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Qualitative Analysis of Aliphatic Organic Compounds in Atmospheric Particulates and their Possible Sources using Gas Chromatography Mass Spectrometry

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Abstract

Air quality monitoring was carried out to determine the concentration of PM₁₀ and its organic composition at Universiti Kebangsaan Malaysia (UKM), Bangi City, and Simpang Empat (SE), Penang City. A High Volume Air Sampler (HVAS) equipped with glass fiber filter paper with a flow rate of 1.13 m³ per minute for 24 hours has been used to collect PM₁₀ samples. The result shown the concentration of PM₁₀ ranged between 44.8 µg/m³ to 59.9 µg/m³ witch is still under the permitted level as suggested by the Department of Malaysian Environment (DME), 150 µg/m³ for 24 hours. A large number of volatile and semi-volatile organic compounds from atmospheric particulates PM₁₀ and soot samples were identified. The particulate organic matter was extracted by using ultrasonic agitation with organic solvents and characterized by gas chromatography and mass spectrometry (GC–MS). Most of the organic compounds identified from PM₁₀ and their possible sources from soot samples consist of aliphatic compounds (alkanes, ketones, aldehydes, alcohols, and fatty acids with both biogenic and anthropogenic origin. The molecular weight of the identified organic compounds including unresolved complex mixture (UCM) ranged between 98.14 g/mol and 408 g/mol. This study indicated that utilization of fossil fuels and biomass burning are the major contributors to the identified organic compounds in the semi-urban atmospheric area.

Keywords: PM₁₀; biomass burning; organic compounds; GC MS

1. Introduction

Atmospheric particulate matter is known to contain a number of organic species, including alkanes, alkenes, carboxylic acids, carbonyl compounds, and aromatic compounds. Organic aerosol pollution has always been an interest of scientists due to of the potential



detrimental effects on human health (e.g., asthma, emphysema) with associated toxic organic compounds (Abelson, 1998; Oanh et al., 2002).

Organic matter in aerosols is composed of a large number of individual compounds and is widespread in the environment. Typical sources include natural and anthropogenic emissions such as forest fires, volcanic eruptions, incomplete combustion of fossil fuels, wood, agricultural waste or leaves, and fugitive emissions from industrial processes (Simoneit et al., 1991a, b).

The organic compositions of urban aerosols have been studied most (Aceves and Grimalt, 1993; Kavouras et al., 1999, 2001; Cortes et al., 2000; Larsen and Baker, 2003; Ohura et al., 2004). However, only a few surveys were conducted in Malaysia with comprehensive data on organic aerosols (Tang, Kachi and Furukawa 1996; Rashid, Lim and Rahmalan 1997). For this reason, it is necessary to carry out more studies with a larger number of samples and for analysis of additional organic compounds in order to obtain more accurate information on the pollution sources.

Hundreds of individual organic compounds have been identified in the organic atmospheric aerosol so far (Hildemann, 1996), however, until the 90ies the analytically accessible components constituted less than 10% of the organic carbon (OC) of urban and rural (Rogge et al., 1993; Puxbaum et al., 2000). The main analytical method used so far to separate and identify individual organic species was Gas Chromatography coupled to Mass spectrometry (GC/MS), with or without derivatization of acid groups into methyl esters derivatives.

In this case, we present the results of the chemical analysis of the organic compounds from an urban ambient particulate matter with an aerodynamic diameter $<10 \mu\text{m}$ (PM₁₀). We have also described the chemical analysis of the organic compounds of different types of soot resulting from the burning of various types of wood as a possible source of organic compounds in PM₁₀ particles.

2. Experimental

2.1 Sampling sites

Atmospheric particulate (PM₁₀) samples were collected from Universiti Kebangsaan Malaysia (UKM) Bangi and Simpang Empat (SE) Penang city. Fig.1. is a map



showing the location of the sampling site. The major emission sources of ambient particles of the field sampling are traffic, forest emission, regional or long-range transported aerosols. In addition traffic derived abrasion particles and resuspended road dusts as well as biological material are suggested to have major contributions to the coarse particles (Aurelie and Roy, 2005)

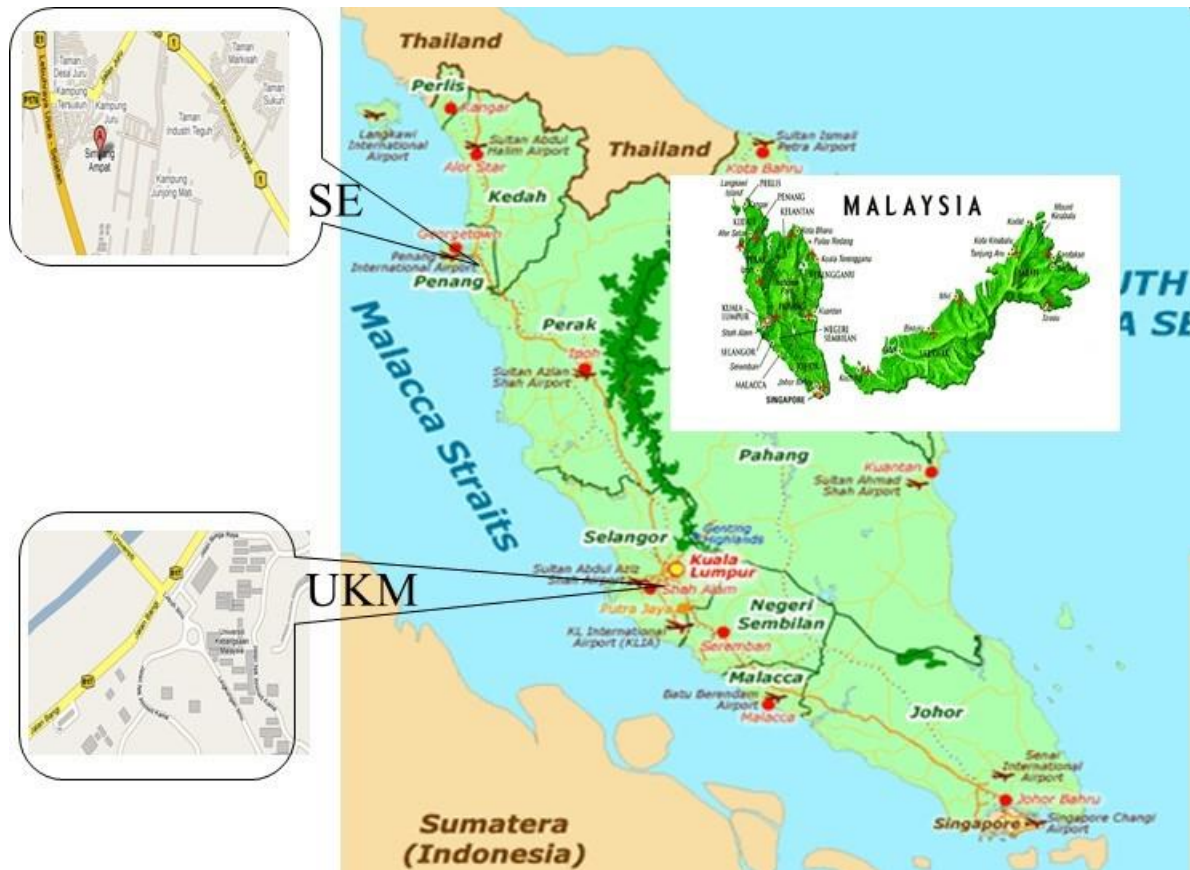


Figure 1 Map of Peninsular Malaysia showing the sampling locations

2.2 Procedure of PM₁₀ sampling

2.2.1 Collection of PM₁₀ Samples

The atmospheric particulate (PM₁₀) samples with a diameter less than 10 μ m (PM₁₀) were collected by using high volume air sampler (HVAS) model Staplex witch provided with a glass-fiber filter paper (GFFs) (20.3cm x 25.4cm) (8inch x 10inch) at flow rate at 1.13 m³/min for a total period of 24 hours (temperature 25-33 °C and humidity 58-93%).



Prior to sampling, the GFFs were wrapped with aluminium foil and combusted at 550 °C for three hours to remove organic background contaminants. The GFFs were weighed under controlled temperature and humidity conditions (25 °C, 50% RH) before and after the sampling in order to determine the concentrations of particulate matter (PM₁₀). The exposed filters were removed from the sampler after 24 hours and folded into half, face to face, and again placed in a plastic bag and wrapped in aluminium foil. After PM₁₀ collection, all filters were conditioned and re-weighed. The difference in weight before and after sampling and the total volume of air sample were used to calculate the PM₁₀ concentration (µgm⁻³) as shown in equation 1. All filters were stored in a freezer (-5 °C) before analysis (Radzi et al., 2004).

$$\text{Concentration of PM}_{10} = W/F_r \cdot t \quad (1)$$

Where:-

W = difference in the weight of the filter paper before and after sampling (µg)

Fr = flow rate (1.13 m³/minute)

t = sampling period 24x60(minutes)

Volume of air sampled=1.13m³/min x (24 x 60) min

2.2.2 Samples analysis

Filters stored in the freezer were held for 30 min at room temperature prior to analysis. The sample treatment and separation procedures follow the method first used by (Simoneit and Mazurek 1982). Each filter was extracted using ultrasonic agitation for three times, each for 20 min periods using 100 ml of dichloromethane/methanol (3:1, v:v) the solvent extract was filtered using a Gelman Swinney filtration Unit containing an annealed glass fiber filter for the removal of insoluble particles (Simoneit and Mazurek 1982). The filtrate was first concentrated by using a rotary evaporator and then by a stream of filtered nitrogen gas. The final volume was adjusted to exactly 4.0 ml by addition of DCM. The total extracts were then analyzed by capillary (GC-MS).

2.3 Procedure of soot sampling and extraction

Soot samples were prepared by cut of various types of wood (pine, oak, rubber, palm oil) to small pieces, drying, placed in iron dish, wrapped in aluminum foils, burned completely up to



300°C in the oven for 3 hours and then sieved (0.6mm) to remove coarse particles. It was noted that these soot samples it's kind of the aerosol PM₁₀ particle size but range to larger diameter.

Soot samples were weighed 5 g, placed in a 3 mm Pyrex glass thimble and inserted in a "Soxhlet" apparatus. Extraction of OCs was done for 6 hours using 250 ml dichloromethane and *n*-Propanol (3:1, v:v) as the extracting solvents. The condenser was passed with ice cold water to ensure that all the OCs in the DCM and Propanol will not evaporate since the solution has a boiling temperature of 70 °C. The OCs with DCM+Propanol solution was filtered using a filtration unit with an annealed glass fiber filter for the removal of insoluble particles (Simoneit and Mazurek, 1982). The mixture was filtered over 0.5 g anhydrous sodium sulfate (Na₂SO₄) as a dehydrating agent (to adsorb water and moisture). Filtrates were concentrated by using rotary evaporated until 5 ml of mixture and then clean up by a stream of filtered nitrogen gas. Aliquots are then taken for direct gas chromatography–mass spectrometry (GC– MS).

2.4 Gas chromatography-mass spectrometry

Gas chromatography mass spectrometry (GC-MS) analyses of the total extracts were performed on a Hewlett-Packard model 6890 GC coupled to a Hewlett-Packard model 5973 MSD. Separation was achieved on a fused silica capillary column coated with DB-5 (30m x 0.25 mm I.D., 0.25 µm film thickness). The GC operating conditions were as follows: temperature hold at 50°C for 2 min, increase from 50 to 300°C at a rate of 6°C/ min with final isothermal hold at 300°C for 20 min. Helium was used as carrier gas. The sample was injected splitless with the injector temperature at 300°C. The mass spectrometer was operated in the electron impact mode (EI) at 70 eV and scanned from 50 to 650 Dalton. Data were acquired and processed with the Chemstation software. Individual compounds were identified by comparison of mass spectra with literature and library data, comparison with authentic standards, and interpretation of mass spectrometric fragmentation patterns.

3. Results and discussion

Ground stations samples for this study, PM₁₀ concentrations, locations, and Air Quality Index (AQI) are summarized in Table 1. And the some of different kinds of soot were extracting as the main possible sources of OCs in PM₁₀. The characterizations of the organic compounds in the samples considered here are listed and categorized as classes in Table (2 and 3). Typical



examples of organic compositions of aerosol particles are discussed and shown in the following text.

The distributions and abundances of the biomass smoke constituents are strongly dependent on combustion temperatures smoldering versus flaming conditions and duration. Thus, the information reported here should not be used as absolute but as relative chemical fingerprints for these sources. The molecular biomarkers are source specific and may be used as confirming tracers for transport and fate studies of smoke emissions from these examples of biomass fuel sources.

Table 1: Date of exposition, locations, PM₁₀ concentration and Air Quality Index (AQI)

Location	PM ₁₀ Concentration (µg/m ³)	AQI
UKM	45.9	Good
-	59.9	Moderate
-	46.3	Good
-	47.5	Good
-	52.4	Moderate
-	48.7	Good
SE	55.3	Moderate
-	49.6	Good
-	46.3	Good
-		

Fig. 2 (a,b) shows an example of GC/MS total ion chromatogram and the mass spectrum of selected OCs obtained, illustrating the presence of all of selected organic compounds in the polar and non-polar fraction of atmospheric particulate matter from the UKM site and sample of soot extraction (pine). The total extracts show the distributions and relative abundances of all the major organic constituents present in each PM₁₀ and soot samples. In order to identify a source specific chemical fingerprint for soot emissions from the four vegetation types, the following discussion will focus on the identity and predominant distributions of aliphatic homologous series and biomarkers. Where possible, comparisons will be made to distinguish



the differences in “chemical fingerprints” between PM₁₀ and soot samples from (pine, oak, rubber, palm oil).

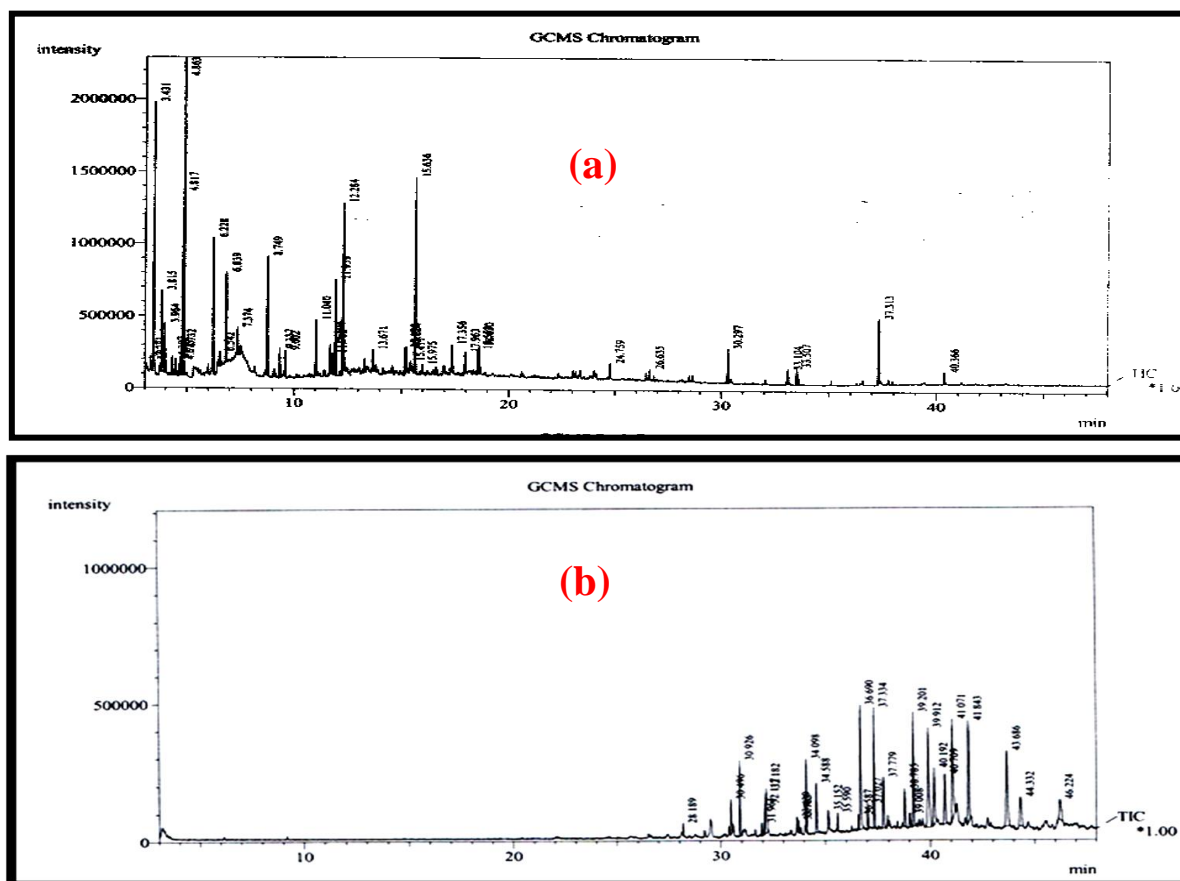


Fig. 2.(a) Total ion current traces of typical GC–MS analyses of PM₁₀ aerosol particle extracts at UKM station and (b) total ion current traces of typical GC–MS analyses of pine soot extracts, numbers over dots on peaks are indicate the retention time (Rt) of alkanes, alkenes, alkanols and alkanolic acids

3.1 Identification of organic compounds

The organic component of ambient particles in both polluted and remote areas is a complex mixture of hundreds of organic compounds (Hahn, 1980; Cass et al., 1982; Rogge et al., 1993abc). Compounds identified in the ambient aerosol include n-alkanes, n-alkanoic acids, aliphatic and so forth (Mazurek et al., 1989; Hildemann et al., 1993). Most of these compounds have also been identified in the current study. Obviously, the majority of the organic compounds in PM₁₀ and soot samples still remain uncharacterized due to compound polarity or molecular weight that fall outside the range of the analytical technique used in this study. There



are three possible stages when losses of organic compounds from the ambient samples (Atmospheric transport, High-volume sampling and Storage before analysis) could have occurred (Jordan, 2005):

3.1.1 Aliphatic compounds

The identities analysis of individual aliphatic compounds present in the various polarity and non-polarity fractions separated from the extract of PM₁₀ and soot samples are summarized in Table (2 and 3).

Table 2: Qualitative analysis of Organic compounds in atmospheric particulate matter (PM₁₀) samples

location	Compound group	Rt (min)	Compound name	formula	M.Wt
SE	<i>n</i> -Alkanes				
		30.42	Icos-5-yne	C ₂₀ H ₃₈	278
		35.53	Eicosane, 2-methyl	C ₂₁ H ₄₄	296
		36.98	3,8-Dimethyl undecane	C ₁₃ H ₂₈	184
		39.27	tetratriacontane	C ₃₄ H ₇₀	478
		42.50	Trans-Squalene	C ₃₀ H ₅₀	410
UKM					
		35.22	Tetracosane	C ₂₄ H ₅₀	338
		38.64	2,6,10,15-tetramethyl heptadecane	C ₂₁ H ₄₄	296
		37.94	Hexacosan	C ₂₆ H ₅₄	366
		39.25	Heptacosane	C ₂₇ H ₅₆	380
		40.84	Octacosane	C ₂₈ H ₅₈	394
		42.75	Nonacosane	C ₂₉ H ₆₀	408
UKM	<i>n</i> -Alkanols				
		5.53	2-butoxyethanol	C ₆ H ₁₄ O ₂	118
		30.59	1-octadecanol	C ₁₈ H ₃₈ O	270
		3.87	2,3-dimethylbutan-2-ol	C ₆ H ₁₄ O	102
UKM	<i>n</i> -Alkanoic acids				
		24.38	tetradecanoic acid	C ₁₄ H ₂₈ O ₂	228
		28.61	hexadecanoic acid (palmitic acid)	C ₁₆ H ₃₂ O ₂	256
		30.68	9,12-octadecadienoic acid	C ₁₈ H ₃₂ O ₂	280
		31.46	heptadecene-(8)-carbonic acid	C ₁₈ H ₃₄ O ₃	298



Table 3: Qualitative analysis of organic compounds presented in the various fractions from samples of soot extracts (pine, oak, rubber, palm oil)

Type of Soot	Compound group	Rt (min)	Compound name	formula	M.wt
Oak	<i>n</i> -Alkanes				
		33.98	Tricosane	C ₂₃ H ₄₈	324
		35.49	tetracosane	C ₂₄ H ₅₀	338
		30.80	Heneicosane	C ₂₁ H ₄₄	296
		10.24	Undecane	C ₁₁ H ₂₄	156
		12.88	Dodecane	C ₁₂ H ₂₆	170
		15.38	Tridecane	C ₁₃ H ₂₈	184
		17.13	Dodecane, 2,6,10-trimethyl	C ₁₅ H ₃₂	212
		19.90	Pentadecane	C ₁₅ H ₃₂	212
		17.70	Tetradecane	C ₁₄ H ₃₀	198
		18.20	Naphthalene, 1,5-dimethyl	C ₁₂ H ₁₂	156
		20.95	2,3,6-trimethylnaphthalene	C ₁₃ H ₁₄	170
		21.95	Hexadecane	C ₁₆ H ₃₄	226
		23.95	Pentadecane, 2,4,10,14-tetramethyl	C ₁₉ H ₄₀	268
		25.76	Octadecane	C ₁₈ H ₃₈	254
		27.53	Nonadecane	C ₁₉ H ₄₀	268
		29.20	Eicosane	C ₂₀ H ₄₂	282
		32.33	N-docosane	C ₂₂ H ₄₆	310
		36.47	17-pentatriacontene	C ₃₅ H ₇₀	490
		36.56	Pentacosan	C ₂₅ H ₅₂	352
		37.87	Hexacosane	C ₂₆ H ₅₄	366
Grass	<i>n</i> -Alkanes				
		32.40	tetratetracontane	C ₄₄ H ₉₀	619
		33.98	Tricosane	C ₂₃ H ₄₈	324
		35.49	Tetracosane	C ₂₄ H ₅₀	338
		30.80	Heneicosane	C ₂₁ H ₄₄	296
		39.20	2,6,10,15,19,23-hexamethyltetracosane	C ₃₀ H ₆₂	422
Pine					
		30.80	Heneicosane	C ₂₁ H ₄₄	296
		23.95	Pentadecane, 2,4,10,14-tetramethyl	C ₁₉ H ₄₀	268
palm oil	<i>n</i> -Alkanols				
		4.10	2,3-dimethylbutan-2-ol	C ₆ H ₁₄ O	102
Oak	<i>n</i> -Alkanoic acids				
		28.44	palmitic acid	C ₁₆ H ₃₂ O ₂	256
palm oil					
		4.57	4-hydroxybutanoic acid	C ₄ H ₈ O ₃	104
		28.44	palmitic acid	C ₁₆ H ₃₂ O ₂	256



(a) *n*-alkanes

The *n*-alkanes distributions attributable to biomass combustion emissions (Abas et al., 1995; and Simoneit, 1984a) and probably derived mainly from the dehydration of *n*-alkanols and by a lesser degree from reduction of *n*-alkanoic acids (Simoneit et al., 1999). In this study *n*-Alkanes were detected in some samples of PM₁₀ and the possible sources from soot samples as calculated and described Table (2 , 3) and plotted in Figure (2a, 2b). None of these distributions are the same and even the air sampling period shows slightly different of *n*-alkane distributions. This variation may reflect source attributes or additional local input. Nevertheless, the C_{max} at 29 or 31 reflects a more tropical source region (Simoneit, 1977, 1979), confirming the long-range transport of these aerosols from source of pollutant to the sampling locations. A minor contribution of *n*-alkanes from C₃₀ to C₃₄, especially for samples from Simpang Empat, Penang city and from C₂₈ to C₂₉ for UKM, Bangi stations (Table 2), should be pointed out. These long chain alkanes are interpreted to be the high molecular weight (410-487g/mol). As these compounds are found in emissions from other sources such as automobiles (Schauer and Kleeman et al., 2002) and vascular plant wax (Simoneit et al., 1991a).

(b) *n*-alkanols

The *n*-alkanols are exclusively, or predominantly, even-carbon-numbered and they occur in the (C₁₈ or C₂₀) to C₂₈ range. Normal primary of alkanols (fatty alcohols) C₁₈ were detected at UKM station after extracts. A similar distribution is observed for the C₁₈ alkanol and an origin from reduction of *n*-alkanoic acids is less likely. In this study *n*-Alkanols were detected in some samples of PM₁₀ and the possible sources from soot samples as calculated and described Table (2,3) and plotted Figure (2a, 2b). However, nonacosan-10-ol as the main source of *n*-Alkanols of waxes from soft and hardwoods (Simoneit, 2002) was not indicated in this study from both PM₁₀ and soot samples and also Octacosan-1,3-diol was not detected in both samples, Probably the formation of previous *n*-alkanes are derived mainly from the dehydration or reduction of *n*-alkanols (Danie, and Simoneit., et al. 1999 and Ambles *et al.*, 1985, 1989a) because a similar distribution is observed for the *n*-alkanols. Some of these alkanols may also be injected into the atmosphere by smoke from biomass burning (Oros and Simoneit, 2001a, b; Simoneit, 2002).



(c) *n*-alkanoic acids

The *n*-alkanoic acids are next major compound group were detected in this study from UKM station and from soot of palm oil and oak as the possible sources, consists of fatty acids range from C₁₄ to C₁₈, with palmitic acid as the dominant homolog (Table 2,3). The C₁₆ and C₁₈ fatty acids are the most prominent alkanolic acids found in urban atmospheric fine particulate matter (Rogge et al., 1993b) and interpreted to be of a biogenic origin from both natural and burning emissions. The homologs <n-C₂₀ may be derived in part from microbial sources, although these acids are ubiquitous in all biota (Simoneit and Mazurek, 1982). Another source in urban areas is from cooking, grilling and food preparation as well as burning, where these alkanolic acids are volatilized directly into the fumes (Rogge et al., 1991). However, the major resolved compound in smoke from the burning of bamboo leaves and twigs, sugar cane and forest litter from Malaysia is palmitic acid.

4. Conclusions

Organic matter of aerosol particles is derived from two major sources and is admixed depending on environmental conditions. These sources are natural biogenic detritus (e.g., plant wax, pollen, etc.) and anthropogenic emissions (e.g., soot, oils, etc.), which also include biomass burning smoke. Thus, various ratios of the major molecular groups in smoke (PM₁₀) and other biomarker tracers are useful for identifying the vegetation sources that were burned. Organic aerosol samples were collected throughout a complete annual cycle at two urban sites in Malaysia, extracted, and then analyzed by GC/MS.

GC-MS Conventional and reactive techniques are valuable for rapid characterization of the organic component in PM₁₀ and soot samples through the direct analysis of a few milligrams of sampling filter and soot sampling. All organic compounds identifications are based on comparisons with authentic standards, GC retention time, literature mass spectra, and interpretation of mass spectrometric fragmentation patterns. The pyrolytic techniques utilized to complement each other and provide information on a wide array of possible precursors (e.g. carbohydrates, lipids, synthetic rubbers, conifer resins) and enhance the reliability of the inferred structural data. Both conventional and reactive pyrolysis evidence the importance of



aliphatic structures and the low contribution of aromatic components (e.g. lignin phenols) in the investigated aerosol particles

The organic compounds were identified, classified, and grouped into different compounds. The total amount of the identified organic compounds, including aliphatic compounds (n-alkanes, n-alkanoic acids and n-alkanols) are commonly found in all biomass combustion emissions. However, most of these series of compounds are indicative of biomass burning, which when coupled with the directly emitted and thermally altered molecular markers can be used as key tracers for assessing and tracking emissions from biomass fuel burning

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