DISSOLVED AND PARTICULATE-ADSORBED HYDROCARBONS IN THE WATERS OF SHATT AL-ARAB RIVER, IRAQ

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(Received January 14, 1991; revised May 6, 1991)

Abstract. N-alkanes were measured both in dissolved and particulate-adsorbed phases of water samples collected from five stations located along Shatt al-Arab River in Iraq. The aim of this study was to provide a background information on the presence, origin, and distribution of n-alkanes in the waters of Shatt al-Arab River. Total concentrations of n-alkanes were lower in the dissolved phase (29.37 $\mu g\ L^{-1}$) than in the particulate-adsorbed fraction (17.62 $\mu g\ g^{-1}$ dw). Distribution of n-alkanes in both fractions is characterized by two distinct patterns. First , for C compounds numbered C_{14} to C_{22}, the 'odd' numbered hydrocarbons were slightly more abundant with C_{17} being the most concentrated. The second is shown within n-alkanes greater than C_{22}. In these the 'odd' numbered hydrocarbons show even greater predominance indicating that much of the n-alkanes in the waters of Shatt al-Arab, may have originated from aquatic biogenic sources; *i.e.*, diatoms, algal forms, bacterial activities, and higher plants. The CPI values were found to be from 0.99 to 1.98 in the dissolved fraction and from 0.70 to 2.10 in the particulate fraction. Petroleum hydrocarbon inputs and urban related oil discharges (anthropogenic pollution) may also have contributed to the input of n-alkanes in Shatt al-Arab, particularly near the city of Basrah (St. IV). Total concentrations at station IV were much higher (8.40 $\mu g\ L^{-1}$ and 5.80 $\mu g\ g^{-1}$) than that for the most upstream station, # I, (3.20 $\mu g\ L^{-1}$ and 1.48 $\mu g\ g^{-1}$) and decreased again further downstream at station V (7.10 $\mu g\ L^{-1}$ and 4.32 $\mu g\ g^{-1}$). The CPI values were around 1.0 in station IV. In a conclusion, results obtained in this study indicate that the n-alkanes in the waters of Shatt al-Arab River are of aquatic biogenic and anthropogenic (petroleum related) origins.

1. Introduction

Petroleum hydrocarbons fall into three major categories; alkanes of aliphatic hydrocarbons, paraffins; alkenes, alkynes; and, aromatic hydrocarbons (D.O.I., 1989). Organic compounds in natural waters can have deleterious effects on water color, odor, and taste, and be toxic to aquatic organisms (Telang *et al.*, 1981). The present study considers hydrocarbons in the major river in southern Iraq (Figure 1).

Hydrocarbons present in the aquatic ecosystem may be of natural and/or anthropogenic origin. Those of natural origin enters the aquatic ecosystem by overland run-off, or ground water seepage. Also, they may be released into water by biogenic activities of the fauna, flora, and/or bacteria. Anthropogenic hydrocarbons can enter water bodies through several routes; airborne fallout, domestic and agricultural discharges, industrial wastes and outfalls, dredging, commercial

Water, Air, and Soil Pollution **61**: 397–408, 1992.

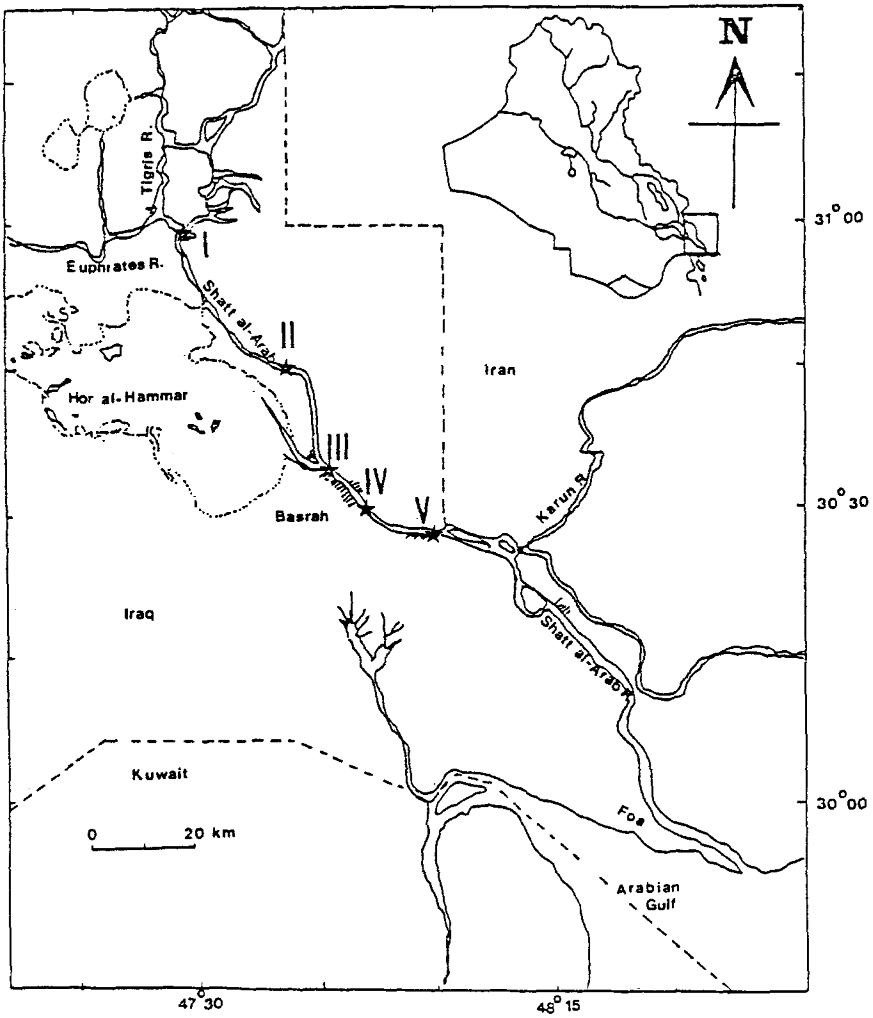

Fig. 1. Sampling stations (I-V) along Shatt al-Arab River, Iraq.

ship and boat activities, oil industry and transportation activities.

Solubility of hydrocarbons in aqueous media is relatively low. The reported concentrations in fresh water samples were found to be from 0.3 to 1.0 $\mu g\ L^{-1}$, and, in sea water samples from 0.5 to 800 $\mu g\ L^{-1}$ (Telang *et al.*, 1981). In sediment, hydrocarbons concentrations were found to be from 10.0 to 3 800 $\mu g\ g^{-1}$ (Hunt, 1961; Telang *et al.*, 1981). Hence, they readily adsorb to the sediment.

In the water systems, hydrocarbons are often associated with suspended particles. As a general rule, particles larger than 0.45 μm are called particulate and less than 0.45 μm as dissolved matter. Accordingly, hydrocarbons that are associated with the smaller fraction are classified as 'dissolved hydrocarbons' and those that are associated with the larger particles (> 0.45 μm) are called 'particulate hydrocarbons' (Cauwet, 1978; Eganhouse and Kaplan, 1981).

The basin of the Shatt al-Arab River has been reported to receive annually considerable amounts of hydrocarbons. Al-Saad (1983) indicated that both biogenic and anthropogenic inputs are transported in the form of dissolved and particulate-adsorbed organic matter within the water column of this economically important water body.

The objective of this study was to gather base-line data concerning the presence, origin, types, and distribution of the normal alkanes in the waters and sediments of Shatt al-Arab River. Due to the extensive oil drilling and transportation activities in the Southern Iraq and the Arabian Gulf region there is increase possibility of oil pollution in the regional ecosystem. Hence, base-line information is necessary for future protection of the estuarine and marine environments in the Gulf area (DouAbul and Bedair, 1986).

2. Materials and Methods

Five stations (I–V) were selected along the longitudinal section of the Shatt al-Arab in southern Iraq (Figure 1) during 1985. Water samples (30 L) were collected from each station at 1 m depth with acid-washed 5-L dark-glass aspirators. All glassware was solvent-washed prior to and after each use.

Within 5 hr, in the laboratory, samples were suction-filtered through 0.45 μm pore size Watman glass fiber (GF/F) filters, which were preignited at 450 °C and preweighted. Material which passed the filters was considered as the 'dissolved fraction', while that retained was considered as the 'particulate fraction'. Filters with particulate fractions were wrapped in preheated, solvent-washed, aluminum foil and stored at approximately minus 20 °C. Unused GF/F filters were similarly stored to provide blanks for the analysis.

Dissolved hydrocarbons were extracted using CCl_4 according to procedure of UNESCO (1976). A total volume of 100 mL CCl_4 was used in two successive extractions. The extracts were combined and anhydrous sodium sulfate was added to remove residual excess water, and to break any emulsion within the extracts. The CCl_4 extract was reduced in volume to approximately 10 mL with a rotary evaporator. The reduced extract was pipetted into a precleaned glass vial. Care was taken to exclude any residual particles of the sodium sulfate. The extract was then evaporated to dryness using a stream of highly purified N_2. The vial was then rinsed twice with fresh hexane. The final volume of hexane was 5 mL. Column chromatography was used for the isolation of the dissolved part. An extract was applied to a column composed of 20 g of deactivated florisil and eluted with 50 mL of hexane. The elutant was further reduced in volume and transferred to a clean brown vial. The vial was sealed, labeled, and kept refrigerated for subsequent analysis by gas chromatography.

Particulate fractions were extracted according to Farrington and Tripp (1975). Briefly, the GF/F filters were placed in solvent pre-extracted cellulose thimbles.

The filters were soxhlet extracted with a mixture of 1:1 bezene:methanol for a 24 hr period. The extract was then reduced in volume to 10 mL with a rotary evaporator and saponified for a 2 hr period with a solution of 4N KOH in 1:1 benzene:methanol. The unsaponified matter was extracted with hexane. The sample was passed through a chromatographic column containing 5% deactivated florisil. Each sample was applied to a column with a very small volume of hexane. N-alkanes were then eluted by the addition of an additional 50 mL of n-hexane. The elutant was reduced in volume, transferred to a brown glass vial, sealed, labeled, and kept refrigerated for gas chromatography.

Considerable caution was taken to minimize contamination of the samples. Procedure blanks consisting of reagents, glassware, and equipment were run regularly. Recoveries were calculated by comparing the peak areas of an internal standard of n-alkanes with a reference solution. The internal standard may have caused a slight interference with the determination, however, the recovery rates were always 90% or higher.

The analyses of the dissolved and particulate-adsorbed n-alkanes were performed with a Perkin-Elmer Sigma-300 capillary gas chromatograph, equipped with flame ionization detector (GC/FID) and a split-less injection port. Quantification of peaks and identification of n-alkanes in chromatograms were achieved using a Perkin-Elmer integrator (Model LC-100). The fused silica capillary column used in the analyses was a wall coated open tubular (WCOT) 50 m × 0.25 mm i.d., SE-30 type. Helium was used as a carrier gas with a linear velocity of 1.5 mL min^{-1}. Operating temperatures for detector and injector were 310 and 300 °C, respectively. The isomers were separated using a programmable temperature regulator, which permits a temperature increment of 4 °C min^{-1}, from 60 to 280 °C, with an isothermal period of 30 min at the end of each injection. The unresolved complex mixture (UCM), which is also called the unresolved envelop, was measured by planimetry.

Statistical analyses for the comparison between samples and stations were carried out using the software system for data analysis (SAS, 1985) procedures. Analysis of variance (ANOVA) procedure was used for the multi, pairwise comparisons, the 0.95 confidence limits, and differences between the means.

3. Results and Discussions

Components and parameters measured in this study are listed in Tables I, II, and III as 'dissolved', 'particulate-adsorbed n-alkanes', and their 'various parametric ratios', respectively.

Estimates of concentrations in the dissolved fraction ranged from 3.20 to 8.40 $\mu\text{g L}^{-1}$ and for the particulate-adsorbed fraction from 1.48 to 5.80 $\mu\text{g g}^{-1}$ dw. In general, the total concentration of normal alkanes in the dissolved fraction was lower (29.37 $\mu\text{g L}^{-1}$) than the total concentration of n-alkanes in the particulate fraction (17.62 $\mu\text{g g}^{-1}$ dw). From above, it can be predicted that total n-alkanes

TABLE I

Concentrations of n-alkanes ($\mu g\ L^{-1}$) in the dissolved fraction, in Shatt al-Arab water samples

Carbon #	Station I	II	III	IV	V	Total
C13	0.13	0.17	0.18	0.28	0.21	0.97
C14	0.12	0.18	0.19	0.32	0.23	1.04
C15	0.11	0.16	0.21	0.58	0.25	1.31
C16	0.14	0.17	0.26	0.26	0.26	1.09
C17	0.15	0.19	0.29	0.28	0.28	1.19
C18	0.16	0.20	0.36	0.31	0.32	1.35
C19	0.18	0.28	0.37	0.30	0.48	1.61
C20	0.14	0.29	0.38	0.30	0.40	1.51
C21	0.13	0.21	0.37	0.31	0.36	1.38
C22	0.12	0.19	0.36	0.32	0.29	1.28
C23	0.15	0.18	0.37	0.35	0.29	1.34
C24	0.11	0.20	0.35	0.46	0.28	1.40
C25	0.21	0.30	0.33	0.54	0.35	1.73
C26	0.16	0.27	0.30	0.50	0.32	1.55
C27	0.12	0.18	0.25	0.44	0.28	1.27
C28	0.15	0.19	0.24	0.38	0.26	1.22
C29	0.20	0.18	0.23	0.36	0.26	1.23
C30	0.18	0.17	0.22	0.34	0.25	1.16
C31	0.16	0.16	0.21	0.32	0.24	1.09
C32	0.11	0.18	0.19	0.30	0.27	1.05
C33	0.13	0.17	0.17	0.31	0.26	1.04
C34	0.14	0.15	0.16	0.30	0.24	0.99
C35	–	–	0.15	0.33	0.25	0.73
C36	–	–	0.16	0.21	0.24	0.61
C37	–	–	–	–	0.23	0.23
Total	3.20	4.37	6.30	8.40	7.10	29.37
CPI[a]	1.09	0.99	1.98	1.10	1.05	

[a] CPI is the carbon preference index. It can be calculated by:

$$\mathrm{CPI} = \frac{\text{Sum of conc. of odd C n-alkanes}}{\text{Sum of conc. of even C n-alkanes}}\ .$$

in the particulate phase is 600 times more concentrated than that of the dissolved phase.

A similar pattern was revealed at each individual site; *i.e.*, each site had a total concentration of the dissolved fraction that was considerably lower than that of the particulate fraction (Table I and II). Concentrations of the dissolved n-alkanes at the five stations are graphically presented in Figure 2. In the same manner, concentrations of the particulate n-alkanes are presented in Figure 3.

Statistical analyses are presented in Tables IV, and V for the 'pairwise comparisons' between stations on basis of the total dissolved and particulate-adsorbed n-alkane fractions. These statistics indicate that station IV (near City of Basrah) is significantly high in both dissolved and particulate-adsorbed n-alkanes burdens. Stations V, III, II, and I had lower levels of dissolved fraction (Table IV), and stations V, II, III,

TABLE II

Concentrations of n-alkanes (μg g^{-1}) in the particulate fraction, in Shatt al-Arab water samples

Carbon #	Station I	II	III	IV	V	Total
C13	0.10	0.10	–	–	0.09	0.29
C14	0.10	0.12	0.05	0.12	0.10	0.49
C15	0.07	0.10	0.06	0.13	0.10	0.46
C16	0.08	0.10	0.09	0.15	0.12	0.54
C17	0.09	0.13	0.18	0.17	0.18	0.75
C18	0.10	0.15	0.20	0.19	0.20	0.84
C19	0.06	0.13	0.23	0.20	0.22	0.84
C20	0.05	0.13	0.25	0.16	0.25	0.84
C21	0.08	0.15	0.20	0.17	0.26	0.86
C22	0.10	0.16	0.18	0.22	0.28	0.94
C23	0.17	0.17	0.16	0.25	0.40	1.15
C24	0.15	0.18	0.20	0.28	0.35	1.16
C25	0.13	0.26	0.19	0.45	0.36	1.39
C26	0.08	0.22	0.18	0.40	0.38	1.26
C27	0.07	0.20	0.15	0.39	0.33	1.14
C28	0.05	0.18	0.14	0.36	0.30	1.03
C29	–	–	0.13	0.30	0.24	0.67
C30	–	–	0.14	0.29	0.16	0.59
C31	–	–	0.13	0.27	–	0.40
C32	–	–	0.12	0.25	–	0.37
C33	–	–	0.12	0.24	–	0.36
C34	–	–	0.14	0.22	–	0.36
C35	–	–	0.11	0.21	–	0.32
C36	–	–	0.10	0.20	–	0.30
C37	–	–	0.09	0.18	–	0.27
Total	1.48	2.48	3.54	5.80	4.32	17.62
CPI[a]	1.08	1.00	0.97	1.04	1.01	

[a] As defined in Table I.

and I for the particulate-adsorbed fraction (Table V).

The carbon preference index (CPI), which represents the ratio of the total concentration of the odd numbered C n-alkanes to the total concentration of the even numbered C n-alkanes, has been used to infer the proportionate sources of hydrocarbons in water and sediments (see Telang *et al.*, 1981). In this study, the distribution of n-alkanes in the water samples of Shatt al-Arab River Estuary, in both fractions, was characterized by the prevalence of two distinct features or patterns. The first is that in C numbered alkanes C_{14} to C_{22} 'odd' numbered hydrocarbons were slightly more abundant. Exceptions were C_{20} in the particulate phase, and a relatively high C_{17} in both phases. The CPI values in both fractions were around 1.1 for C_{14} to C_{22} n-alkanes. The second pattern is seen in n-alkanes greater than C_{22} (CPI values were also around 1.1 in both fractions). The dominant species in this pattern was also the odd numbered hydrocarbons but in a more pronounced way (see Figure 4, which represents a gas chromatogram showing the

TABLE III

Ratios of various parameters for both, dissolved and particulate n-alkanes in the waters of Shatt al-Arab

Station	C17 / Pristane	C18 / Phytane	Pristane / Phytane	UCM[a]
Dissolved				
I	1.15	1.14	0.92	1.80
II	1.18	1.11	0.88	2.30
III	1.20	1.38	0.92	2.80
IV	1.21	1.24	0.92	3.50
V	1.21	1.23	0.88	3.00
Particulate				
I	1.80	1.42	0.71	0.70
II	0.16	1.50	8.00	0.90
III	1.38	1.25	0.81	1.10
IV	1.54	1.46	0.84	2.10
V	1.80	1.66	0.83	1.60

[a] UMC the unresolved complex mixture or the unresolved envelop.

separation of A: dissolved, and, B: particulate-adsorbed n-alkanes in the water samples of Shatt al-Arab River).

Presence of the even numbered n-alkanes shown on the gas chromatogram may indicate some microbial derived hydrocarbons in the sediments (Grimalt *et al.*, 1985) or a possible anthropogenic input from petroleum. In the literature, both

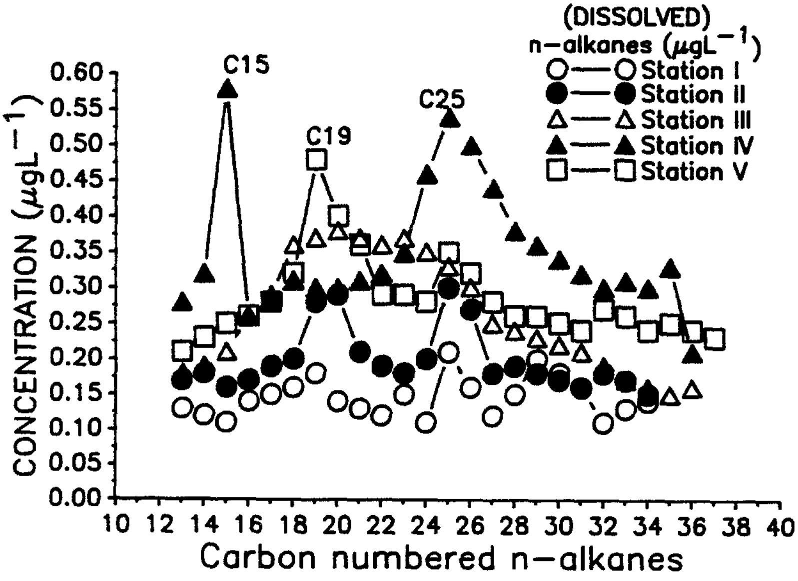

Fig. 2. Concentrations of the dissolved n-alkanes in Shatt al-Arab River.

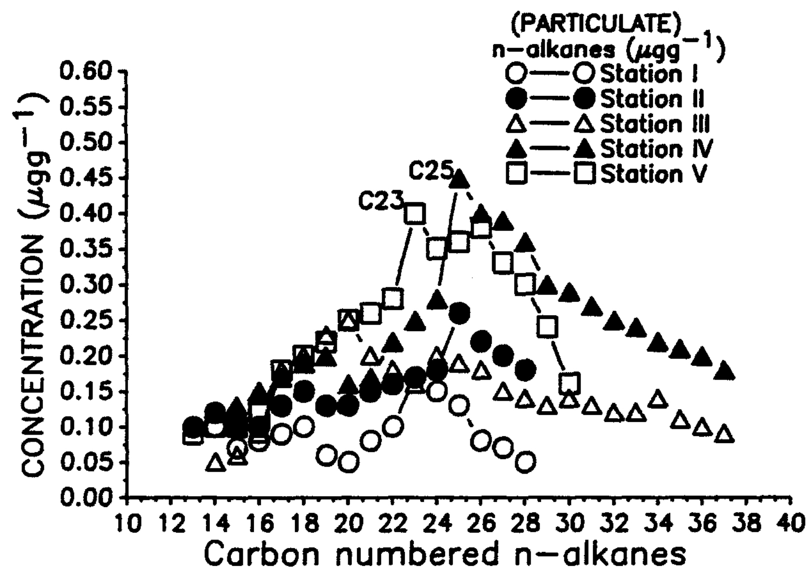

Fig. 3. Concentrations of the particulate-adsorbed n-alkanes in the Shatt al-Arab River.

bacteria and petroleum hydrocarbons are known to have CPI values of 1.0. On the other hand, dominance of the odd numbered n-alkanes was often reported as a result of terrestrial plant inputs (Caldicott and Eglinton, 1973). The CPI values for this type of n-alkanes input is often >4 (Telang *et al.*, 1981).

Al-Saad and Bedair (1989) indicated that a high percentage of hydrocarbons was found associated with high chlorophyll-a concentrations in the waters of Shatt al-Arab. Therefore, dominance of the oddly numbered n-alkanes along with the occurrence of high chlorophyll-a concentrations may provide evidence for aquatic rather than terestrial biogenic origin of the n-alkanes in the waters of Shatt al-

TABLE IV

Analysis of variance comparisons for the total dissolved n-alkanes at stations I–V. Comparisons significant at the 0.05 level are indicated by (*)

Site comparison	Simultaneous lower confidence limit	Difference between means	Simultaneous upper confidence limit	
IV - V	0.0139	0.0660	0.1181	*
IV - III	0.0349	0.0875	0.1401	*
IV - II	0.0975	0.1514	0.2052	*
IV - I	0.1507	0.2045	0.2584	*
V - III	-0.0306	0.0215	0.0736	
V - II	0.0321	0.0854	0.1387	*
V - I	0.0852	0.1385	0.1918	*
III - II	0.0100	0.0639	0.1177	*
III - I	0.0632	0.1170	0.1709	*
II - I	-0.0018	0.0532	0.1082	

TABLE V

Analysis of variance comparisons for the total particulate-adsorbed n-alkanes in stations I–V. Comparisons significant at the 0.05 level are indicated by (*)

Site comparison	Simultaneous lower confidence limit	Difference between means	Simultaneous upper confidence limit	
IV - V	-0.0593	0.0017	0.0626	
IV - II	0.0236	0.0867	0.1498	*
IV - III	0.0377	0.0942	0.1506	*
IV - I	0.0861	0.1492	0.2123	*
V - II	0.0178	0.0850	0.1522	*
V - III	0.0315	0.0925	0.1535	*
V - I	0.0803	0.1475	0.2147	*
II - III	-0.0556	0.0075	0.0706	*
II - I	-0.0066	0.0625	0.1316	
III - I	-0.0081	0.0550	0.1181	

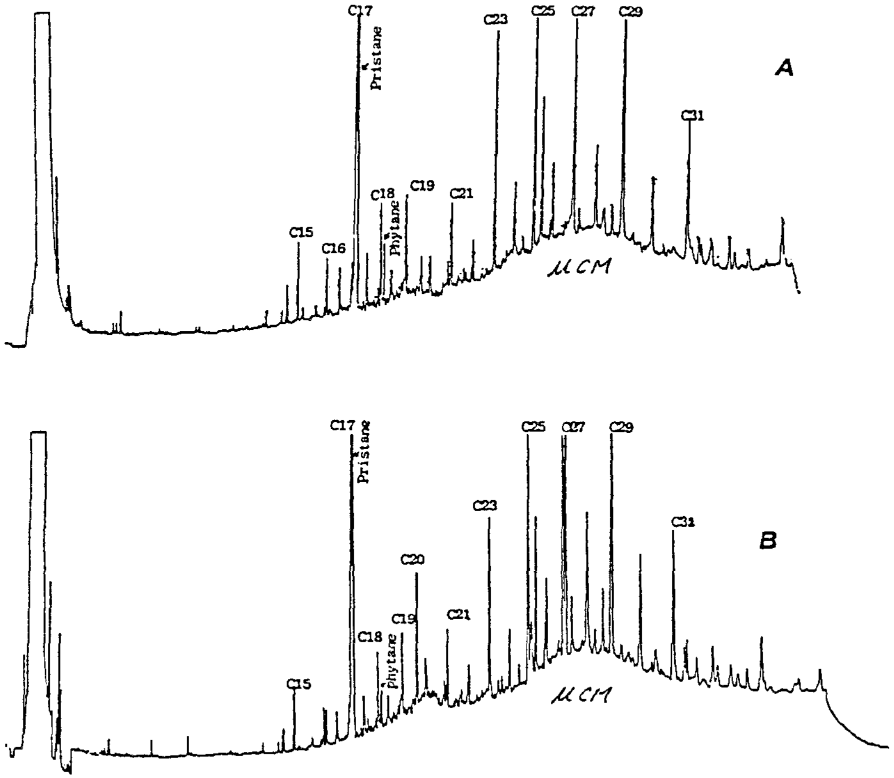

Fig. 4. Capillary gas chromatogram (GC/FID) of the n-alkanes in the water samples of Shatt al-Arab River, Iraq. (A) Dissolved, and (B) particulate-adsorbed fractions.

Arab. Other evidence on the aquatic biogenic origin of the n-alkanes may be derived from diatom population studies. In that, a mixed diatom population was found to contain a range of n-alkanes from C_{15} to C_{31} (Henderson *et al.*, 1972). Diatoms have been found to be the most dominant group of phytoplankton in Shatt al-Arab River (Al-Saboonchi and Al-Saad, 1988). On the CPI scale, the aquatic organisms were found to have 0.4 to 1.5 values (Clark and Blumer, 1967).

Earlier studies (Clark and Blumer, 1967; Youngblood and Blumer, 1973) have documented that odd numbered n-alkanes less than C_{21}, such as C_{15} and C_{17} are commonly encountered in algal species. This may be considered further evidence for the aquatic biogenic origin of the n-alkanes in Shatt al-Arab River. On the other hand, the relatively high C_{17} may indicate that photo-synthetic organisms are the main source of Estuary n-alkanes.

Concerning the distribution of the C_{25} to C_{31} n-alkanes, the prevalence of the odd numbered species within this range provides evidence for the contribution of terresrial plant input of n-alkanes (Eglinton and Hamilton, 1967) and is the first record of this type of hydrocarbon input for Shatt al-Arab waters. The CPI values for this range were found around 1.4. This CPI value, however, is lower than that reported for terrestrial plants (often >4). Examination of the gas chomatogram indicated the presence of C_{27}, C_{29}, and C_{31} n-alkanes, which are the major hydrocarbon constituents in higher plants (Telang *et al.*, 1981).

Some saturated hydrocarbons, such as pristane and phytane, which are natural by-products of weathered petroleum products, were also detected in the water samples (Figure 4). Although pristane is documented to results biogenically from planktonic organisms, phytane is rarely linked to biogenic processes (Venkatesan and Kaplan, 1982). These isoprenoid hydrocarbons are of particular interest because of their stability in the environment. In the present study, the ratios of these isoprenoid alkanes were close to unity and may therefore indicate a balance between biogenic and anthropogenic inputs (Table III).

The relatively broad unresolved complex mixture (UCM), which is shown in Figure 4, is reported as a characteristic of persistent petroleum residues. Earlier studies indicated that UCM is normally associated with either past oil spillage or chronic discharges (Farrington and Meyers, 1975; Farrington *et al.*, 1977; Masspec Analytical, 1983; Shaw *et al.*, 1985). The noticeably broad UCM along with the well known activities that can be related to the production, use, and transportation of petroleum products around the city of Basrah have made this source most likely.

On the other hand, the relatively high concentrations of n-alkanes estimated in the downstream stations IV and V (Figure 1, Tables I and II) suggest substantial petroleum input into the Shatt al-Arab River system. This fact is seen clearly in the comparison of station IV located near the center of the city of Basrah (the highest hydrocarbon concentrations) with station I. The latter station showed the lowest total n-alkane concentrations; since it is quite far from the harbor activities, active water transportation, and relatively heavy industrial and domestic discharges around the city of Basrah (Tables I and II).

In general, stations I to V showed a pattern of 'North to South' (upstream to downstream) increase in the total concentration of n-alkanes; i.e., the total hydrocarbon concentration is three fold higher in station IV than that in station I. Station V, located farther South and away from the direct anthropogenic inputs near the city of Basrah, had less total concentration than station IV. Factors of purification within the Estuary system, such as sinking, mixing, flashing, weathering, and bacterial degradation probably contribute to the reduced hydrocarbon levels in that lower part of the estuary.

The 'North to South' increase in the total concentration of n-alkanes pattern shown in this study is in complete agreement with that of the petroleum hydrocarbon concentrations determined in the sediments of Shatt al-Arab (DouAbul *et al.*, 1984). These n-alkanes both in waters and in sediments of the Estuary may represent a strong evidence of the oil pollution in the Estuary.

In a summary, the major points in this study are as following: 1. Normal alkanes in the waters of Shatt al-Arab may have originated from various aquatic biogenic sources, such as diatoms, algae, and bacteria; 2. Anthropogenic petroleum pollution is pronounced within the vicinity of the city of Basrah, and may represent another source of the n-alkanes in Shatt al-Arab system; and, 3. Information presented in this study can be considered as base-line data concerning the presence, origin, and levels of n-alkane compounds in the waters of Shatt al-Arab River in Iraq, during the year of 1985.

Acknowledgments

The authors must thank the University of Basrah, Marine Science Center, Iraq, for providing funds and analytical facilities. The guidance and supervision of Dr. A. Z. DouAbul, help and sympathy of our families, laboratory assistants, and others are greatly appreciated. Also, thanks are due to Duke University Marine Laboratory, Beaufort, North Carolina, U.S.A. for use of computer and statistical analysis software. We are indebted to the constructive suggestions received from an anonymous reviewer during the development of this paper, and to Drs. D. C. Grant and R. B. Forward for revising the draft.

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