



# Trends in Phytochemical Research (TPR)

Journal Homepage: <http://tpr.iau-shahrood.ac.ir>



Original Research Article

## Chemical compositions of essential oils from *Etlingera brevilabrum*: A comparative analysis using GC×GC/TOFMS

BEHNAM MAHDAVI ✉

Department of Chemistry, Faculty of Science, Hakim Sabzevari University, Sabzevar, Iran

### ABSTRACT

In this report, essential oils from the rhizome, stem and leaf of *Etlingera brevilabrum* were isolated using a Clevenger-type apparatus and subsequently characterized using a comprehensive two-dimensional gas chromatography (GC×GC/TOFMS) approach. Regarding the spectral assignments, 77 constituents were totally identified. The rhizome oil contained oxygenated monoterpenes (26.4%) and monoterpene hydrocarbons (25.9%), while the total percentages of these groups in the stem oil were found to be 27.5% and 25.2%, respectively. The leaf oil dominated by monoterpene hydrocarbons (32.4%) and oxygenated monoterpenes (27.3%). The major characterized compounds for the rhizome oil were 1,8-cineole (10.8%) and β-phellandrene (8.8%). The stem oil was mainly composed of 1,8-cineole (12.0%) and β-pinene (8.5%). However, monoterpene hydrocarbons involving β-pinene and α-pinene (8.7% and 5.5%) were identified as the main components in the leaf oil profile.

© 2017 Islamic Azad University, Shahrood Branch Press, All rights reserved.

### ARTICLE HISTORY

Received: 29 January 2017  
Revised: 19 February 2017  
Accepted: 26 February 2017  
ePublished: 01 March 2017

### KEYWORDS

Comprehensive 2D gas chromatography  
*Etlingera brevilabrum*  
Essential oil  
1,8-Cineole  
Hydrodistillation

### 1. Introduction

Conventional one-dimensional gas chromatography, which is usually used to identify constituents of different mixtures, does not provide sufficient separation for highly complex mixtures such as petrochemical samples, food stuffs and natural tissues extracts (Dallüge et al., 2002a). There are many reports in literature using 1D gas chromatography for the analysis of essential oils from plant materials (Mohammadhosseini et al., 2016; Mohammadhosseini et al., 2013; Mohammadhosseini et al., 2015). The essential oils are usually composed of terpenoid compounds and their derivatives. The structural differences between these groups of compounds are minimal and also their MS spectral patterns are very similar in many cases. Therefore, the peaks identification of these compounds is very difficult or even impossible in some cases (Oprean et al., 1998). Comprehensive two-dimensional gas chromatography (GC×GC) is a method which can distinctly increase the general performance of

separation and the subsequent analysis (Phillips and Beens, 1999). Recently, comprehensive 2D GC in combination with time-of-flight mass spectrometry (GC×GC/TOFMS) has gained much attention as a new technology due to its more chemical selectivity than GC/MS. Therefore, this strategy can be preferably considered as an appropriate option to the analysis of metabolites (Hope et al., 2005). Not only GC×GC/TOFMS is known as an excellent instrument to detect components in complex mixtures, but also it is capable of generating trilinear data. Using this type of data, it is handy to deconvolute individual compounds from a group, partially those overlapped together from only one sample (Sinha et al., 2004).

In principle, the GC×GC/TOFMS method consists of two GC systems (GC×GC) having columns with different polarities. The columns are connected by an interface with an integrated cryogenic trap. The trap continually condenses compounds eluted from the primary column and releases them from time to time as short pulses to the secondary column. Parameters such as duration and frequency of both condensation

✉ Corresponding author: Behnam Mahdavi  
Tel: +98-51-44013319; Fax: +98-51-44012669  
E-mail address: [b.mahdavi@hsu.ac.ir](mailto:b.mahdavi@hsu.ac.ir)

and injection pulses are variable and allow precise tuning of the instrument according to the general requirements of the respective analyses. Since the GC×GC produces very narrow peaks (down to 50 ms), depending on the frequency of cryogenic modulation, a TOF-MS detector with a high acquisition rate is required. The pulsed nature of an ionization source of the TOF-MS system highly enhances the system accuracy by avoiding spectral skewing common in a continuous ionization mode. Therefore, GC×GC combined with TOF-MS detection operates with a high precision independent of the used concentration range (Kalinová et al., 2006; Van-Deursen et al., 2000).

*Etilingera* species are known as the largest genus of *Zingiberaceae* family. The plants of this genus can grow up to 6 m in height (Chan et al., 2008). Furthermore, they have different commercial and traditional uses. In Sabah, Malaysia, natives have used the young shoots, flower, and fruits of *E. elatior*, *E. littoralis*, and *E. rubrolutea* as condiment from long years ago. Fruits and young stems of *E. littoralis*, and flowers of *E. maingayi* are widely consumed in Thailand (Chan et al., 2007). *E. brevilabrum* is characterized with the presence of many purple spots on the upper and under sides of its leaves, especially on the new ones as in Fig. 1. The plant has some medicinal and pharmaceutical uses. Plant leaves are used against long-lasting fever for children by rubbing the body with the roasted leaves. In addition, sap from shoot of *E. brevilabrum* is frequently used to cure some eye problems, while the corresponding base is prescribed as an effective medicine against stomachache. Furthermore, leaves of *E. brevilabrum* are pounded and applied to an infected skin leg that looks dry (Poulsen, 2006).



Fig. 1. *Etilingera brevilabrum* species (Poulsen, 2006).

In this study, using a GC×GC/TOF-MS instrument, chemical compositions of the essential oils from three parts of *E. brevilabrum* (rhizome, stem and leaf) were analyzed. The identified chemical profiles of the essential oils have been also compared with the previous reports using GC-FID and GC-MS systems. Moreover, chemical composition of essential oils from other *Etilingera* species have been discussed in due parts.

## 2. Experimental

### 2.1. Plant material

The different parts of *E. brevilabrum* including rhizomes, stems, and leaves were collected in May 2009 from its natural habitat across the road of Kampung Kipandi, Sabah, Malaysia (GPS: 5°87'13" N, 116°25'02" E) which was situated alongside the Penampang-Tambunan road at an altitude of 600-700 m on the Crocker range. The collected plant species was recognized by Mr. Sani Miran, a botanist of the Universiti Kebangsaan Malaysia. A voucher specimen for the plant (WYA 500) was deposited at the Universiti Kebangsaan Malaysia Herbarium (HUKMB).

### 2.2. Essential oils extraction

The different parts of *E. brevilabrum* were dried and ground to a fine powder. Then, one hundred gram portion from each part was separately subjected to hydrodistillation in a Clevenger-type apparatus for about 5 h. The distilled essential oils were taken up in *n*-hexane (Merck), dried over anhydrous sodium sulphate (Merck) until the last traces of water were removed. The obtained oils were stored in tightly closed vials at 4-6 °C before analysis. The mean yields of the essential oils were 0.03%, 0.07%, and 0.24% (w/w), based on three replicate extractions for rhizome, stem and leaf, respectively.

### 2.3. GC×GC-ToF-MS analysis

The isolated essential oils were subjected to the GC×GC-ToF-MS LECO Pegasus 4D (LECO, St. Joseph, MI, USA). This system consisted of an Agilent GC 6890N gas chromatograph with a dual stage jet cryogenic modulator (licensed from Zoex) and a secondary oven. A high-speed ToF mass spectrometer was used as the detector. A Rtx-5MS, 30 m×250 µm I.D., 0.1 µ film thickness (Restek) was used as the first dimension column and a Rxi17, 0.79 m×100 µm I.D., 0.1 µ film thickness (Restek) was used as a second-dimension column. In each analysis, ultrapure Helium as the carrier gas flowed with a constant rate of 1.0 mL/min. The primary oven temperature was programmed from 45 °C (2 min.) to 230 °C (5 min.) at 4 °C/min intervals, whereas the secondary oven temperature was programmed from 50 °C (2 min.) to 235 °C (5 min.) applying a 3 °C/min ramp. The temperatures of MS transfer line and MS source temperature were respectively adjusted at 250 °C and 220 °C. The modulation time was 5 s and the modulator temperature was kept at 30 °C offset relative to the secondary oven. The ToF-MS system was operated at a spectrum storage rate of 100 spectra/second. The mass spectrometer was operated in the EI mode at 70 eV over the range 50-400 m/z under a voltage of 1600 V.

Total ion chromatograms (TIC) were processed using an automated data processing software Chroma TOF (LECO) at S/N threshold 500. Contour plots were used to evaluate the general quality of the separation and peak identification manually.

One commercial database (US National Institute of Science and Technology (NIST) V. 2.0-Mainlib and Replib) was used. The key parameters involving mass spectral match factor, similarity, reverse > 800 and probability > 1000, were used to decide whether a peak was correctly identified or not. The detection information including retention times, similarity, reverse, and probability, make the reliability of each analysis significantly increased. The similarity and reverse factors indicate how well a mass spectrum matches the library spectral patterns. Since the organic isomers have similar mass spectra, a complimentary criterion, namely the probability is often used to determine whether the peaks with the same name belong to one compound or several compounds (Wu et al., 2004). Practically speaking, a similarity and reverse number above 800, indicate that an acquired mass spectrum usually shows a good match with the library spectrum (Zhu et al., 2005). On the other hand, the probability values above 9000 indicate that the mass spectra are highly unique and the corresponding identification would be justifiable (Dallüge et al., 2002b). The GC×GC total ion contour (TIC) plots of the essential oils from *E. brevilabrum* under different column systems and the optimized separation conditions are presented in Fig. 2.

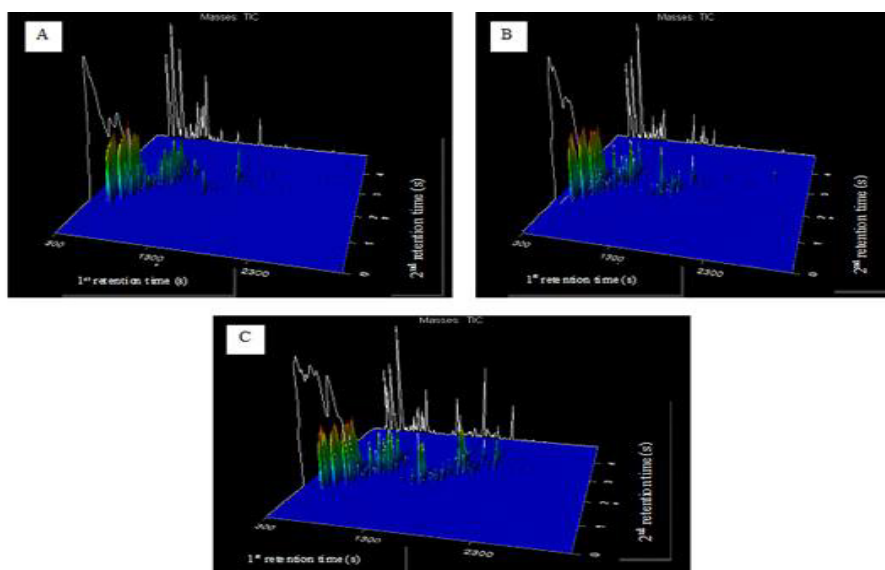
### 3. Results and Discussion

#### 3.1. Profiling of essential oils of *E. brevilabrum*

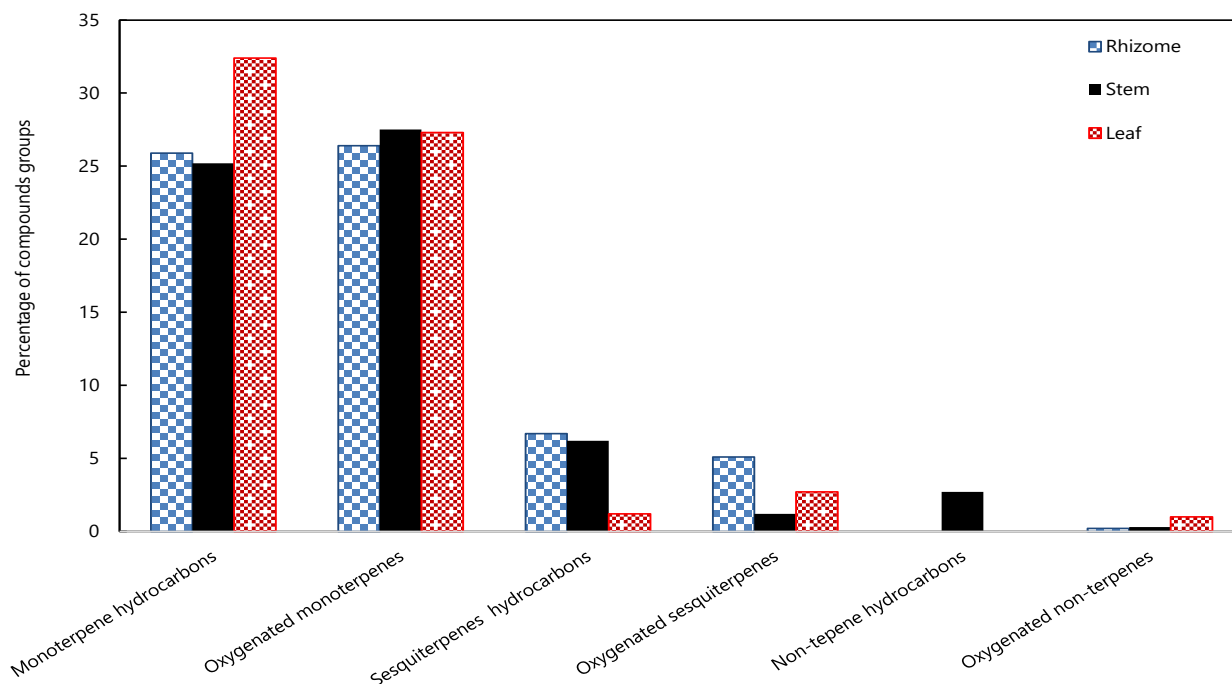
A total of 77 specified compounds were

recognized which could be classified into 6 groups including monoterpene hydrocarbons (23 compounds), oxygenated monoterpenes (34 compounds), sesquiterpene hydrocarbons (12 compounds), oxygenated sesquiterpenes (2 compounds), non-terpene hydrocarbons (2 compounds), and oxygenated non-terpene hydrocarbons (4 compounds). For the leaf oil, most compounds belong to monoterpene hydrocarbons (32.4%), but rhizome and stem oils were dominated by the presence of oxygenated monoterpenes (26.4 and 27.5%, respectively). This study revealed that oxygenated monoterpenes (27.3%) were the second class of compounds for the leaf oil, whereas monoterpene hydrocarbons were the second class in the rhizome and stem oils (25.9 and 25.2%, respectively). It was also noted that the amount of sesquiterpene hydrocarbons for the leaf oil (1.1%) was less than the rhizome and stem oils. In addition, oxygenated sesquiterpenes for the rhizome and leaf oils exhibited higher percentages than the stem oils. The results have been demonstrated in Fig. 3.

Chemical compositions of the oils from rhizome, stem, and leaf of *E. brevilabrum* are shown in Table 1. As mentioned earlier, all the analyzed essential oils contained large proportions of monoterpene hydrocarbons and oxygenated monoterpenes. However, there were some differences in the amount of the main components in the studied profiles. The most abundant monoterpene compounds in the rhizome oil were  $\beta$ -phellandrene (8.8%), camphene (5.3%), and  $\beta$ -myrcene (2.8%), respectively; while  $\beta$ -pinene (8.5%), *trans*- $\beta$ -ocimene (3.5%), and 3-methyl-apopinene (3.1%), were among the major monoterpene hydrocarbons in the stem oil. In the leaf oil of *E. brevilabrum*,  $\beta$ -pinene (8.7%),  $\alpha$ -pinene (5.5%), and limonene (2.7%) were identified as main monoterpene hydrocarbons contributing to the oil profile. The



**Fig. 2.** The GC×GC total ion contour (TIC) plots of essential oils from different parts of *Etingera brevilabrum*. A: The leaf oil, B: The stem oil, C: The rhizome oil.



**Fig. 3.** Percentage of six constituting-groups occurring in the essential oils of rhizome, stem, and leaf of *E. brevilabrum*.

prevailing oxygenated monoterpenes in the rhizome oil were 1,8-cineole, myrtenal, and myrcenol (10.8%, 1.8%, and 1.6% respectively). Additionally, 1,8-cineole, *p*-menth-1-en-8-ol, and *exo*-fenchol (12.0%, 2.5%, and 2.1% respectively) were found as oxygenated monoterpenes with high percentages in stem oil, while in the leaf oil, the main oxygenated monoterpenoids were *p*-menth-1-en-8-ol, myrtenal, and 6,6-dimethyl-2-methylenebicyclo[3.1.1]heptan-3-one (4.7%, 3.8%, and 3.8% respectively). Caryophyllene oxide was recognized as the major oxygenated sesquiterpene occurring in all oil profiles (rhizome: 3.9%, stem: 1.1% and leaf: 2.7%).

### 3.2. Comparison of the chemical profiles of *E. brevilabrum* with the previous reports

In our previous study on the essential oils of *E. brevilabrum* using GC-FID and GC-MS techniques, the rhizome oil was made up of oxygenated monoterpenes (40.2%), monoterpene hydrocarbons (28.2%), oxygenated sesquiterpenes (19.8%), and sesquiterpene hydrocarbons (7.0%), respectively. For the stem oil, the decreasing order was as follows. Monoterpene hydrocarbons (66.8%)>oxygenated monoterpenes (13.8%)>oxygenated sesquiterpenes (7.3%)>sesquiterpene hydrocarbons (4.8%). For the leaf oil, the total order of the compounds groups was similar to the stem profile (Mahdavi et al., 2013). A previous study on Malaysian *E. elatior* showed that its rhizome and stem oils were dominated by oxygenated monoterpenes (47.3 and 54.3%) and monoterpene hydrocarbons (35.0 and 33.6%), whereas sesquiterpene hydrocarbons (45.1%) and

monoterpene hydrocarbons (29.8%) showed higher percentage in the plant leaf oil (Jaafar et al., 2007). Phenylpropanoid derivatives (55.5-67.4%) were the main group of compounds for the leaf oil of *E. cevuga* (Vahirua-Lechat et al., 1993). Phenolic compounds (95.7%) were reported to be the major constituents in the rhizome oil of *E. punicea* (Tadtong et al., 2009).

$\beta$ -Pinene (52.6%),  $\alpha$ -thujene (28.6%) and *o*-cymene (7.8%) were identified as the major components in the oils from the dried leaves of *E. brevilabrum* using GC-FID and GC-MS. For the stem oil, limonene (28.6%),  $\beta$ -pinene (21.6%), and  $\alpha$ -thujene (13.9%); and for the rhizome oil, 1,8-cineole (27.6%),  $\beta$ -pinene (13.4%), caryophyllene oxide (10.5%) and  $\alpha$ -thujene (10.1%) were the main compounds (Mahdavi et al., 2013). In another study, we reported the chemical composition of the essential oils from fresh rhizomes, stolon, stems, and leaves of *E. brevilabrum*. Accordingly, the main compounds in the leaves oil were  $\alpha$ -thujene (38.1%), *p*-cymen-7-ol (8.0%), and  $\beta$ -pinene (7.8%). However, we found high contents of  $\delta$ -3-carene (43.2%) and  $\alpha$ -thujene (17.7%) in the stem oil. On the other hand, perilla aldehyde (19.6%), bornyl acetate (17.7%), and verbenyl acetate (6.7%) were reported as the major components in the oils isolated from the plant rhizomes. The stolon oil of *E. brevilabrum* comprised  $\beta$ -pinene (30.6%), *p*-cymen-7-ol (25.0%), and *exo*-fenchol (8.2%), as well (Mahdavi et al., 2016). In another study, dealing with the analysis of the essential oil from the rhizome of *E. punicea*, high contents of methyl chavicol (95.7%) were characterized (Tadtong et al., 2009). The leaf oil of *E. cevuga* from different locations was dominated by methyl eugenol (40.9-45.7%), (*E*)-methyl isoeugenol (8.6%-16.5%), eugenol

**Table 1**  
Percentage composition along with the similarity, reverse, and probability of each characterized constituent in the essential oils from the rhizomes, stems, and leaves of *Etingera brevilabrum* using GC×GC-ToF-MS technique.

Compounds name	Rhizome			Stem			Leaf					
	Similarity*	Reverse	Probability	Area %	Similarity	Reverse	Probability	Area %	Similarity	Reverse	Probability	Area %
Monoterpenes												
Tricyclene	927	927	4856	0.2	897	897	2664	0.1	-	-	-	0
1-Isopropyl-4-methylbicyclo[3.1.0]hex-2-ene	921	921	6801	0.6	909	909	6353	0.3	917	917	6441	0.5
α-Pinene	-	-	-	0	914	914	3888	4.1	834	840	4585	5.5
Cyclofenchene	844	844	1456	1.6	-	-	-	0	-	-	-	0
3-Methyl-apopinene	-	-	-	0	822	830	1724	3.1	818	818	1417	0.7
trans-β-Ocimene	-	-	-	0	822	830	1724	3.7	-	-	-	0
α-Thujene	-	-	-	0	-	-	-	0	820	820	5301	0.3
Ocimene	-	-	-	0	838	844	1523	0.3	-	-	-	0
Camphene	929	931	4835	5.3	855	855	5240	1.6	927	931	4973	1.8
4-Methylene-1-(1-methylethyl)-bicyclo[3.1.0]hex-2-ene	815	815	2914	0.1	817	817	2626	0.2	832	834	2758	0.7
Sabinene	931	931	5036	0.6	873	873	4740	0.7	814	814	4262	0.4
β-Myrcene	837	837	5472	2.8	-	-	-	0	-	-	-	0
β-Pinene	864	864	3961	1.8	893	893	3127	8.5	958	961	6087	8.7
α-Phellandrene	-	-	-	0	904	907	6949	0.2	899	902	5456	1.6
m-Cymene	952	953	5723	1.0	961	962	5909	1.0	830	928	4307	1.2
o-Cymene	-	-	-	0	-	-	-	0	918	929	3837	2.0
Limonene	882	882	4162	0.6	-	-	-	0	863	863	5005	2.7
β-Phellandrene	821	833	3715	8.8	-	-	-	0	913	913	6657	2.5
β-cis-Ocimene	912	912	4065	0.4	923	923	4109	0.6	906	906	4207	0.1
l-Terpinene	874	874	4909	0.3	901	901	5995	1.0	916	916	6072	1.9
4-Methyl-3-(1-ethylethylidene)-Cyclohexene	-	-	-	0	902	902	2395	0.2	923	923	1780	1.3
Dehydro-p-cymene	-	-	-	0	-	-	-	0	907	907	3078	0.4
Dihydromyrcene	806	824	2128	1.8	-	-	-	0	-	-	-	0
Oxygenated monoterpenes												
1,8-Cineole	948	948	9538	10.80	863	866	9602	12.00	-	-	-	0
5-Isopropyl-2-methylbicyclo[3.1.0]hexan-2-ol	840	840	3325	0.23	852	852	3789	0.22	830	831	4086	0.71
Fenchone	951	951	8345	0.30	947	947	8257	0.42	925	925	5289	0.13
α-Pinene epoxide	886	886	7174	0.23	-	-	-	0	-	-	-	0
β-Linalool	-	-	-	0	852	852	7278	0.48	-	-	-	0
Perillene	826	832	7719	0.29	-	-	-	0	803	808	5728	0.49
exo-Fenchol	960	960	6554	0.97	967	967	6506	2.07	928	928	5990	0.62
α-Campholenal	895	899	9083	0.50	890	893	9061	0.43	906	918	9297	1.86
trans-Limonene oxide	824	835	2654	0.04	-	-	-	0	-	-	-	0
Pinocarveol	916	916	9368	1.31	899	899	8940	0.93	931	931	9477	3.16
Camphor	942	946	4072	0.76	926	931	5123	0.12	-	-	-	0
cis-Verbenol	830	830	2942	1.23	823	823	2870	0.64	828	828	1948	1.63
2,6,6-Trimethyl-bicyclo[3.1.1]heptan-3-one	909	909	6776	0.03	912	912	7444	0.07	927	927	7010	1.30
6,6-Dimethyl-2-methylene-bicyclo[2.2.1]heptan-3-one	848	849	6799	1.3	852	853	5023	1.3	840	844	5330	3.8
Pinocarvone	-	-	-	0	-	-	-	0	801	804	5133	0.2
Isoborneol	949	952	3671	0.9	906	909	3803	0.7	-	-	-	0
Borneol	-	-	-	0	-	-	-	0	848	848	5136	0.6
3-Pinanone	873	880	7416	0.2	908	909	7766	0.8	927	929	7754	2.4

Table 1 (Continued)

$\alpha$ -Terpineol	-	-	0	915	915	6466	2.5	944	944	6686	4.7
Myrcenol	943	943	1.7	-	-	-	0	-	-	-	0
$\beta$ -Terpinyl acetate	899	908	0.8	-	-	-	0	-	-	-	0
<i>p</i> -Cymen-8-ol	-	-	0	-	-	-	0	869	869	8239	0.2
Myrtenol	928	928	1.4	909	909	8447	1.2	911	911	9326	3.2
Myrtenal	952	953	1.8	951	951	9136	1.5	933	935	9154	3.8
<i>trans</i> -Piperitol	-	-	0	-	-	-	0	811	818	6999	0.3
Verbenone	-	-	0	-	-	-	0	876	876	5402	0.2
<i>cis</i> -Carveol	875	875	0.1	-	-	-	0	892	892	7336	0.4
Carvone	899	901	0.2	877	889	4298	0.1	863	897	5402	0.3
Myrtenyl acetate	-	-	0	-	-	-	0	801	801	3657	0.1
<i>p</i> -Cymen-7-ol	-	-	0	-	-	-	0	855	855	6407	0.1
Sesquiterpenes											
$\alpha$ -Copaene	872	872	0.3	875	875	3598	0.3	-	-	-	0
$\alpha$ -Gurjunene	866	876	1.7	882	890	3594	0.1	-	-	-	0
$\beta$ -Cadinene	810	810	1.8	-	-	-	0	-	-	-	0
$\alpha$ -Santalene	878	879	0.2	-	-	-	0	-	-	-	0
$\beta$ -Caryophyllene	927	927	1.1	947	947	4668	2.3	925	925	4210	1.1
( <i>Z,E</i> )- $\alpha$ -Farnesene	898	900	0.1	888	890	4004	1.0	-	-	-	0
$\alpha$ -Caryophyllene	916	916	0.1	920	920	7408	0.3	862	862	6210	0.1
$\tau$ -Gurjunene	869	871	0.2	2023	-	-	0	-	-	-	0
Germacrene D	872	877	0.5	898	909	5883	0.9	-	-	-	0
$\tau$ -Elemene	-	-	0	844	847	1194	0.6	-	-	-	0
$\tau$ -Muurolene	849	857	0.3	-	-	-	0	-	-	-	0
$\delta$ -Cadinene	872	880	0.5	876	883	4616	0.7	-	-	-	0
Oxygenated Sesquiterpenes											
Spathulenol	815	828	0.3	-	-	-	0	-	-	-	0
Caryophyllene oxide	842	842	4.8	876	899	6094	1.1	901	923	7460	2.6
Nonterpenes hydrocarbons											
3,4-dimethyl-1,5-	-	-	0	842	842	4002	1.5	-	-	-	0
Cyclooctadiene	-	-	0	837	837	2108	1.2	-	-	-	0
5-methyl-1,3,6-Heptatriene	-	-	0	837	837	2108	1.2	-	-	-	0
Oxygenated Nonterpenes											
2-Pentyl-furan	894	894	0.1	892	892	8049	0.1	-	-	-	0
2-Nonanone	-	-	0	904	904	7192	0.1	914	914	7670	0.1
Norinone	922	922	0.2	888	888	6114	0.1	933	933	7070	0.8
4-(1-methylethyl)-2-	-	-	0	-	-	-	0	873	873	6915	0.1
Cyclohexen-1-one	-	-	0	-	-	-	0	873	873	6915	0.1

\*Similarity and reverse &gt; 800 and probability &gt; 1000 were used to decide whether a peak was correctly identified or not.

(3.6-8.4%), and (*Z*)-methyl isoeugenol (0.8-1.5%) (Vahirua-Lechat et al., 1993). A similar study presented the rhizome oil of *E. cougar* contained methyl eugenol (47.4%), (*E*)-methyl isoeugenol (18.2%),  $\alpha$ -pinene (3.9%), and  $\beta$ -pinene (3.0%) (Vahirua-Lechat et al., 2010). The rhizomes oil of *E. sphaerocephala* var. *grandiflora* was characterized by 1,8-cineole (16.8%),  $\alpha$ -phellandrene (12.7%), and (*E*)- $\beta$ -ocimene (8.9%). For the stems oil of the plant, 1,8-cineole (17.4%),  $\alpha$ -phellandrene (9.7%), and  $\alpha$ -pinene (9.5%) were found with rather high percentages. The leaf oil mainly consisted of  $\alpha$ -phellandrene (12.3%), diprene (10.3%), and pseudolimonene (4.6%) (Yahya et al., 2010). The leaf oil of *E. linguiforme* was dominated by the presence of 1,8-cineole (39.7%),  $\beta$ -pinene (13.3%),  $\alpha$ -pinene (7.8%), linalool (7.4%),  $\beta$ -elemene (6.6%), and  $\alpha$ -selinene (5.5%). However, methyl chavicol (49.9%), methyl eugenol (32.3%), and  $\beta$ -pinene (4.7%) were detected as major constituents in the rhizome oil of the plant (Bhuiyan et al., 2010). The leaf oil of Malaysian *E. littoralis* was composed of (*E*)-methyl isoeugenol (37.7%),  $\beta$ -pinene (30.4%), and  $\beta$ -phellandrene (8.6%); while (*E*)-methyl isoeugenol (58.1%) and sandaracopimara-8(14), 15-diene-3 $\beta$ -ol (9.1%) were the major constituents of the rhizome oil. The leaf oil of Malaysian *E. elatior* collected from Penang Botanic Garden, was marked by myrcene (13.5%),  $\alpha$ -humulene (11.8%), and  $\beta$ -caryophyllene (10.7%); the rhizome oil was rich in camphene (18.0%),  $\beta$ -pinene (16.9%), and  $\alpha$ -pinene (8.6%). The leaf oil of *E. elatior* var. *Thai queen* was characterized by high percentages of  $\alpha$ -pinene (24.4%), dodecanol (18.9%), and dodecanal (15.9%); while camphene (15.1%), dodecanol (12.9%), dodecanal (10.6%), bornyl acetate (10.7%), and  $\beta$ -pinene (9.1%) were the main compounds in the rhizome oil (Wong et al., 2010). Jaafar et al. (2007) reported chemical constituent of the essential oils of different parts from *E. elatior* which were collected from different regions of Malaysia (Kampong Paya; Kepala Batas; and Penang). For the leaf oil, (*E*)- $\beta$ -farnesene (27.9%),  $\beta$ -pinene (19.7%), caryophyllene (15.4%); and for the stems oil 1,1-dodecanediol diacetate (34.3%), (*E*)-5-dodecene (27.0%), and decanal (16.5%) were the major constituents. The essential oil from the whole plant of the other Malaysian *E. elatior*, that was collected from Selangor was dominated by  $\beta$ -pinene (24.9%), 1-dodecene (24.3%), 2,6,6-trimethyl-bicyclo[3.1.1]hept-2-ene (11.6%), and dodecanal (8.2%) (Abdelwahab et al., 2010). Other studies on *Etilingera* species have expressed that non-terpenic compounds such as (*Z*)-9-hexadecen-1-ol, cyclotetradecane, *n*-dodecyl acetate, cyclododecane, for *E. fulgens* and dodecanoic acid, cyclododecane, and (*E*)-2-tetradecene for *E. venusta* which were the most abundant components of the volatile oils from the different parts of the *Etilingera* plants (Khaleghi et al., 2012a, b).

#### 4. Concluding remarks

Among the 77 identified compounds in the essential oils of *E. brevilabrum*, fifty two compounds were found in its rhizome oil. The rhizome oil was dominated by oxygenated monoterpenes and monoterpene hydrocarbons (26.4 and 25.9%). 1,8-Cineole (10.8%) was the major compound of the rhizome oil. Forty eight compounds contributed to the stem and leaf oil profiles. Similar to the rhizome oil, the stem oil mainly consisted of oxygenated monoterpenes and monoterpene hydrocarbons (27.5 and 25.2%). 1,8-Cineole (12.0%) was the main component in the stem oil. Monoterpene hydrocarbons and oxygenated monoterpenes (32.4 and 27.3%) were identified as the major groups for the leaf oil.  $\beta$ -Pinene (8.7%) was detected as the main component for the leaf oil. According to the findings of the current report, *E. brevilabrum* can be considered as a natural source of oxygenated monoterpenes and monoterpene hydrocarbons.

#### Conflict of interest

The author declares that there is no conflict of interest.

#### References

- Abdelwahab, S.I., Zaman, F.Q., Mariod, A.A., Yaacob, M., Ahmed Abdelmageed, A.H., Khamis, S., 2010. Chemical composition, antioxidant and antibacterial properties of the essential oils of *Etilingera elatior* and *Cinnamomum pubescens* Kochummen. *J. Sci. Food. Agric.* 90(15), 2682-2688.
- Bhuiyan, M.I., Chowdhury, J., Begum, J., Azim, M., 2010. Aromatic plants of Bangladesh: constituents of leaf and rhizome oil of *Etilingera linguiforme*. *Dhaka Uni. J. Sci.* 58(1), 13-15.
- Chan, E., Lim, Y., Omar, M., 2007. Antioxidant and antibacterial activity of leaves of *Etilingera* species (Zingiberaceae) in Peninsular Malaysia. *Food Chem.* 104(4), 1586-1593.
- Chan, E., Lim, Y., Wong, L., Lianto, F., Wong, S., Lim, K., Joe, C., Lim, T., 2008. Antioxidant and tyrosinase inhibition properties of leaves and rhizomes of ginger species. *Food Chem.* 109(3), 477-483.
- Dallüge, J., van Rijn, M., Beens, J., Vreuls, R.J.J., Brinkman, U.A.T., 2002a. Comprehensive two-dimensional gas chromatography with time-of-flight mass spectrometric detection applied to the determination of pesticides in food extracts. *J. Chromatogr. A* 965(1-2), 207-217.
- Dallüge, J., van Stee, L.L.P., Xu, X., Williams, J., Beens, J., Vreuls, R.J.J., Brinkman, U.A.T., 2002b. Unravelling the composition of very complex samples by comprehensive gas chromatography coupled to time-of-flight mass spectrometry: Cigarette smoke. *J. Chromatogr. A* 974(1-2), 169-184.



- Hope, J.L., Prazen, B.J., Nilsson, E.J., Lidstrom, M.E., Synovec, R.E., 2005. Comprehensive two-dimensional gas chromatography with time-of-flight mass spectrometry detection: analysis of amino acid and organic acid trimethylsilyl derivatives, with application to the analysis of metabolites in rye grass samples. *Talanta* 65(2), 380-388.
- Jaafar, F.M., Osman, C.P., Ismail, N.H., Awang, K., 2007. Analysis of essential oils of leaves, stems, flowers and rhizomes of *Etlingera elatior* (Jack) R. M. Smith. *Malay. J. Anal. Sci.* 11(1), 269-273.
- Kalinová, B., Jiros, P., Zďárek, J., Wen, X., Hoskovec, M., 2006. GC×GC/TOF MS technique-A new tool in identification of insect pheromones: Analysis of the persimmon bark borer sex pheromone gland. *Talanta* 69(3), 542-547.
- Khaleghi, F., Yaacob, W., Din, L.B., Khalilzadeh, M.A., 2012a. Chemical compositions of several parts of *Etlingera venusta* from Malaysia. *J. Essent. Oil-Bear Plants* 15(5), 686-693.
- Khaleghi, F., Yaacob, W., Din, L.B., Khalilzadeh, M.A., 2012b. Volatile oil compositions of several parts of *Etlingera fulgens* from Malaysia. *J. Essent. Oil-Bear. Plants* 15(2), 180-185.
- Mahdavi, B., Yaacob, W., Din, L.B., Aisha, M.S., 2013. Essential oil composition of three air-dried parts of *Etlingera brevilabrum*. *J. Essent. Oil-Bear. Plants* 16(1), 17-22.
- Mahdavi, B., Yaacob, W., Din, L.B., Lee, Y.H., Nazlina, I., 2016. Chemical composition, antioxidant, and antibacterial activities of essential oils from *Etlingera brevilabrum* Valetton. *Rec. Nat. Prod.* 10(1), 22-31.
- Mohammadhosseini, M., Akbarzadeh, A., Shafaghat, A., Hashemi-Moghaddam, H., Mohammadi Nafchi, A., Ashouri, H., 2016. Chemical composition of the essential oils from flowers and leaves of *Marsdenia erecta* using microwave assisted hydrodistillation technique. *J. Essent. Oil-Bear. Plants* 19(4), 863-874.
- Mohammadhosseini, M., Mahdavi, B., Akhlaghi, H., 2013. Characterization and chemical composition of the volatile oils from aerial parts of *Eryngium bungei* Bioss.(Apiaceae) by using traditional hydrodistillation, microwave assisted hydrodistillation and head space solid phase microextraction methods prior to GC and GC/MS analyses: A comparative approach. *J. Essent. Oil-Bear. Plants* 16(5), 613-623.
- Mohammadhosseini, M., Mahdavi, B., Shahnama, M., 2015. Chemical composition of essential oils from aerial parts of *Ferula gummosa* (Apiaceae) in Jajarm Region, Iran using traditional hydrodistillation and solvent-free microwave extraction methods: A comparative approach. *J. Essent. Oil-Bear. Plants* 18(6), 1321-1328.
- Oprean, R., Tamas, M., Sandulescu, R., Roman, L., 1998. Essential oils analysis. I. Evaluation of essential oils composition using both GC and MS fingerprints. *J. Pharmaceut. Biomed.* 18(4-5), 651-657.
- Phillips, J.B., Beens, J., 1999. Comprehensive two-dimensional gas chromatography: a hyphenated method with strong coupling between the two dimensions. *J. Chromatogr. A* 856(1-2), 331-347.
- Poulsen, A.D., 2006. *Etlingera* of Borneo, 1st Ed. Natural History Publications (Borneo) in Association with Royal Botanic Garden Edinburgh, Kota Kinabalu. 263p.
- Sinha, A.E., Fraga, C.G., Prazen, B.J., Synovec, R.E., 2004. Trilinear chemometric analysis of two-dimensional comprehensive gas chromatography-time-of-flight mass spectrometry data. *J. Chromatogr. A* 1027(1-2), 269-277.
- Tadtong, S., Wannakhot, P., Poolsawat, W., Athikomkulchai, S., Ruangrunsi, N., 2009. Antimicrobial activities of essential oil from *Etlingera puniceae* rhizome. *J. Health. Res.* 23(2), 77-79.
- Vahirua-Lechat, I., Francois, P., Menut, C., Lamaty, G., Bessiere, J.M., 1993. Aromatic plants of French Polynesia. I. Constituents of three Zingiberaceae: *Zingiber zerumbet* Smith, *Hedychium coronarium* Koeing and *Etlingera cevuga* Smith. *J. Essent. Oil. Res.* 5(1), 55-59.
- Vahirua-Lechat, I., Mitermite, Y., Menut, C., 2010. Aromatic Plants of French Polynesia. IV. Composition and chemical variations of the essential oils of leaves of *Etlingera cevuga* (Seeman) R.E. Smith. *J. Essent. Oil Res.* 22(5), 407-409.
- Van-Deursen, M.M., Beens, J., Janssen, H.G., Leclercq, P.A., Cramers, C.A., 2000. Evaluation of time-of-flight mass spectrometric detection for fast gas chromatography. *J. Chromatogr. A* 878(2), 205-213.
- Wong, K.C., Sivasothy, Y., Boey, P.L., Osman, H., 2010. Essential Oils of *Etlingera elatior* (Jack) R. M. Smith and *Etlingera littoralis* (Koenig) Giseke. *J. Essent.Oil Res.* 22(5), 461-466.
- Wu, J., Lu, X., Tang, W., Kong, H., Zhou, S., Xu, G., 2004. Application of comprehensive two-dimensional gas chromatography-time-of-flight mass spectrometry in the analysis of volatile oil of traditional Chinese medicines. *J. Chromatogr. A* 1034(1-2), 199-205.
- Yahya, M.A.A., Yaacob, W.A., Din, L.B., Nazlina, I., 2010. Analysis of essential oils of *Etlingera sphaerocephala* var. *grandiflora* by two-dimensional gas chromatography with time-of-flight mass spectrometry. *Malay. J. Anal. Sci.* 14(1), 32-40.
- Zhu, S., Lu, X., Xing, J., Zhang, S., Kong, H., Xu, G., Wu, C., 2005. Comparison of comprehensive two-dimensional gas chromatography/time-of-flight mass spectrometry and gas chromatography-mass spectrometry for the analysis of tobacco essential oils. *Anal. Chim. Acta* 545(2), 224-231.