925	937	0-00-0	0.04
35	1040	1.41	τ-Cadinol *	C ₁₅ H ₂₆ O	906	914	11/1/5937	4.24
36	1110	1.27	cis-7-Tetradecen-1-ol	C ₁₄ H ₂₈ O	887	908	40642-43-1	0.09
37	995	1.29	4aH-cycloprop[e]azulen-4a-ol, decahydro-1,1,4,7-tetramethyl- *	C ₁₅ H ₂₆ O	874	875	95975-84-1	0.52
38	995	1.38	Caryophyllenyl alcohol *	C ₁₅ H ₂₆ O	859	860	0-00-0	3.21
39	700	1.24	p-menth-1-en-8-ol	C ₁₀ H ₁₈ O	850	856	0-00-0	0.02
40	1300	2.00	1-Heptatriacotanol	C ₃₇ H ₇₆ O	830	832	105794-58-9	1.39
41	1005	1.38	(-)-Globulol	C ₁₅ H ₂₆ O	827	837	489-41-8	1.17
42	330	1.16	3-Hexanol, 4-methyl-	C ₇ H ₁₆ O	815	815	615-29-2	0.02
43	1050	1.47	α-Cadinol	C ₁₅ H ₂₆ O	801	815	481-34-5	3.95
44	1125	1.27	9,12-Tetradecadien-1-ol, (Z,E)-	C ₁₄ H ₂₆ O	788	794	51937-00-9	0.03
45	1010	1.38	Ledol	C ₁₅ H ₂₆ O	786	792	577-27-5	2.21
46	1045	1.67	1H-Inden-1-one, 2,3-dihydro-3,4,7-trimethyl-	C ₁₂ H ₁₄ O	776	814	35322-84-0	0.75

(continue)

Continued (TABLE 1)

Peak	¹ R.T. (s)	² R.T. (s)	Name	Formula	Similarity	Reverse	CAS	Area (%)
47	1145	1.66	2-Methyl-E,E-3,13-octadecadien-1-ol	C ₁₉ H ₃₆ O	773	776	0-00-0	3.47
48	730	1.40	1-Butanol, 2,3-dimethyl-	C ₆ H ₁₄ O	764	811	19550-30-2	0.02
49	965	1.29	Epiglobulol	C ₁₅ H ₂₆ O	762	765	0-00-0	0.71
50	1000	1.41	Spathulenol	C ₁₅ H ₂₄ O	756	760	6750-60-3	2.41
51	1040	1.49	Tetracyclo[6.3.2.0(2,5).0(1,8)]tridecan-9-ol, 4,4-dimethyl-	C ₁₅ H ₂₄ O	810	814	0-00-0	4.98
52	1045	1.47	Santalol, cis,à-Carboxylic acids	C ₁₅ H ₂₄ O	751	765	19903-72-1	1.15 0.63
53	310	1.03	Acetic acid, 2-methyl-	C ₄ H ₈ O ₂	890	925	79-31-2	0.06
54	1230	1.33	Palmitic acid	C ₁₆ H ₃₂ O ₂	890	892	57-10-3	0.15
55	765	1.17	Pelargic acid	C ₉ H ₁₈ O ₂	880	891	112-05-0	0.02
56	1110	1.31	Myristic acid	C ₁₄ H ₂₈ O ₂	868	880	544-63-8	0.05
57	965	1.62	1-(3,3-Dimethyl-1-yl)-2,2-dimethylcyclopropene-3-carboxylic acid	C ₁₂ H ₁₆ O ₂	771	796	0-00-0	0.35
			Esters					5.31
58	490	1.60	τ-Valerolactone	C ₅ H ₈ O ₂	947	948	108-29-2	0.03
59	490	1.47	2(5H)-Furanone, 5,5-dimethyl-	C ₆ H ₈ O ₂	892	892	20019-64-1	0.02
60	570	1.51	2(3H)-Furanone, 5-ethenyldihydro-5-methyl-	C ₇ H ₁₀ O ₂	886	893	1073-11-6	0.02
61	400	1.38	β-Angelcalactone	C ₅ H ₆ O ₂	871	874	591-11-7	0.05
62	990	1.46	3-Hexen-1-ol,benzoate, (Z)-*	C ₁₃ H ₁₆ O ₂	850	906	25152-85-6	0.49
63	850	1.37	1,3-2H-Isobenzofuranone, 3,3,7-trimethyl-	C ₁₁ H ₁₂ O ₂	845	852	57732-90-8	0.23
64	1275	1.90	Deoxysericealactone *	C ₆ H ₂₀ O ₄	833	840	19892-19-4	3.18
65	595	1.40	n-Caproic acid vinyl ester	C ₈ H ₁₄ O ₂	826	833	3050-69-9	0.31
66	1030	1.70	2,4-2H-Benzo[c]furanone, 3,3,4-trimethyl-	C ₁₁ H ₁₂ O ₂	782	798	146950-80-3	0.20
67	1045	1.86	Acetic acid, 3-cyclohex-1-enyl-1-methylprop-2-ynyl ester	C ₁₂ H ₁₆ O ₂	780	792	162518-99-2	0.14
68	840	1.51	δ-Undecalactone	C ₁₁ H ₂₀ O ₂	766	791	104-67-6	0.06
69	1070	1.94	Benzo[c]furanone, 3,3,4,7-tetramethyl-	C ₁₂ H ₁₄ O ₂	761	777	37740-08-2	0.58
			Ethers					24.38
70	1,010	1.4	cis-Z-α-Bisabolene epoxide*	C ₁₅ H ₂₄ O	811	814	0-00-0	9.51
71	1060	1.48	Aromadendrene oxide-(1)	C ₁₅ H ₂₄ O	797	830	0-00-0	1.92
72	1160	1.73	trans-Z-α-Bisabolene epoxide	C ₁₅ H ₂₄ O	782	816	0-00-0	8.36
73	970	1.32	Diepi-α-cedrene epoxide	C ₁₅ H ₂₄ O	778	782	0-00-0	0.72
74	950	1.37	1-Oxaspiro[2.5]octane, 5,5-dimethyl-4-(3-methyl-1,3-butadienyl)-	C ₁₄ H ₂₂ O	764	767	0-00-0	2.20
75	930	1.27	Caryophyllene oxide	C ₁₅ H ₂₄ O	751	769	1139-30-6	1.67
			Ketones					10.68
76	700	1.44	Melilotal	C ₉ H ₁₀ O	909	914	122-00-9	0.04
77	1190	1.22	2-Nonadecanone	C ₁₉ H ₃₈ O	887	913	629-66-3	0.09
78	985	1.63	2H-Benzocyclohepten-2-one, 3,4,4a,5,6,7,8,9-octahydro-4a-methyl-, (S)-	C ₁₂ H ₂₈ O	859	878	55103-71-4	0.64
79	760	1.35	Carvenone	C ₁₀ H ₁₆ O	854	898	499-74-1	0.06
80	395	1.45	2-Butanone	C ₄ H ₈ O	829	863	78-93-3	0.06
81	975	1.73	1(2H)-Naphthalenone, 5-ethyl-3,4-dihydro- *	C ₁₂ H ₁₄ O	817	817	51015-31-7	1.16
				C ₇ H ₁₀ O	787	870	30434-64-1	0.05
82	795	1.40	2-Cyclopenten-1-one, 3,4-dimethyl-					
83	1115	1.58	Longipinocarvone	C ₁₅ H ₂₂ O	785	799	0-00-0	5.68
84	800	1.32	2,4-Dimethyl-3-nitrobicyclo[3.2.1]octan-8-one	C ₁₀ H ₁₅ NO ₃	784	803	0-00-0	0.04
85	580	1.45	2-Heptanone, 6-methyl-	C ₈ H ₁₆ O	781	784	928-68-7	0.02
86	865	1.36	3,4-Methylenedioxyphenyl acetone	C ₁₀ H ₁₀ O ₃	768	796	4676-39-5	0.02

(continue)

Continued (TABLE 1)

Peak	¹ R.T. (s)	² R.T. (s)	Name	Formula	Similarity	Reverse	CAS	Area (%)
87	975	1.67	11-Oxatetracyclo[5.3.2.0 (2,7).0 (2,8)] dodecan-9-one	C ₁₁ H ₁₄ O ₂	780	780	0-00-0	0.84
88	830	1.36	3-Cyclopenten-1-one, 2,2,5,5-tetramethyl-	C ₉ H ₁₄ O	777	890	81396-36-3	0.43
89	920	1.28	2-Cyclopenten-1-one, 2-pentyl-	C ₁₀ H ₁₆ O	762	795	25564-22-1	0.59
90	890	1.30	α -Ionone	C ₁₃ H ₂₀ O	759	760	127-41-3	0.19
91	995	1.50	2-Heptanone, 6-(3-acetyl-2-methyl-1- cyclopropen-1-yl)-6-methyl- Aldehydes	C ₁₄ H ₂₂ O ₂	750	759	65868-86-2	0.79 1.48
92	330	1.12	Prenal	C ₅ H ₈ O	938	947	107-86-8	0.04
93	1140	1.22	Tetradecanal *	C ₁₄ H ₂₈ O	930	955	124-25-4	0.23
94	625	1.11	Nonanal	C ₉ H ₁₈ O	920	920	124-19-6	0.03
95	740	1.27	β -citral/Neral	C ₁₀ H ₁₆ O	916	916	106-26-3	0.04
96	1290	2.04	2-[4-methyl-6-(2,6,6-trimethylcyclohex-1- enyl)hexa-1,3,5-trienyl]cyclohex-1-en-1- carboxaldehyde Others	C ₂₃ H ₃₂ O	787	787	0-00-0	1.15 10.12
97	1000	1.46	Oxacyclotetradeca-4,11-diyne	C ₁₃ H ₁₈ O	750	789	6568-32-7	2.74
98	1175	1.71	1b,5,5,6a-Tetramethyl-octahydro-1-oxa- cyclopropa[a]inden-6-one	C ₁₃ H ₂₀ O ₂	750	802	0-00-0	7.39

* Compounds were also identified by one-dimensional GC X GC ToFMS t_r = Retention time using primary and secondary column Rtx-5MS and Rtx-17. The peak in (bold) mean that the identified compound is with good match

two unsaturated linear aldehydes and a partly unsaturated cyclic aldehydes.

Among the 16 ketones, there were three saturated linear ketones and 13 with saturated or partly unsaturated cyclic ketones which was made up of mainly longipinovarvone (5.68%) and C₁₂H₁₄O (1.16%).

There were 12 saturated or partly unsaturated cyclic esters from C₅ to C₁₆, which made up mainly of deoxysericealactone and accounted for 3.18% leaf oil while there were six saturated or partly unsaturated cyclic ethers from C₁₄ to C₁₅ including four phenyl cyclic compounds and two oxygenated sesquiterpenes. There were five carboxylic acids C₄ to C₁₆ including four saturated linear acids and one double-bond unsaturated acid.

For comparative purposes, the preliminary analysis on one-dimensional (1D) GC X GC was used to compare

with two-dimensional (2D) GC X GC results. Forty-two components with similarities above 800 were identified. These components could be classified into 8 classes of compounds without the carboxylic acids (Table 2). The 1-D and 2-D results were found to possess some compositional similarities, which revealed 29 same components (Table 1). The results showed that there was an agreement between the two analysis methods when the peak purity and match quality in 1D were high enough. However, in this study, 1-D and 2-D results were differential with its quantity of the hydrocarbons and the alcohols. These might be due to the comprehensive two-dimensional gas chromatography (GC X GC) which was considered to be a better and more powerful separation tool in GC and an identification tool for analyzing complex volatile oils (Marriot & Shellie 2002; Wu et al. 2004).

TABLE 2. Comparison of one-dimensional and two-dimensional GC X GC TOFMS

Class of compounds	% Area	
	1-D GC X GC	2-D GC X GC
Hydrocarbons	38.83	9.73
Aromatic hydrocarbons	4.23	3.58
Alcohols	25.59	34.09
Aldehydes	1.16	1.48
Ketones	2.42	10.68
Esters	2.00	5.31
Ethers	17.56	24.38
Carboxylic acids	-	0.63
Others	8.22	10.12

* Based of the similarity of all individual components in the leaf oil above 750

CONCLUSION

A qualitative analysis of the individual compounds in *Litsea fulva* essential oils performed by comprehensive two-dimensional gas chromatography (GC × GC) coupled with time-of-flight mass spectrometer (TOF/MS) was found to contain 98 compounds, which included 30 hydrocarbons, 22 alcohols, five acids, 16 ketones, five aldehydes, 12 esters, six ethers and two other compounds. The most abundant of *L. fulva* leaf oil contained alcohols and ethers, with 34.09% and 24.38%, respectively. The major components of these oils were cis-Z- α -bisabolene epoxide (9.51%), trans-Z- α -bisabolene epoxide (8.36%), C₁₃H₂₀O₂ (7.39%), longipinocarvone (5.68%), τ -Cadinol (4.24%), C₁₅H₂₄O (4.98%) and α -cadinol (3.95%). The study also showed that the comprehensive two-dimensional gas chromatography (GC × GC) is a better and more powerful separation tool in GC and an identification tool for analyzing complex volatile oils compared with the one-dimensional GC.

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- Khong Heng Yen*
School of Chemistry and Environmental Studies
Faculty of Applied Sciences
Universiti Teknologi MARA
Jalan Meranek
94300 Kota Samarahan, Sarawak
Malaysia
- Laily B Din, Norzamzurina Ismail & Mohd Ambar Yarmo
School of Chemical Sciences & Food Technology
Faculty of Science and Technology
Universiti Kebangsaan Malaysia
43400 Bangi, Selangor, D.E.
Malaysia
- Zuriati Zakaria
Malaysia Japan International Institute of Technology
Universiti Teknologi Malaysia
54100 Kuala Lumpur
Malaysia
- Nor Hadiani Ismail
Faculty of Applied Sciences
Universiti Teknologi MARA
40450 Shah Alam, Selangor, D.E.
Malaysia
- *Corresponding author; email: khonghy@sarawak.uitm.edu.my

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