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## FULL LENGTH ARTICLE

# Occurrence and sources of aliphatic hydrocarbons in surface soils from Riyadh city, Saudi Arabia

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UCM

**Abstract** Soil particles contain a variety of anthropogenic and natural organic components derived from many sources such as industrial and traffic fossil fuel emissions and terrestrial biota. The organic contents of soil and sand from the Arabian region have not fully characterized. Thus, samples of fine soil particles (sieved to  $<125\ \mu\text{M}$ ) were collected from the Riyadh area in November 2006 (late summer) and February 2007 (late winter). The samples were extracted with a mixture of dichloromethane/hexane and analyzed by gas chromatography–mass spectroscopy (GCMS) in order to characterize the chemical composition and sources of aliphatic hydrocarbons. The results showed that both anthropogenic and natural biogenic inputs were the major sources of the aliphatic hydrocarbons in these extracts. Vehicular emission products and discarded plastics were the major anthropogenic sources in the fine particles of the soils and ranged from 64% to 96% in November 2006 and from 70% to 92% in February 2007. Their tracers were *n*-alkanes, hopanes, sterane, plasticizers and UCM. Vegetation was also a major natural source of hydrocarbon compounds in samples ranging from ~0% to 18% in November 2006 and from 1% to 13% in February 2007 and included *n*-alkanes and triterpenoids.

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## 1. Introduction

Surface soils contain organic matter derived from many sources such as natural terrestrial plant wax, urban and industrial emissions from fossil fuel use and smoke from biomass burning (Al-Mutlaq, 2006; Al-Mutlaq et al., 2002, 2007; Awny et al., 1995; Casalicchio and Lercker, 1974; Kindler et al., 2009; Mahato and Banerjee, 1980; Rushdi et al., 2004, 2005, 2006, 2010; Simoneit, 1998; Simoneit et al., 1983, 1999). The organic compounds in soils comprise fatty acids, sterols,

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*n*-alkanes, *n*-alkanols and other lipids from terrestrial higher plants; sugar-derivatives, methoxyphenols and resin acids from biomass burning; polynuclear aromatic hydrocarbons (PAHs) from combustion processes; pesticides and herbicides from agricultural activities; and dicarboxylic acids and other polar oxidation products (Al-Mutlaq et al., 2002, 2007; Rushdi et al., 2004, 2010; Simoneit, 1980, 1984, 1986, 1989).

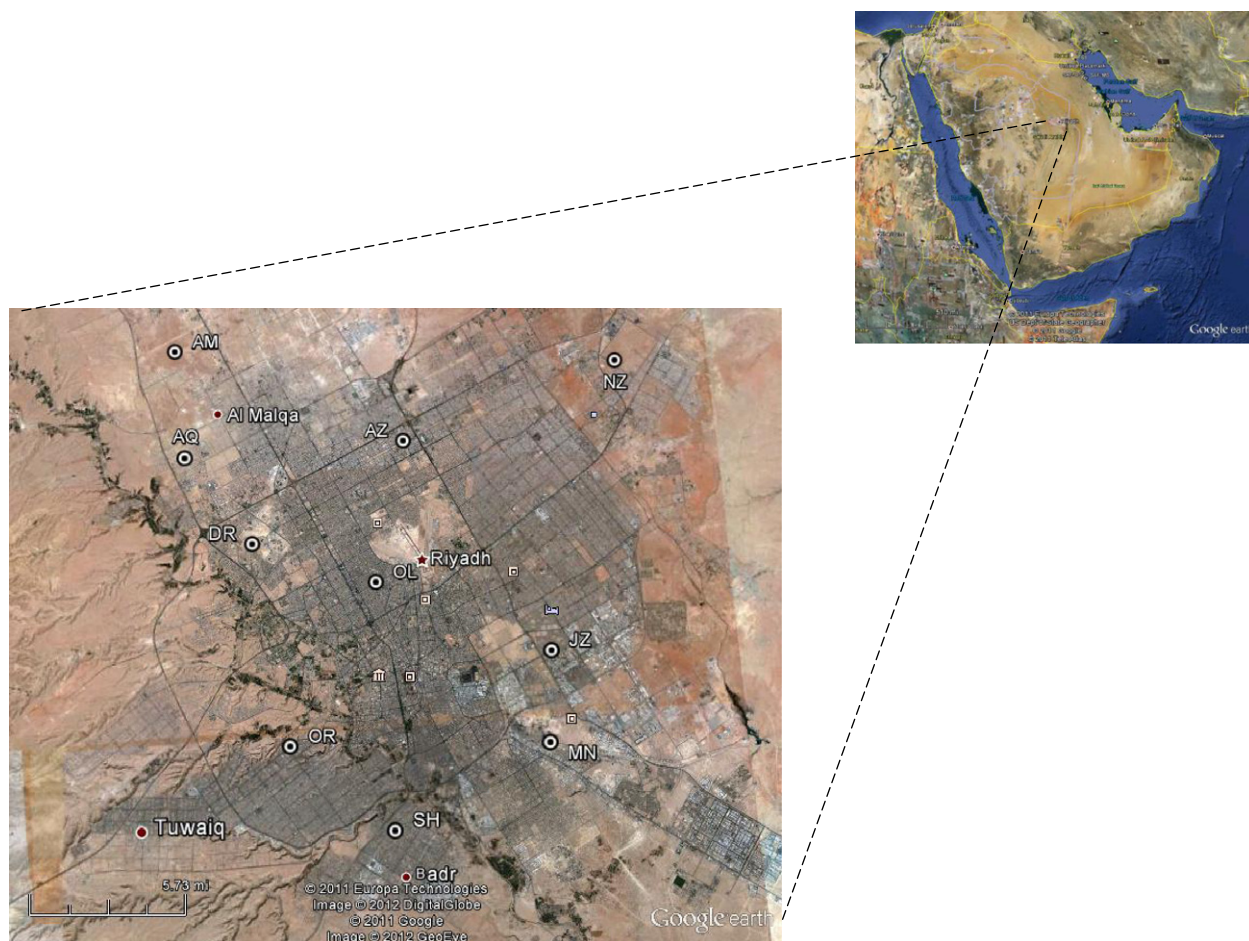
The organic contents of surface soil and sand have not been fully characterized for the Arabian Peninsula region. The preliminary results have shown that soil and sand dusts from Kuwait and Saudi Arabia contained a mixture of anthropogenic and natural organic matter (Al-Mutlaq, 2006, 2002, 2007; Rushdi et al., 2005, 2006). In the city of Riyadh, the anthropogenic and natural sources ranged from 30% to 55% and from 20% to 57% of the total lipid tracers, respectively (Rushdi et al., 2005). Soil and sand samples from the vicinity of Kuwait City showed that the anthropogenic sources ranged from 8% to 88% and the natural sources ranged from 15% to 78% of the total lipid compounds (Rushdi et al., 2006). Plasticizers, UCM, *n*-alkanes, hopanes steranes, *n*-alkanols, *n*-alkanoic acids, *n*-alkanes, methyl alkanoates, sterols and triterpenoids were major compounds and spatially varied (Al-Mutlaq et al., 2002, 2007; Rushdi et al., 2005, 2006). Therefore, the major purpose of this paper is to: (1) identify the higher molecular weight aliphatic hydrocarbon compounds in fine particles of soils from the metropolitan area of Riyadh,

Kingdom of Saudi Arabia, during two different times; (2) examine the relative changes in the source abundances of these organic tracers from the late summer of 2006 to the late winter of 2007 and (3) to evaluate the potential sources of the aliphatic hydrocarbons in the ambient atmosphere from soil and sand fine particles.

## 2. Experimental methods

### 2.1. Sampling sites

Soil samples were collected in November 2006 (late summer) and February 2007 (late winter) from the metropolitan area of Riyadh in Saudi Arabia to characterize the hydrocarbon contents by gas chromatography–mass spectrometry (GC–MS) analysis. The samples were collected by scraping the uppermost layers of the soils. The locations of the samples are shown in Fig. 1. All samples were collected in and around the city of Riyadh and represented urban areas ranging from highly populated with different human activities to rural areas where human activities are limited to agriculture. The sites included Diriyah (DR), Al-Aqeeq (AQ), Al-Amariah (AM), Al-Ezdihar (AZ), Al-Shifa (SH), Al-Jazirah (JZ), Al-Manakh (MN), Al-Oreja (OR), and Olya (OL). All samples were found to be mixtures of soil and sand, except sample AM, which was entirely soil.



**Figure 1** Map showing the sampling sites and locations of the soil samples.

## 2.2. Extraction procedure

The soil samples were collected in pre-extracted glass bottles using pre-cleaned spatula and stored in freezer. Each sample was sieved to obtain fine particles ( $< 125 \mu\text{m}$ ) before total organic matter extraction. The extraction was performed three times by adding methylene chloride/hexane (40 mL 2:1 v/v) to about 5 g of the sieved fine particles of each sample. The sample was ultrasonicated for 20 min and then filtering through pre-extracted glass microfiber filters (*Whatman*<sup>®</sup>, GF/A filters) before extract collection. Each total extract was concentrated under nitrogen blow-down at room temperature to approximately 1.0–1.5 mL before GC–MS analysis.

Blanks of sampling bottles (used for sample collections), solvents (used for sample extractions) and quartz filter papers (used for extract separation) were tested for possible contaminants. The analyses showed that all blanks were contamination free. A pre-extracted soil was spiked with a hexacosane standard to estimate the recovery of the extraction procedure, which was found to range from 87% to 91%.

## 2.3. Instrumental analysis

The analyses of the total extracts were carried out by GC–MS, using a Hewlett–Packard 6890 GC coupled to a 5973 Mass Selective Detector with a DB-5 (*Agilent*) fused silica capillary column (30 m  $\times$  0.25 mm i.d., 0.25  $\mu\text{m}$  film thickness) and helium as the carrier gas. The GC was temperature programmed from 65  $^{\circ}\text{C}$  (2 min initial time) to 300  $^{\circ}\text{C}$  at 6  $^{\circ}\text{C min}^{-1}$  (isothermal for 20 min final time). The MS was operated in the electron impact mode at 70 eV ion source energy. The data were acquired and processed with a Hewlett–Packard ChemStation.

Compounds were identified by GC retention index and comparison of mass spectra with those of authentic standards, literature and library data, and characterized mixtures. The relative concentrations of the identified compounds were determined by the integration of the relative responses of their peaks. Unknown compounds were characterized by interpretation of the fragmentation pattern of their mass spectra.

## 3. Results

The soil and sand fine particle of the hydrocarbon total extracts from the metropolitan Riyadh area are given in Table 1. They varied in contents and levels depending on the locations and human activities. This variation is illustrated in the samples from two different sites of the city (Fig. 2). The extractable hydrocarbon compounds consisted mainly mixtures of *n*-alkanes, hopanes, steranes, plasticizers, UCM (unresolved complex mixture of branched and cyclic hydrocarbons) and triterpenoids.

The *n*-alkanes were detected in all samples and ranged from  $\text{C}_{15}$  to  $\text{C}_{37}$  with a maximum concentration ( $C_{\text{max}}$ ) at heptacosane or nonacosane ( $\text{C}_{27}$  or  $\text{C}_{29}$ , Table 1, Figs. 2a, and 3a). The relative concentrations of total *n*-alkanes ranged from 11% to 33% of the total extract.

Hopane biomarkers were found mainly in all samples. They ranged from  $\text{C}_{27}$  to  $\text{C}_{35}$  with a  $C_{\text{max}}$  at 29 or 30, (e.g., Fig. 3c) and the relative concentrations ranged from  $\sim 0.00$  to 8.7% of the total hydrocarbons. The  $\text{C}_{27}$  to  $\text{C}_{29}$  sterane biomarkers were detectable in all samples (e.g., Fig. 3d) ranging between

$\sim 0.00\%$  and 13% of the total extracts. Plasticizers, including diethyl-, isobutyl-, dibutyl- bis(2-ethylhexyl)- and dioctyl-phthalates, were significant components of these samples (Table 1; Figs. 2 and 3f), with relative concentrations of 7.4% to 74.7%. (See Fig. 4).

Triterpenoids were detected in all samples, with relative concentrations ranging from  $\sim 0.00\%$  to 7.0%. They were mainly  $\alpha$ - and  $\beta$ -amyryns and  $\alpha$ - and  $\beta$ -amyrones (Fig. 3e).

## 4. Discussion

The presence of *n*-alkanes (in part), biomarkers (e.g., hopanes and steranes, Fig. 3c and d and Table 1), and UCM, as well as plasticizers in the samples (Table 1 and Fig. 3f) indicate anthropogenic inputs from traffic and litter (plastics) (Abas and Simoneit, 1996; Al-Mutlaq et al., 2002; Broddin et al., 1980; Rogge et al., 1993a,b; Rushdi et al., 2005, 2010; Simoneit, 1984, 1986, 1998).

The presence of *n*-alkanes with a carbon number predominance of about unity (i.e., CPI  $\sim 1.0$ , Simoneit et al., 1991a) suggests a source from vehicle emissions. Crude petroleum, the primary source of gasoline, diesel fuel, and lubricating oil, contains *n*-alkanes ranging up to about  $\text{C}_{35}$  with essentially no carbon number predominance (CPI  $\sim 1$ ) (Bray and Evans, 1961; Cooper and Bray, 1963; Rogge et al., 1993c) with lower carbon number maxima in the range of  $\text{C}_{22}$  to  $\text{C}_{25}$  (Hunt, 1979; Simoneit, 1984). The results indicate that fossil fuel utilization is a major input of organic matter to the samples of Al-Manakh, Al-Amariah, Al-Jazirah, Al-Ezdihar and Olya, where high traffic and human activities are found. The lowest inputs were observed in the soils from the Diriyah, Al-Aqeeq and Al-Amariah of less traffic and industrial activities.

The presence of hopane and sterane biomarkers in the soil samples confirms the input from traffic emissions (Simoneit, 1984). Hopanes and steranes are not present in gasoline and diesel fuel because they belong to the higher boiling fraction of crude petroleum, but they are found in lubricating oil. Hopanes and steranes are present in exhaust particles from both auto and diesel engines (Simoneit, 1984, 1985). These particles fall out and become entrained in urban road soils and sands. The C-22 S/(R + S) ratios of the homohopanes ( $\text{C}_{31}$  and  $\text{C}_{32}$ ), which have been used as maturity indices for petroleum (Ensminger et al., 1974; Seifert and Moldowan, 1978), range between 0.55 and 0.63 in samples from the city further indicating anthropogenic petroleum inputs (Table 1) The data confirm that crude oil products are one of the sources of the organic matter in the soils. The major petroleum component is derived from lubricating oil in vehicle emissions, and that with direct oil from vehicles is represented by the UCM (Table 1 and Fig. 2). These petroleum residues were not detected in samples from Diriyah, Al-Aqeeq and Al-Amariah, although a minor UCM is also present. The absence of significant concentrations of PAHs indicates that organic compounds from biomass burning, i.e., soot fallout (Simoneit, 1998; Simoneit et al., 1999) are not a major input.

Shopping bags and discarded plastics are likely the major sources of the plasticizers, because they were not detected in blanks from solvents, sample containers, or analytical procedures.

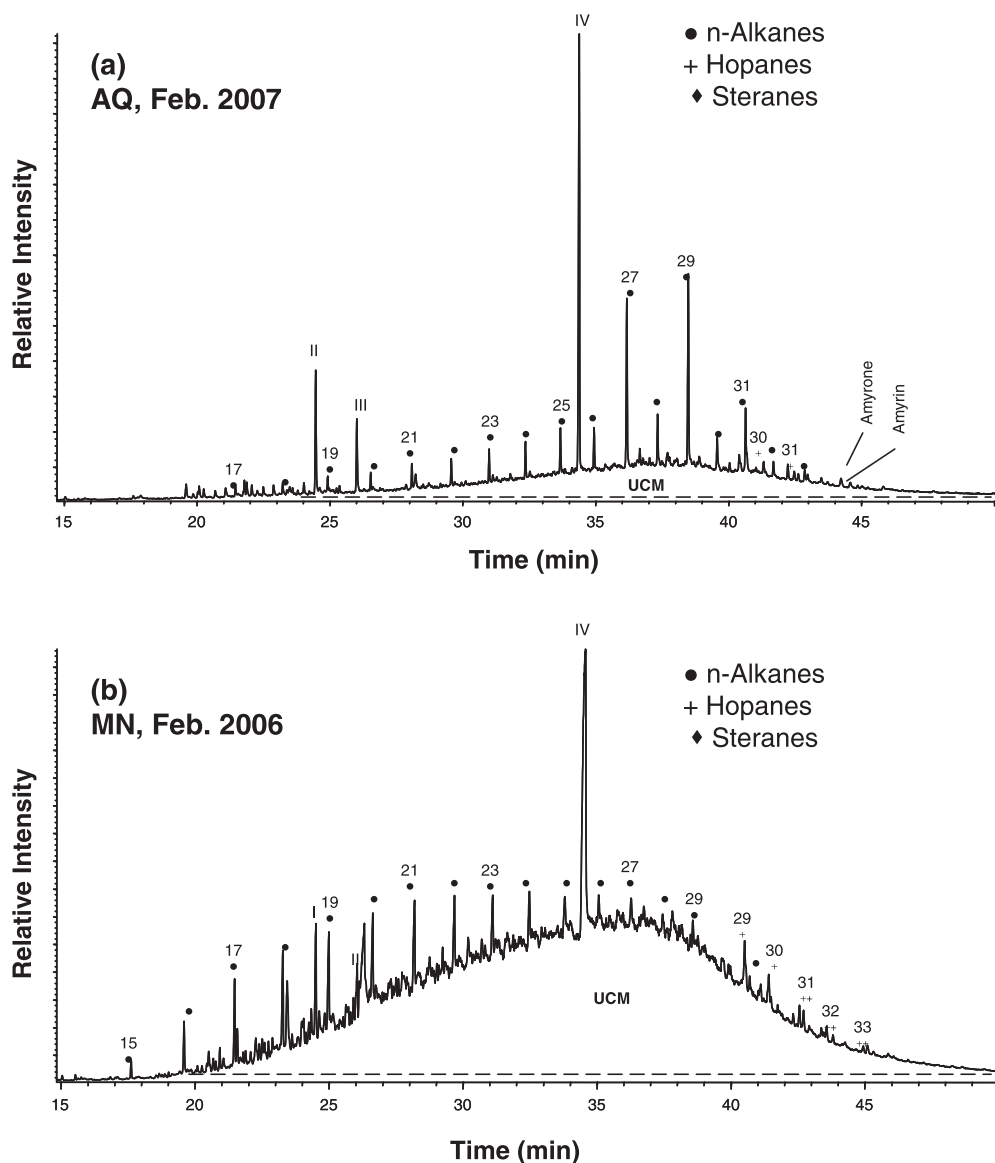
The occurrence of wax *n*-alkanes and triterpenoids validates the natural source input from higher plant biomass

**Table 1** Relative concentration (%) and various parameters of solvent-extractable organic matter of soil samples from the Riyadh area, Saudi Arabia.

	AD		AG		AM		AZ		SH		JZ		MN		OR		OL	
November-2006	February-2007	November-2006	February-2007	November-2006	February-2007	November-2006	February-2007	November-2006	February-2007	November-2006	February-2007	November-2006	February-2007	November-2006	February-2007	November-2006	February-2007	
<i>n-Alkanes</i>																		
Total	10.9	20.2	26.3	33.9	25.5	Low	26.2	20.73	16.57	24.84	24.72	24.69	22.34	11.41	9.16	10.78	16.67	14.86
Range	21–33	14–37	14–37	14–37	15–36		14–37	14–37	14–37	14–37	14–37	14–37	14–37	14–37	14–37	14–37	14–37	14–37
C <sub>max</sub>	29	29	29	29	23		27,29	27	29	27,29	29	27	21,22	19,20	25	25,27	29	29
CPI (o/e) <sup>a</sup>	3.47	2.09	1.71	2.23	1.02		3.15	1.60	2.06	3.10	1.95	1.31	1.11	1.02	1.29	1.32	1.45	1.27
CPI (o/e) (15–26)	1.21	1.35	0.95	0.94	0.91		1.10	0.97	0.96	1.10	0.93	0.93	0.96	0.97	0.91	0.86	0.96	0.94
CPI (o/e) (25–35)	3.60	2.20	1.90	2.58	0.80		3.65	1.90	2.12	3.73	2.33	1.31	1.00	0.77	1.24	1.27	1.47	1.28
T(15–24)/(25–35) <sup>b</sup>	0.19	0.59	0.56	0.32	1.94		0.19	0.79	0.22	0.22	0.54	0.78	2.18	4.07	0.57	0.33		0.70
<i>Wax n-alkanes</i>																		
Total	5.1	5.0	5.9	12.2	T		12.99	4.09	5.26	12.34	7.05	2.37	0.48	0.01	0.79	1.23	2.52	1.33
<i>Hopane biomarkers</i>																		
Total	T	T	3.9	1.0	T		2.19	4.27	1.20	1.90	3.90	6.44	5.37	8.68	2.79	2.59	6.94	6.29
Range			27–35	27–35			27–35	27–35	27–35	27–35	27–35	27–35	27–35	27–35	27–35	27–35	27–35	27–35
C <sub>max</sub>			29	29			29	29	29	29	30	29	29	30	29,30	29	30	30
C <sub>31</sub> S/(R + S) <sup>c</sup>			0.58	0.59			0.58	0.56	0.57	0.58	0.58	0.60	0.57	0.55	0.57	0.55	0.57	0.57
C <sub>32</sub> S/(R + S) <sup>c</sup>			0.60	0.62			0.60	0.63	0.60	0.61	0.63	0.62	0.62	0.61	0.60	0.63	0.63	0.60
<i>Sterane biomarkers</i>																		
Total	T	T	1.5	0.6	T		0.73	2.75	0.78	T	2.84	3.06	7.65	12.98	0.84	0.98	2.06	2.06
Range			27–29	27–29			27–29	27–29	27–29		27–29	27–29	27–29	27–29	27–29	27–29	27–29	27–29
C <sub>max</sub>			29	29			29	27,29	29		29	27	27	27	27	27	27	27
<i>Plasticizers</i>																		
Total	53.5	49.1	39.7	30.1	60.8		30.11	27.75	53.04	46.71	20.62	26.68	7.36	14.47	74.72	69.72	44.42	50.18
<i>Triterpenoids</i>																		
Total	2.4	0.0	4.4	3.2	0.0		4.83	0.62	1.53	0.74	6.98	0.16	0.17	0.00	0.77	0.72	0.65	0.00
UCM	2.2	6.0	10.5	15.7	5.7		14.76	28.13	11.84	9.31	23.27	30.86	52.66	45.62	5.09	6.74	18.75	19.11
U:R	0.20	0.30	0.40	0.46	0.10		0.56	1.36	0.71	0.38	0.94	1.25	2.36	4.00	0.56	0.63	1.13	1.29
Unknown	25.9	19.7	7.7	3.2	8.0		8.15	11.67	9.78	4.15	10.62	5.74	3.97	6.83	5.84	7.24	8.00	6.17

T = trace.

<sup>a</sup> CPI =  $\sum(C_{15} + C_{17} + \dots + C_{37}) / \sum(C_{14} + C_{16} + \dots + C_{36})$ .<sup>b</sup> T =  $\sum(C_{15} + C_{16} + \dots + C_{24}) / \sum(C_{25} + C_{26} + \dots + C_{37})$ .<sup>c</sup> 22S/(22S + 22R) for C<sub>31</sub> and C<sub>32</sub>.



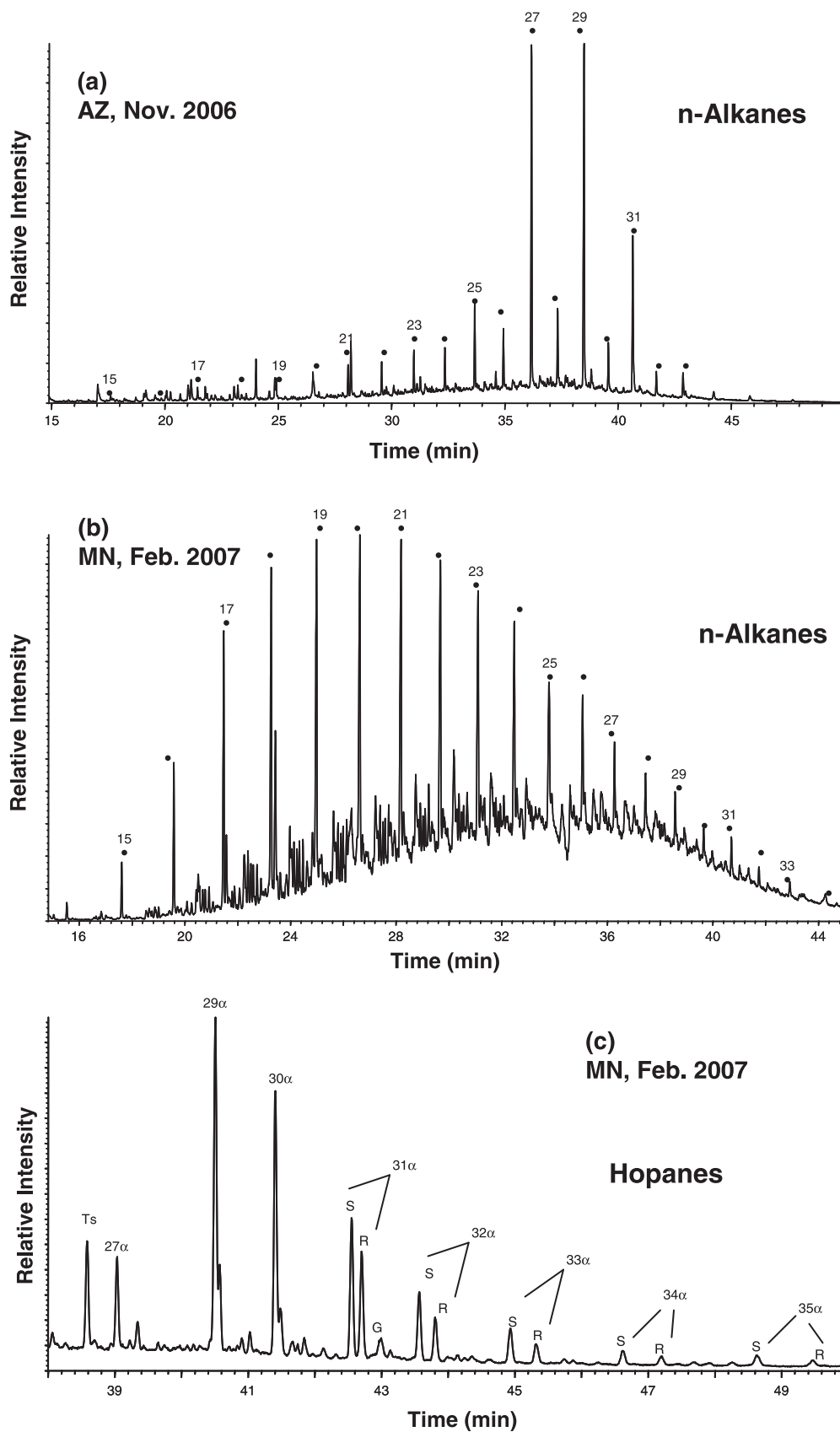
**Figure 2** GC-MS total ion current traces of total extracts of the soil samples from the city of Riyadh showing the major organic components in February 2007: (a) AQ (Aqeeq site); (b) MN (Al-Manakh site) (Numbers refer to the carbon chain length and symbols are: I = diethyl-, II = isobutyl-, III = dibutyl-, IV = diethylhexyl phthalate (octoil), V = dioctyl phthalate, H = hopanes, and UCM = unresolved complex mixture).

(Broddin et al., 1980; Barbier et al., 1981; Oros et al., 1999; Rogge et al., 1993d; Rushdi et al., 2010; Simoneit, 1977, 1980, 1986; Simoneit et al., 1991b,c; Standley and Simoneit, 1987). Plant wax *n*-alkanes show a  $C_{max}$  in the range of  $C_{25}$  to  $C_{33}$ , which depends on the plant species as well as the season and locality (e.g., Abas and Simoneit, 1998; Eglinton and Hamilton, 1967; Mazurek et al., 1991; Rogge et al., 1993c; Rushdi et al., 2006; Simoneit and Mazurek, 1982; Stephanou and Stratigakis, 1993). A CPI value of  $>1.0$  is an indicative of a contribution from natural sources, whereas a  $CPI \sim 1.0$ , as previously discussed, indicates a source from fossil fuel combustion (Hunt, 1979; Mazurek and Simoneit, 1984; Simoneit, 1984). The odd numbered *n*-alkanes were dominant in the Diriyah, Al-Aqeeq, Al-Ezdihar, Al-Shifa and Al-Jazirah samples, with CPI ranging from 1.60 to 3.47. The samples from

Al-Amariah, Al-Manakh, Al-Oreja and Olya showed no strong number carbon preference with CPIs ranging from 1.02 to 1.43 (Table 1). This odd carbon number preference ( $CPI > 1$ ) of the *n*-alkanes supported by the  $C_{max}$  mainly at 27 and/or 29 is distinctive of higher plant waxes (e.g., Table 1, Fig. 3a, Simoneit, 1989).

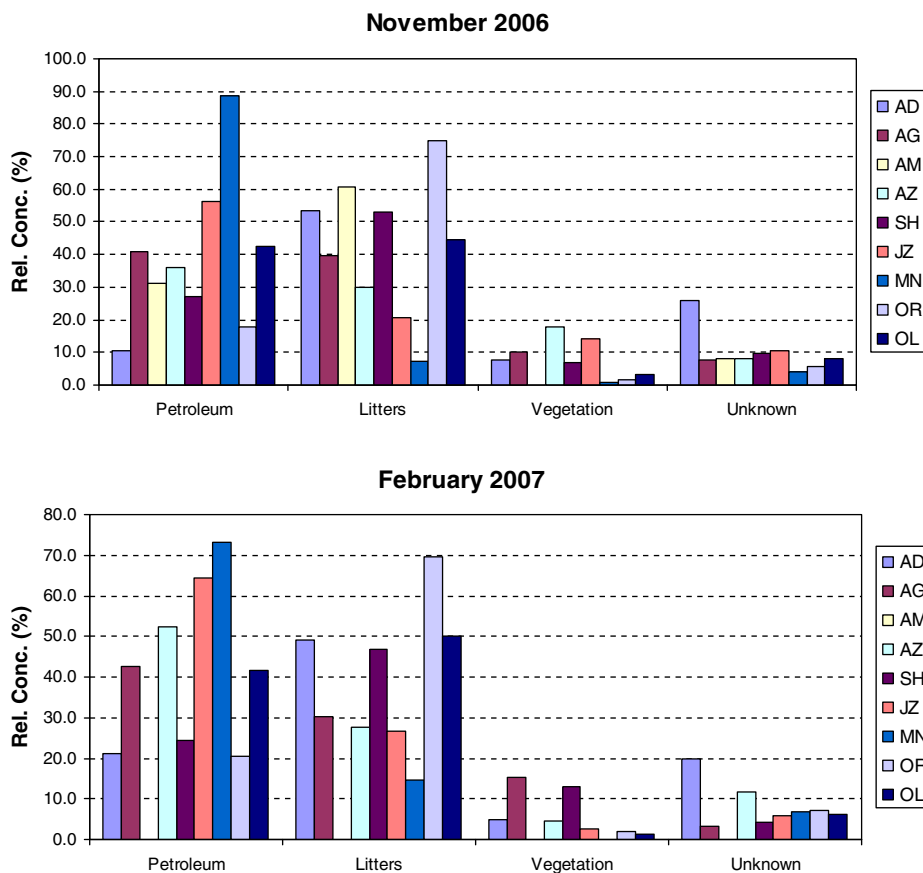
Triterpenoids can be derived from higher vascular plant waxes, microbial activities in soil, and cooking (Barbier et al., 1981; Boon et al., 1979; Huang and Meinschein, 1976; Lee et al., 1979; Rogge et al., 1991; Simoneit, 1989; Simoneit et al., 1983). The triterpenoids ( $\alpha$ - and  $\beta$ -amyryns and  $\alpha$ - and  $\beta$ -amyrones) also originate from vascular plant waxes (Simoneit, 1989) (Table 1).

Briefly, the results of organic chemical analysis of the soil fine particles samples from metropolitan Riyadh show that



**Figure 3** Examples of typical GC-MS key ion plots for various compound series found in the soil samples: (a) and (b)  $m/z$  85 *n*-alkanes (CPI = 3.15 and 1.02 for AQ and MN, respectively); (c)  $m/z$  191 hopanes; (d)  $m/z$  217 and 218 steranes, and (e)  $m/z$  218 amyrene and amyrin; (f)  $m/z$  149 plasticizers (symbols as in Fig. 2).





**Figure 4** The percent fractions of various sources of hydrocarbons in soil samples from the city of Riyadh- Saudi Arabia.

anthropogenic and biogenic organic matter are the major sources of lipid compounds in soils. These sources are from vehicle emissions, urban activities and regional vegetation. The occurrence of *n*-alkanes, hopane and sterane biomarkers, and UCM indicates anthropogenic sources from petroleum product utilization (e.g., traffic emissions). The presence of a mixture of various lipid classes in the total extracts is an indication of natural biogenic sources, mainly from the surrounding vegetation, microbial/fungal biomass and detrital organic matter in soils.

It is obvious that the contribution of anthropogenic versus natural biogenic sources varies and depends on the locations of the samples and the season (Fig. 4). The anthropogenic sources in the city of Riyadh during late winter (February 2007) and late summer (November 2006) were  $73 \pm 29$  and  $82 \pm 12\%$  of the total hydrocarbons, respectively. The main anthropogenic sources of hydrocarbons include fossil fuel combustion [ $38 \pm 23\%$  (late winter) and  $39 \pm 23\%$  (late summer), based on *n*-alkanes, hopanes, steranes and UCM] and market places and leisure related activities [ $35 \pm 21\%$  (late winter) and  $42 \pm 44\%$  (late summer) based on plasticizers]. In general there is an increase in anthropogenic relative to natural lipid compounds in late winter. The results indicate that there is no significant seasonal change in the percentages of fossil fuel combustion in the city between late winter and late summer, indicating that traffic is the major source of anthropogenic organic matter and pollutants throughout the year. Littering, which is most likely another major source of organic matter in the city, also shows no significant variation.

The contributions of natural sources, which are mainly from vegetation, were  $5 \pm 6\%$  in late winter and  $7 \pm 6\%$  in late summer of the total hydrocarbons in the city. The contribution of natural organic matter outside the city did not change between late winter and late summer. The natural sources of lipid compounds are higher in rural and surrounding areas relative to the city center.

## 5. Conclusions

The analyses of fine soil particles from the Riyadh metropolitan area show that both anthropogenic and natural biogenic sources contribute to their organic matter contents. Anthropogenic sources are characterized by the presence of *n*-alkanes with CPI  $\sim 1$ , hopane and sterane biomarkers, and UCM from urban traffic emissions and plasticizers from plastic litter and detritus (e.g., shopping bags). The anthropogenic sources comprise between 70–92% (late winter) and 64–96% (late summer) of the total extractable hydrocarbons. The abundances of anthropogenic organic compounds in the city depend on the location, season, and types of urban activities. They are at relatively low levels in the rural samples.

The natural sources of organic compounds are primarily from higher plant wax (i.e., vegetation) and amount to 1–13% (late winter) and 0.0–18% (late summer) of the total hydrocarbons of the soils and sands. The natural components of the lipid compounds include *n*-alkanes and triterpenoids. Their concentrations are lower in the city compared to the surrounding rural areas.



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