Asian Journal of Plant Sciences

ISSN 1682-3974

science alert

ANSInet
an open access publisher
http://ansinet.com

OPEN ACCESS Asian Journal of Plant Sciences

ISSN 1682-3974
DOI: 10.3923/ajps.2016.101.108

Research Article
Analysis of Aroma Profile of Agarwood Incense Smoke by SPME and GC-FID combined with GC-MS

[1]N. Ismail, [2]M. Ibrahim, [1]S. Zareen, [2]S.N. Tajuddin, [1]M.H.F. Rahiman and [1]M.N. Taib

[1]Faculty of Electrical Engineering, Universiti Teknologi MARA, 40450 Shah Alam, Selangor, Malaysia
[2]Faculty of Industrial Sciences and Technology, Universiti Malaysia Pahang, 26300 Gambang, Pahang, Malaysia

Abstract
Analysis of the aroma profile of agarwood incense smoke by varying the sampling time is presented in this study. The compound extraction was performed with the implementation of using Gas Chromatography-Flame Ionization Detector (GC-FID), Gas Chromatography-Mass Spectroscopy (GC-MS) and solid phase microextraction (SPME) on commercial, low and high quality agarwood. The extraction is based on agarwood smoke and headspace volatile (vapor) via SPME fibre type DVB-CAR-PDMS under three different sampling 15, 30 and 60 min. The result revealed that the agarwood smoke are made of three major groups, monoterpene hydrocarbon, sesquiterpene hydrocarbon and oxygenated sesquiterpene. The decreasing of chemical compounds composition in several compounds during the extraction showed that sampling time gave effect to the compounds composition. The finding is very significant and it is beneficial for further analysis especially for agarwood grading.

Key words: Agarwood, SPME, GC-FID, GC-MS, quality

Received: May 10, 2016 **Accepted:** August 13, 2016 **Published:** September 15, 2016

Citation: N. Ismail, M. Ibrahim, S. Zareen, S.N. Tajuddin, M.H.F. Rahiman and M.N. Taib, 2016. Analysis of aroma profile of agarwood incense smoke by SPME and GC-FID combined with GC-MS. Asian J. Plant Sci., 15: 101-108.

Corresponding Author: N. Ismail, Faculty of Electrical Engineering, Universiti Teknologi MARA, 40450 Shah Alam, Selangor, Malaysia

Competing Interest: The authors have declared that no competing interest exists.

Data Availability: All relevant data are within the paper and its supporting information files.

INTRODUCTION

Agarwood is the resin-impregnated heartwood of *Aquilaria* species and belongs to Thymelaeceace family. Agarwood is believed to have tonic and therapeutic properties[1,2]. Its oil has high demand in the perfume industry as evidenced by the recent expansion of the range of uses. They include new products such as agarwood incense, soap and shampoo.

Agarwood has been used as a high quality incense antiquity. Incense is the origin of Latin words, incendere which means 'to burn', consist aromatic material release the aroma when it burned. Incense smoke is usually used to mask odour and as an aromatherapy. The Chinese describe its smell as 'a sweet, deep but balances fragrance'. The people continue using it in their religious and festive celebrations, as do Arabians, Indian and Japanese. Traditionally, high quality incense agarwood is decided based on the basis of individual requirements as well as physical characteristics such as density, colour, solubility and odour. It is also needed an expert in the agarwood industry to identify its quality[3]. However, there is limited information on the quality of different agarwood oils or incense smoke and their research is ongoing[2,4].

There are two types of smoke incense, Eastern incense is processed from natural plants such as sandalwood, patchouli and agarwood whereas Western incense which produced from resin in tree peel, flowers and essential oils[5]. The incense burned to produce pleasant fragrance in the home, in public areas including shopping malls and worship venue[5]. Nowadays, incense is available in various forms including joss stick, cones, coils and rocks and its usage is extensive.

Previously, some researchers have been studied the chemical composition of agarwood essential oil, determination of chromones and terpenes group of Cambodian agarwood oil (*Aquilaria agallocha*) by Alkhathlan *et al.*[6], determination of volatile compounds from *Aquilaria malaccensis*[7] and isolation of the sesquiterpenes group from *Aquilaria agallocha*[8]. However, characterization of agarwood incense or smoke is not widely studied. Therefore, as ongoing research in agarwood grading, this study is aim to analyze the complex mixture of volatile agarwood smoke using solid phase microextraction (SPME) and Gas Chromatography-Mass Spectrometry (GC-MS) combined with Gas Chromatography-Flame Ionization Detector (GC-FID). The study is carried out by varying the sampling time during the extraction.

Solid phase microextraction (SPME): The SPME technique is acceptable either in gaseous sample matrix, solid matrix or aqueous sample matrix analysis. It is also well-suited with analyte separation either by HPLC or gas chromatography and imparts linear results for broad concentrations of analytes. Pawliszyn[9] indicates that SPME method can be used in direct extraction which the coating fibre adsorbs analyte from matrix directly. Previous studies have shown that SPME technique is useful for any kind of sample matrix and way of extraction, extraction of the gaseous matrix reported by Tran and Marriott[5] using both ways of extraction onto incense smoke and other studies to determine the volatile aromatic compound from aqueous matrix by using the headspace extraction method[10-12].

Gas Chromatography-Flame Ionization Detector (GC-FID): The GC-FID is one of important analysis instruments especially in the natural product and perfumery industry and widely used to analyze essential oil, fatty acid and terpene group, monoterpene and sesquiterpene[13,14]. The sample was carried using a carrier gas such as helium (He) or nitrogen (N_2) into a vapor circumstances via a narrow fused silica column with advanced stationary phase films leap to the surface and cross linked to increase thermal stability. Figure 1 showed the schematic diagram of FID sensor. The diagram also is equipped with the column which is installed on the oven and is controlled by temperature which can be heated up to 450°C, with separation of a broad range of compounds.

Gas Chromatography-Mass Spectrometer (GC-MS): Gas Chromatography-Mass Spectroscopy (GC-MS) is a combination two techniques, gas chromatography is for mixture compounds separation and mass spectroscopy for an individual compound characterization. With these combined techniques, GC-MS able to evaluate qualitative and quantitative the mixture compounds[15-17]. Generally, GC-MS is used for identification and quantitation of mixture compounds (volatile and semivolatile) as well as for unknown compounds structure determination with matching spectra with reference spectra and preceding spectra understanding[18]. In standard GC-MS practice, the ions is needed for mass analysis. The ions or charged particles developed with electron impact ionization (EI). A high energy electron beam (70 eV) is bombard to the gas molecule from the GC. An electron, who hit a molecule, put the energy to eliminate another electron to produce a positive charge ion. The EI ionization typically generates individually charged ions

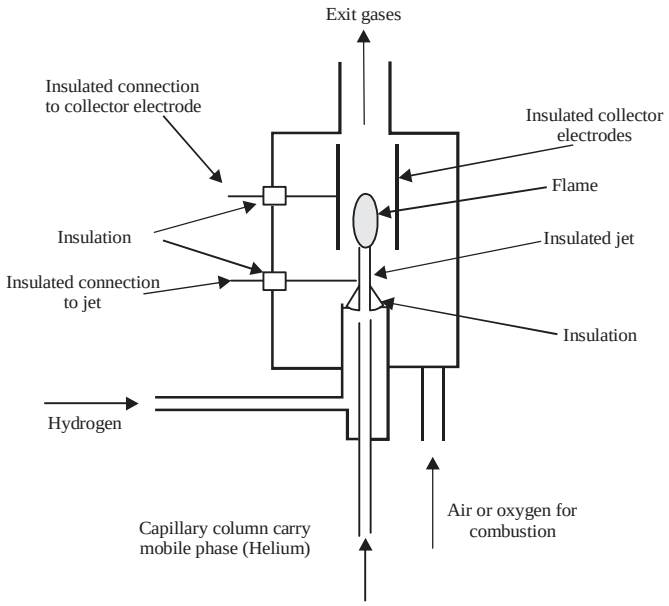

Fig. 1: Schematic diagram of FID sensor

with a single electron. There are some types of ionization of chemical analysis, yet the EI mode is frequently used in laboratories research and industries because it can use in various applications, gas saving and straightforward.

MATERIALS AND METHODS

Sample preparation: Three different types of agarwood (*A. malaccensis*) chipwood, low quality, high quality and commercial were used in this study. Low and high quality of agarwood chipwood were purchased from Gua Musang (Kelantan), Malaysia and for commercial, the agarwood was purchased from local suppliers, Konsesi Utama Sdn Bhd, Malaysia. All samples were in dry condition while purchasing. A 100 g of dry samples (low and high quality) is ground into milled agarwood and dried again (40°C) in oven until the weight is constant and stored prior to extraction. The rest of chipwood samples were stored for burning purpose.

Chemical compounds extraction-solid phase microextraction (SPME): The extraction of agarwood chipwood was done using the solid phase microextraction (SPME). The SPME equipment was purchased from Supelco Inc., Bellefonte, PA, USA. A 50/30 µm divinylbenzene-carboxen-polydimethysiloxane (DVB-CAR-PDMS). The extraction mode of smoke was adapted from previous study[5]. Figure 2 shows The SPME fibre was put directly on the top of the inverted glass funnel. The sample (joss stick form) was burned under the funnel and allow the smoke stream allow the passage. This allows absorption of smoke stream into SPME fibre. The sampling was taking at 15, 30 and 60 min.

Method of analysis: There are several instruments used to analyze a sample such as gas chromatography, HPLC and another few instruments. In this study, a gas chromatography is choosing to analyze the volatile compound of incense, the gas chromatography coupled with flame ionization detector (GC-FID) and mass spectrometer detector (GC-MS).

Gas Chromatography-Flame Ionization Detector (GC-FID) and Gas Chromatography-Mass Spectrometer (GC-MS): The incense sample was analyzed by using an Agilent 7890 Gas Chromatography-Flame Ionization Detector (GC-FID) equipped with a 30 m long fused silica DB-1 capillary column (0.25 mm of internal diameter and 0.25 µm of film thickness). Helium was used as a carrier gas with a flow rate of 1.2 mL min^{-1}. The manual SPME injector was set in splitless mode using a narrow SPME inlet liner. The oven temperature was programmed at 60°C initially and increased at a rate of 3°C min^{-1} to a final temperature of 250°C and maintain for 5 min. Electron impact ionization (EI) mass spectra were collected at 70 eV ionization voltages at range 20-500 µ. The compounds were identified based on comparison between

their Retention Indices (RI) and published data. Retention indices or kovalts index were calculated using a linear hydrocarbon (C_8-C_{22}). Peak identification of volatile components was based on NIST library.

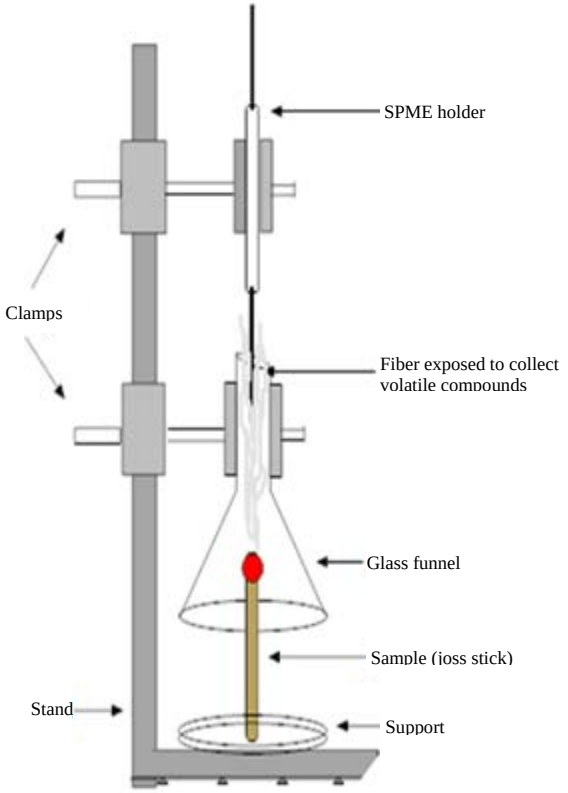

Fig. 2: Schematic diagram of direct extraction of smoke Source: Trans and Marriott[5]

RESULTS AND DISCUSSION

The characteristic of agarwood was observed based on aroma released during the burning process. High quality agarwood showed the best contribution in fragrance of smoke, strong and sweet aromas released while burning. Meanwhile, low quality agarwood produced fewer odours and commercial presented a strong and acrid aroma. Table 1 listed volatile compounds for commercial, low and high quality agarwood. It is observed that there are notable differences in the chemical components among the three sample. Generally, it was found that agarwood smoke was made up of three major groups i.e., monoterpene hydrocarbon, sesquiterpene hydrocarbon and oxygenated sesquiterpene. Compounds in oxygenated sesquiterpene was identified to contribute the most. The compound observation among oxygenated sesquiterpenes showed that epoxybulnesene produced the highest percentage area in high quality agarwood with 15.72, 14.81 and 12.96% for 15, 30 and 60 min, respectively. The α-elemol gave the highest percentage area in commercial incense with the percentage of 4.98, 3.00 and 1.12% for 15, 30 and 60 min, respectively. For low quality, the compounds with the highest percentage area are different to each sampling time (15, 30 and 60 min). The highest percentage area at 15 min sampling time is α-elemol (5.12%), at 30 min is dehydrojinkoh-eremol (4.33%) and at 60 min is epi-α-bisabolol (2.93%). It gives a remark that the compounds of agarwood is relate to the quality of agarwood and they are dependent to each other.

Table 1: Chemical composition (percentage relative peak area) of agarwood smoke

		Time extraction									
		15 min			30 min			60 min			
Compound	KI	Commercial	Low quality	High quality	Commercial	Low quality	High quality	Commercial	Low quality	High quality	Identification
Monoterpene hydrocarbon											
Furfuryl alcohol	830	0.02	0.15	4 -	0.23	-	-	0.82	0.13	0.07	6 RI
Benzaldehyde	935	0.17	0.29	0.85	0.14	-	-	0.75	0.27	0.89	RI, 7 MS
Phenol	956	-	-	0.16	0.02	-	-	-	0.03	0.04	RI
p-methylanisol	1002	0.21	0.63	1.92	0.07	-	-	0.85	0.38	0.12	RI
2-hydroxy-benzaldehyde	1003	-	0.13	0.4	0.03	-	-	0.33	0.17	0.19	RI, MS
Guaiacol	1062	-	-	-	1.46	0.32	0.33	-	-	0.91	RI
Acetophenone	1066	1.8	3.52	3.09	0.68	-	-	6.29	3.55	1.22	RI, MS
p-methoxyphenol	1198	-	-	0.19	-	-	-	1.02	-	-	RI
p-vinylphenol	1199	-	1.25	1.11	0.31	1.51	-	-	-	1.13	RI
4-phenyl-2-butanone	1210	2.3	7.86	7.24	0.14	1.16	2.03	1.71	5.12	6.58	RI, MS
p-vinylguaiacol	1286	6.39	6.82	2	1.03	1.29	0.56	6.77	1.88	1.58	RI
3,4-dimethoxyphenol	1312	8.48	8.65	4.82	2.65	3.74	1.82	5.82	0.61	1.09	RI
Vanilin	1367	2.13	0.99	0.46	1.3	0.33	0.21	0.1	2.49	0.34	RI

Table 1: Continue

Compound	KI	Time extraction: 15 min			30 min			60 min			Identification
		Commercial	Low quality	High quality	Commercial	Low quality	High quality	Commercial	Low quality	High quality	
Sesquiterpenes hydrocarbon											
β-maaliene	1414	3.89	3.02	0.21	1.6	1.98	1.4	3.19	0.51	1.63	RI, MS
α-guaiene	1440	0.34	0.9	0.43	0.14	0.32	0.26	0.69	0.33	0.24	RI, MS
Aromadendrane	1443	0.52	0.25	0.26	0.21	0.26	0.19	0.17	0.61	0.47	RI, MS
γ-gurjunene	1472	1.08	0.3	0.75	0.05	0.18	0.9	0.85	0.25	0.8	RI
β-agarofuran	1474	0.26	1.42	1.09	0.05	0.41	1.22	1.14	0.19	1.55	RI
β-selinene	1486	0.94	0.5	0.45	0.41	0.56	0.76	0.83	0.73	2.22	RI, MS
α-muurolene	1496	2.53	2.09	1.59	1.05	-	2.03	0.44	0.61	1.93	RI
γ-guaiene	1499	0.57	0.17	0.11	0.47	3.93	0.34	0.48	1.69	0.26	RI
α-bulnesene	1530	0.94	0.24	0.22	0.24	1.2	0.68	0.76	1.32	0.27	RI
Oxygenated sesquiterpenes											
α-elemol	1503	4.98	5.12	0.89	3	0.44	0.41	1.12	0.19	0.59	RI
Nor-ketoagarofuran	1555	3.69	0.19	0.66	2.41	1.28	2.85	3.51	0.87	0.51	RI, MS
Tridecanol	1561	-	-	-	1.5	0.28	-	0.29	0.3	-	RI
Epoxybulnesene	1572	0.61	1.15	15.72	1.32	1.97	14.81	0.53	1.55	12.96	RI
Caryophellene oxide	1600	0.72	0.52	3.02	0.5	1.08	0.96	-	0.58	2.1	RI
Guaiol	1603	-	0.49	-	-	0.52	0.91	0.2	-	-	RI
Humulene epoxide II	1606	0.73	0.73	0.19	0.48	0.62	1.55	0.34	0.49	1.35	RI
1,5-epoxy-nor-ketoguaiene	1614	0.83	1.14	0.51	1.04	-	-	0.04	0.29	3.91	RI
10-epi-γ-eudesmol	1619	0.16	0.69	0.76	1.7	3.43	1.82	0.38	2.09	0.39	RI
Agarospirol	1631	0.74	0.71	2.06	2.08	1.01	2.64	0.73	0.67	5.07	RI, MS
Epi-α-cadinol	1640	0.1	0.11	0.36	-	0.57	1.95	-	-	-	RI
Jinkoh-eremol	1643	0.21	0.24	0.25	0.69	1.25	1.02	-	0.78	1.47	RI
Kusunol	1650	2.52	0.2	0.33	-	-	-	1.69	0.33	0.61	RI
α-eudesmol	1652	0.27	0.17	0.34	1.78	1.01	1.39	0.31	0.29	0.59	RI
Bulnesol	1664	2.2	4.42	0.73	0.51	1.28	0.56	-	0.66	0.69	RI
Dehydrojinkoh-eremol	1673	0.37	-	0.37	-	4.33	2.52	-	1.68	0.54	RI
Epi-α-bisabolol	1678	0.42	-	0.3	5.96	3.17	1.96	-	2.93	-	RI
α-bisabolol	1683	0.36	1.07	-	0.37	-	0.9	-	0.21	0.23	RI
Selina-3,11-dien-9-one	1687	0.26	0.2	0.09	0.36	1.66	1.17	-	0.5	0.15	RI
Pentadecanal	1695	0.39	1.93	0.11	0.78	0.9	0.44	-	0.42	0.16	RI
Rotundone	1703	0.24	-	0.17	1.59	3.35	1.43	0.17	1.32	0.2	RI
Selina-3,11-dien-9-ol	1721	0.09	0.24	0.55	0.45	1.58	0.49	0.11	0.63	0.62	RI
Selina-4,11-dien-14-oic acid	1728	0.36	0.15	0.45	1.58	3.35	0.42	0.16	0.73	0.19	RI
Selina-3,11-dien-14-al	1735	0.66	0.12	-	1.56	0.99	1.19	0.71	1.6	0.34	RI
9,11-eremophiladien-8- one	1740	0.32	0.09	-	1.24	0.76	0.78	-	0.9	0.04	RI
Selina-3,11-dien-14-ol	1750	-	-	-	0.58	0.66	1.53	-	0.72	0.02	RI
Guaia-1(10),11-dien-9-one	1752	-	0.09	-	0.72	1.58	-	-	0.74	0.07	RI
Selina-4,11-dien-14-al	1758	-	-	0.07	-	-	0.81	-	0.25	0.05	RI
Guaia-1(10),11-dien-15-ol	1770	-	0.06	0.19	4	1.51	0.23	-	0.58	0.16	RI
Selina-3,11-dien-14-oic acid	1775	0.07	0.1	0.12	1.19	1.34	0.14	-	1.01	0.26	RI
Sinenofuranol	1776	-	0.09	-	-	0.76	0.74	-	0.42	0.08	RI
Dihydrokaranone	1799	0.27	-	-	0.75	0.87	0.34	-	0.6	-	RI
Guaia-1(10),11-dien-15-al	1806	-	-	-	0.76	0.75	0.38	-	0.52	-	RI
Karanone	1812	0.52	-	-	0.69	-	0.14	0.42	-	-	RI
Oxo-agarospirol	1822	0.05	-	-	1.79	0.86	0.08	-	0.56	-	RI
Pentadecanoic acid	1842	-	-	-	0.22	0.51	0.38	-	0.58	-	RI
Hexadecanol	1865	-	0.13	-	0.26	1.03	0.12	-	1.5	0.17	RI
Eudesmol	1880	0.08	0.69	-	0.34	0.63	0.68	-	-	0.05	RI
Palmitic acid	1912	-	-	-	0.7	1.42	0.1	-	1.42	0.02	RI
2-hydroxyguaia-1(10),11-dien-oic acid	1932	-	-	-	0.36	0.6	0.07	-	0.39	0.05	RI
9-hydroxyselina-4,11-dien-14-oic acid	1948	1	-	-	-	0.43	0.24	-	-	0.02	RI
n-hexadecanoic acid	1950	-	-	-	0.86	0.21	0.04	-	0.96	-	RI, MS
1,5-diphenyl-3-pentanone	1962	-	-	-	-	0.29	0.3	-	0.27	-	RI
Guaia-1(10),11-dien-15,2-olide	2019	-	-	0.13	0.26	-	-	-	0.06	0.17	RI

KI: Kovats retention indices on DB-1 column, -: Not identified, RI: Linear retention indices relative to the retention time on DB-1 column of a homologous series of n-alkanes (C_8-C_{22}), MS: Identification by comparison of MS with those of the NIST library

The other point of view is the result also revealed that contribution from several compounds decreased when the extraction time increased. Specifically for high quality, compound epoxybulnesene gave a high percentage area (15.72%) for the initial 15 min extraction which then reduced to 14.81% at the 30 min mark and continued to reduce to 12.96% at 60 min. Besides that, other compounds also showed decreasing in term of composition. They are 3,4-dimethoxyphenol, α-elemol, bulnesol, caryophellene oxide and humulene epoxide II. For 3,4-dimethoxyphenol, the compound's composition decreased from 8.65% at 15 min, to 3.74% at 30 min and 0.61% at 60 min. It was followed by α-elemol, where the composition is 5.12% at 15 min, 0.44% at 30 min and 0.19% at 60 min. After that is bulnesol where the composition is 4.42% at 15 min, 1.28% at 30 min and 0.66% at 60 min. Not limited to that, caryophellene oxide and humulene epoxide II also show the similar trend. The composition for caryophellene oxide (in high quality) decreased from 3.02% at 30 min to 2.10% at 60 min and the composition for humulene epoxide II, the composition is decreasing from 0.73% at 15 min to 0.48 and 0.34% at 30 and 60 min, respectively for commercial wood, followed by 0.73% at 15 min decreases to 0.62 and 0.49% at 30 and 60 min, respectively for low quality wood. These results showed that those compounds composition are lost at long sampling time. The scenari marks that the sampling time during extraction gave effect to the percentage area of chemical compounds.

Figure 3 shows graphical observation of chemical compositions in Table 1. Specifically it is on the chemical composition on three types of agarwood; commercial, low and high quality agarwood using 15, 30 and 60 min sampling time. Generally, it can be seen that, agarwood smoke is a

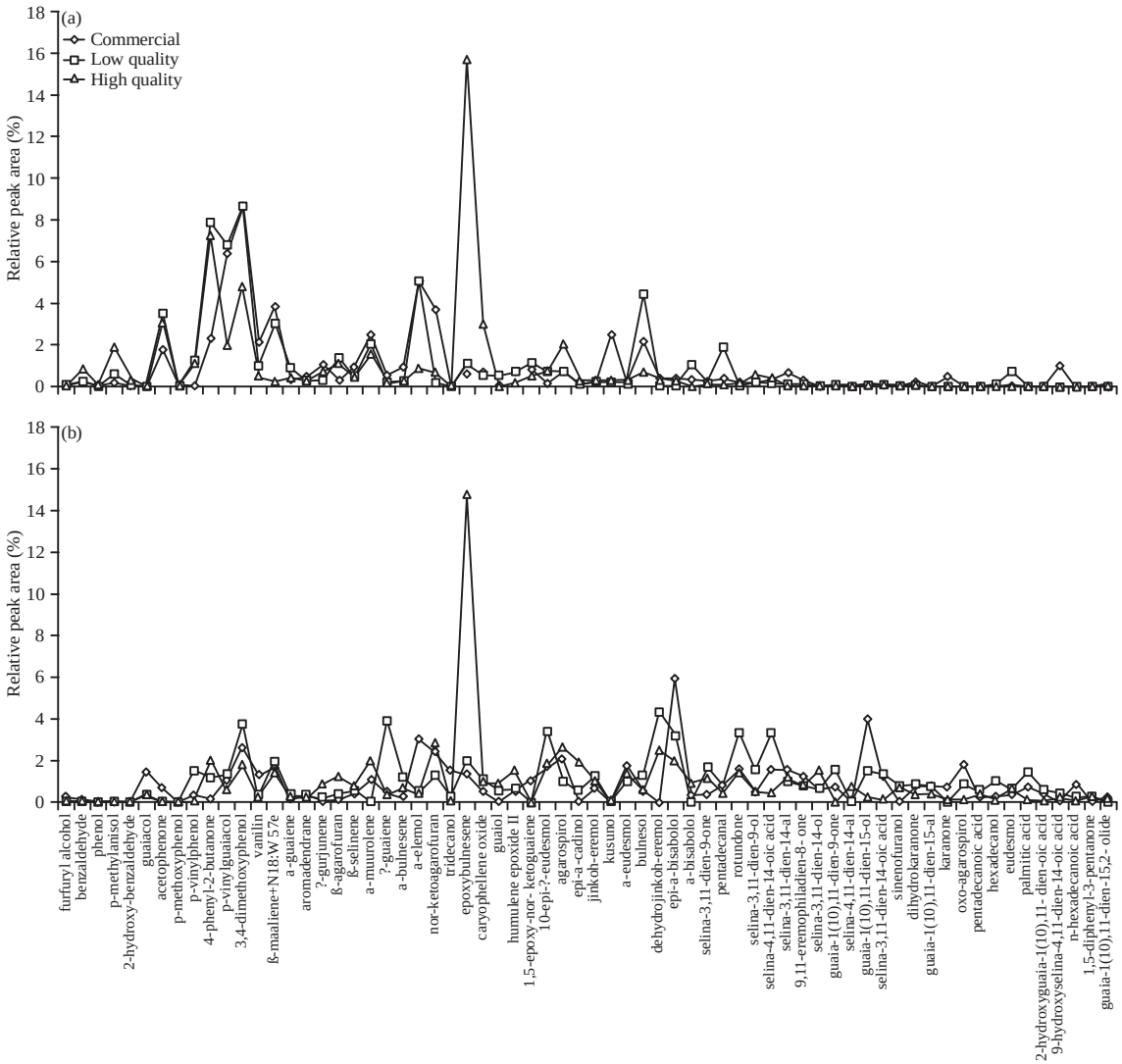

Fig. 3(a-c): Continue

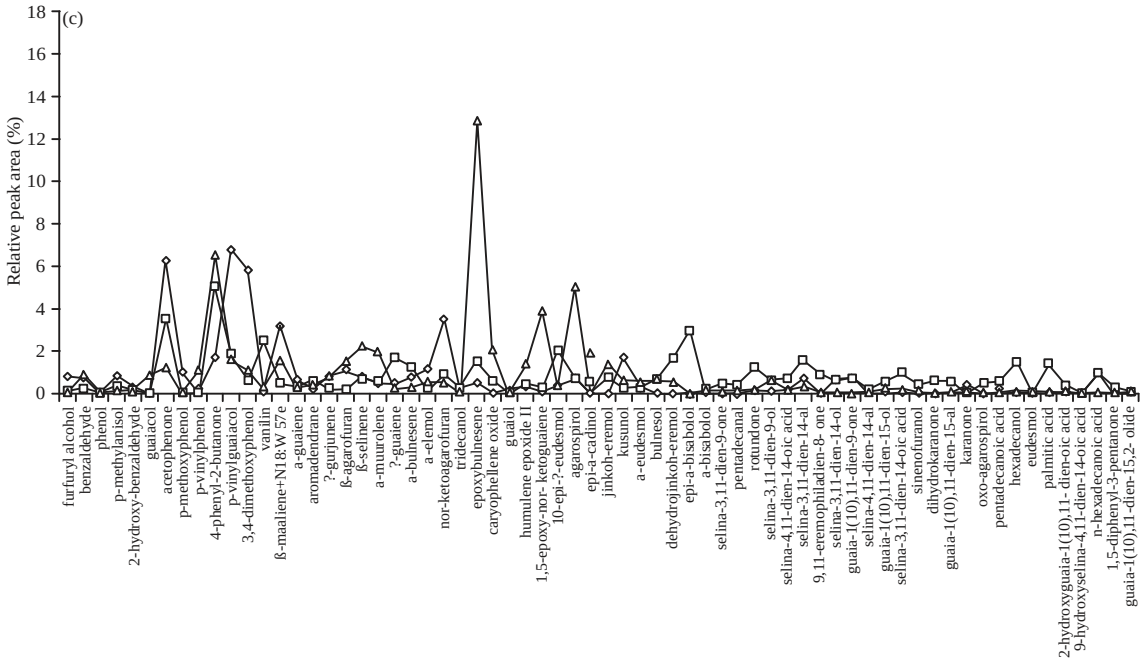

Fig. 3(a-c): Chemical composition on commercial, low and high quality agarwood using sampling time, (a) 15 min, (b) 30 min and (c) 60 min

mixture and variation of monoterpene hydrocarbon, sesquiterpene hydrocarbon and oxygenated sesquiterpene. Among all, it was found that epoxybulnesene afforded the highest relative peak area (%), in which it is applicable for all sampling time (15.72% at 15 min, 14.81% at 30 min and 12.96% at 60 min). In Fig. 3a and c, there is similar trend occurred provided by commercial, low and high quality agarwood smoke. It was that from rotundone (at 15 min) and from α-bisabolol (at 60 min) to guaia-1(10), 11-dien-15, 2-olide, the overall the peak area (%) after these compounds are small, i.e., from rotundone less than 1.00 at 15 min and from α-bisabolol less than 2.00 at 60 min. However, for 30 min sampling time, it is a consistent plot of all compounds. Their relative peak area (%) is varied to each other and within 4.00, except for the epoxybulnesene.

CONCLUSION

Analysis of aroma profile of agarwood incense smoke by SPME and GC-FID combined with GC-MS has been presented in this study. It was found that the agarwood smoke is made up of three major groups, monoterpene hydrocarbon, sesquiterpene hydrocarbon and oxygenated sesquiterpene. Variation of three different sampling time during extraction, 15, 30 and 60 min showed that chemical compounds decreases as the sampling time of extraction increases. This finding is important and significant, thus is beneficial for further analysis especially in agarwood grading.

ACKNOWLEDGMENTS

The data used in this study is obtained from Faculty of Industry Sciences and Technology, University Malaysia Pahang, Malaysia. The authors would like to thank all staff involved, Faculty of Electrical Engineering and e-Science Fund (02-01-06-SF0092) for financial supports.

REFERENCES

1. Barden, A., N.A. Anak, T. Mulliken and M. Song, 2000. Heart of the matter: Agarwood use and trade and CITES implementation for Aquilaria malaccensis. A TRAFFIC Network Report, TRAFFIC International, Cambridge, UK., pp: 1-52.
2. Chang, I.T., L.S. Ng and A.A. Kadir, 1997. A review on agur (gaharu) producing *Aquilaria* species. J. Trop. For. Prod., 2: 272-285.
3. Abdullah, A., N.K.N. Ismail, T.A.A. Kadir, J.M. Zain, N.A. Jusoh and N.M. Ali, 2007. Agar wood grade determination system using image processing. Proceedings of the International Conference on Electrical Engineering and Informatics, June 17-19, 2007, Indonesia, pp: 427-429.

4. Chen, H., Y. Yang, J. Xue, J. Wei, Z. Zhang and H. Chen, 2011. Comparison of compositions and antimicrobial activities of essential oils from chemically stimulated agarwood, wild agarwood and healthy *Aquilaria sinensis* (Lour.) Gilg trees. Molecules, 16: 4884-4896.
5. Tran, T.C. and P.J. Marriott, 2007. Characterization of incense smoke by solid phase microextraction-Comprehensive two-dimensional gas chromatography (GC×GC). Atmos. Environ., 41: 5756-5768.
6. Alkhathlan, H.Z., H.M. Al-Hazimi, F.S. Al-Dhalaan and A.A. Mousa, 2005. Three 2-(2-phenylethyl) chromones and two terpenes from agarwood. Nat. Prod. Res., 19: 367-372.
7. Tajuddin, S.N. and M.M. Yusoff, 2010. Chemical composition of volatile oils of *Aquilaria malaccensis* (Thymelaeaceae) from Malaysia. Nat. Prod. Commun., 5: 1965-1968.
8. Maheshwari, M.L., T.C. Jain, R.B. Bates and S.C. Bhattacharyya, 1963. Terpenoids-XLI: Structure and absolute configuration of α-agarofuran, ⨿-agarofuran and dihydroagarofuran. Tetrahedron, 19: 1079-1090.
9. Pawliszyn, J., 2012. Handbook of Solid Phase Microextraction. Elsevier, New York, USA., ISBN-13: 9780124160170, Pages: 496.
10. Richter, J. and I. Schellenberg, 2007. Comparison of different extraction methods for the determination of essential oils and related compounds from aromatic plants and optimization of solid-phase microextraction/gas chromatography. Anal. Bioanal. Chem., 387: 2207-2217.
11. Pripdeevech, P., W. Khummueng and S.K. Park, 2011. Identification of odor-active components of agarwood essential oils from Thailand by solid phase microextraction-GC/MS and GC-O. J. Essent. Oil Res., 23: 46-53.
12. Hamm, S., J. Bleton, J. Connan and A. Tchapla, 2005. A chemical investigation by headspace SPME and GC-MS of volatile and semi-volatile terpenes in various olibanum samples. Phytochemistry, 66: 1499-1514.
13. Bartle, K.D. and P. Myers, 2002. History of gas chromatography. Trends Anal. Chem., 21: 547-557.
14. Van Asten, A., 2002. The importance of GC and GC-MS in perfume analysis. Trends Anal. Chem., 21: 698-708.
15. Deng, C., N. Liu, M. Gao and X. Zhang, 2007. Recent developments in sample preparation techniques for chromatography analysis of traditional Chinese medicines. J. Chromatogr. A, 1153: 90-96.
16. Eyres, G.T., P.J. Marriott and J.P. Dufour, 2007. Comparison of odor-active compounds in the spicy fraction of hop (*Humulus lupulus* L.) essential oil from four different varieties. J. Agric. Food Chem., 55: 6252-6261.
17. Augusto, F., A.L. Lopes and C.A. Zini, 2003. Sampling and sample preparation for analysis of aromas and fragrances. Trends Anal. Chem., 22: 160-169.
18. Glassmeyer, S.T., K.E. Shanks and R.A. Hites, 1999. Automated toxaphene quantitation by GC/MS. Anal. Chem., 71: 1448-1453.