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A STUDY OF ALIPHATIC HYDROCARBONS LEVELS OF SOME WATERS AND SEDIMENTS AT AL-GABAL AL-AKHDER COAST REGIONS

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ABSTRACT

Determinate the concentration of individual aliphatic hydrocarbons in water and sediment samples collected from different locations which distributed on Al-Gabal Al-Akhdar coast. The total aliphatic hydrocarbons TALHs in the water samples were fluctuated between (8.680-88.885 μg/L). While in the sediment were ranged between (26.313-1069.183 ng/g). High levels of carbonate were recorded in all sediment samples. The total carbon contents were ranged between (1.15-2.6%). **T**he special TALHs compound ratios Pr/Ph the detection of these two components is often used as good indicators of petroleum contamination while a CPI approaching unity indicates great inputs from aquatic organisms the ratio of (n-C_{17}/pr) and (n-C_{18}/ph) indicating contributions from biogenic origins and contribution from petrogenic sources.

Key words: Aliphatic hydracarbon, Al-Gabal Al-Akhder coast.

INTRODUCTION

Mediterranean sea appears to suffer from high anthropogenic pressure due to inputs from; industrial, sewage effluents, storm water drains, shipping activities, spillage, rivers, atmospheric-fallout, coastal activities and natural oil seeps[1]. The annual inputs of petroleum hydrocarbons are about 750 × 103 tons, among which the land-based industrial inputs amount are about 221 × 103 tons per year[2]. Thus, the distribution of hydrocarbons in the environment can vary greatly from one area to another. Clark[3] stated that polycyclic aromatic hydrocarbons (PAHs) and aliphatic contamination in sediments originate mainly from the following sources:

(a) accidental spills; (b) partial combustion of fuels (pyrolytic combustion of fossil fuels such as in vehicles, heating and power plants, industrial processes and/or open burning);

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(c) forest and grass fires (d) biosynthesis by marine or terrigenous organisms (land plants, animals, bacteria, macroalgae and microalgae, bacterial and chemical degradation of naturally occurring lipids produced hydrocarbons such as phytanes, hopenes and sterenes); (e) early digenetic transformation of non hydrocarbon natural products to hydrocarbons.

Hydrocarbon composition can be significantly changed due to selective dissolution, chemical reactions, evaporation, photo-oxidation and biodegradation. Simple aromatics and short chain alkanes are rapidly lost, but higher molecules such as hopanes and steranes are little affected and can be particularly useful in source investigations[4].

PAHs (poly aromatic hydrocarbons) are one of the more significant classes of organic chemicals that in the recent years have given rise to a growing concern regarding harmful effects to man and other living organisms.

EXPERIMENTAL

Materials and methods

Sampling

Surface water samples were collected using a Nisken bottle from 8 locations, distributed along Al-Gabal Al-Akhder (LIBYA) during summer 2011 (Fig. 1). A total of 4 surface sediment were collected from the sites. The locations were selected, taking into consideration the expected polluted area due to industrial and human activities. Sediments were collected utilizing a stainless-steel grad from which the top 3 cm were scooped into pre-cleaned wide-mouth glass bottles, frozen, and transported to the laboratory.

Water samples were extracted on board, stored at -4ºC, and transported on the laboratory for aliphatic and PAHs analysis using well-established techniques[5]. Seawater samples were extracted three times with 60 mL of dichloromethane in a separating funnel. Sample extracts were combined and concentrated by rotary evaporation to 5 mL. Finally, samples were concentrated under a gentle stream of pure nitrogen to a final volume of 1 mL.

Sediment analysis

Organic carbon was determined using acid/dichromate titration method as reported[6]. About 0.2-0.5 g was dried and placed in a 500 mL Erlenmeyer flask. Exactly 10 mL of 1 N $K_2Cr_2O_7$ solution was added to the sediment and the two were mixed by swirling the flask. 20 mL of concentration H_2SO_4 were added by burette and mixed by gentle rotation of the flask for about one minute. After 30 min, the solution was diluted to 200 mL volume with distilled water, and 10 mL (85% H_3PO_4). 0.2 g NaF and 15 drops of diphenylamine indicator were added to the sample flask. The solution was back titrated with 0.5 N ferrous ammonium sulfate solution. The result of the analysis were calculated by the following equation:

$$\% \text{ Organic carbon} = 10\ (1\text{-}T/S)\ [1.0\ N\ (0.003)\ (100/W)]$$

Where; T = Sample titration, mL ferrous solution, S = Standardization blank titration, mL ferrous solution, 0.003 = meq. Weight of carbon, 1.0 N = Normality of $K_2Cr_2O_7$ in mL and W = Weight of sediment sample in grams.

About 3-4 g of each of sediment sample was taken and weighed in an aluminum dish. Samples were oven-dried at 105°C to a content for each sample. Before chemical treatment, individual samples were removed from the refrigerator and allowed to thaw at room temperature for about 5 h. Each sample was then thoroughly mixed, and 30 g of the sediment was mixed with 90 g of anhydrous sodium sulphate. Duplicates were taken from each sediment sample. The sediment sample was then extracted in a Soxhlet extractor with 250 mL of hexane for 8 h and then re-extracted for 8 h into 250 mL of dichloromethane. The extracts were then concentrated to a few milliliters in a rotary evaporator al low temperature (35ºC), followed by concentration with a nitrogen gas stream down to a volume 1 mL.

Clean-up and fractionation were performed by passing the extract (water or sediment or biota samples) through a silica/alumina column. Silica column was prepared by slurry packing 20 mL (10 g) of silica, followed by 10 mL (10 g) of alumina and finally 1 g of anhydrous sodium sulfate. Elution was performed using 40 mL of hexane (F1: aliphatic fractions), then 40 mL of 90/10: hexane/dichloromethane (F2: aromatic fractions), followed by 20 mL of 50/50 : hexane/dichloromethane (F3: polyaromatic fractions), both of F2 and F3 fractions were combined to give PAHs. Finally, eluted samples were concentrated under a gentle stream of purified nitrogen to about 0.2 mL, prior to injection into GC/FID for PAHs analysis.

All samples were analyzed by a Hewlett Packard 5890 series II GC gas chromatograph equipped with a flame ionization detector (FID). The instrument was operated in split less mode with the injection port maintained at 290°C and the detector maintained at 300°C. Samples were analyzed on a fused silica capillary column HP-1, with 100% dimethyl polysiloxane (30 m length, 0.32 mm i.d., 0.17, m film thickness). The oven temperature was programmed from 60 to 290°C, changing at a rate of 3 C min^{-1} and maintained at 290 C for 25 min. The carrier gas was nitrogen flowing at 1.2 mL min^{-1}.

A stock solution containing the following PAHs was used for quantification: naphthalene, acenaphthylene, accnaphthene, fluorene, phenanthrene, anthracene, fluoranthene, benzo (a) anthracene, chrysene, benzo (b) fluoranthene, benzo (k) fluoranthene, pyrene, benzo (a) pyrene, dibenzo (a,h) anthracene, benzo (ghi) perylene and indeno (1,2,3-cd) pyrene by dilution to create a series of calibration standards of PAHs at 0.1, 0.25, 0.5, 0.75, 1.0, 2.0, 5.0 and 10,….g mL^{-1}. The detection limit was approximately 0.01, …g mL^{-1} for each PAH. For analytical reliability and recovery efficiency of the results, six analysis were conducted on PAH reference materials, HS-5 and 2974 (provided by EIMP-IAEA). The laboratory results showed a recovery efficiency ranging from 92 to 111% with a coefficient of variation (CV) of 8-14% for all studied pollutants (16 PAHs fractions). All solvents were of pesticide grade purchased from Merck, and appropriate blanks (1000-fold concentrates; two for each batch of analysis for both of water, sediment and samples) were analyzed.

Fig. 1: The stations of study

RESULTS AND DISCUSSION

Aliphatic hydrocarbons (ALIP)

Water sample

The concentrations of total aliphatic hydrocarbons (TALIP) and n-alkanes from C_{10}-C_{40} as well as the isoprenoid hydrocarbon individual concentrations are shown in (Table 1). TALIP in the water samples varied from 8.680 to 88.885 µg/L with an average of (31.421 µg/L). The maximum concentration at station (1) and the low concentrations were recorded at station (8).

Table 1 give total concentration of n-alkanes ranging from C_{10}-C_{40}. The concentration below their limits of detection were given a value of zero for the calculation. In the present study the total n-alkanes varied from 7.952 to 81.37 µg/L. The average of total n-alkanes concentration was 28.40 µg/L. Higher concentration occurred at sites (EL-hamama) with value 81.37 µg/L Site (1).

Figs. 2-6 show the individual concentrations (µg/L) of aliphatic hydrocarbons in water samples and the Fig. 7 shows the total aliphatic hydrocarbons in water samples. Table 1 shows that Station 1 was the most contaminated stations with 88.885 µg/L. This clearly reflects flourishing of zooplankton as biogenic inputs of aliphatic hydrocarbons.

Table 1: The concentrations (µg/L) of aliphatic hydrocarbons of water samples

Station Name	Concentration µg/L								Average
	1	**2**	**3**	**4**	**5**	**6**	**7**	**8**	
C12	0.441	0.310	—	0.022	—	0.334	0.279	—	0.277
C13	0.058	0.203	—	0.226	—	—	—	0.083	0.142
C14	0.259	0.282	0.039	0.509	0.016	0.172	0.148	0.319	0.218
C15	0.962	0.140	0.243	0.087	0.030	0.014	—	—	0.246
C16	0.053	0.132	0.026	0.316	0.123	0.889	1.040	0.951	0.441
C17	12.328	13.487	1.254	11.628	1.903	0.826	1.168		6.085
Pristane	1.067	2.702	1.423	1.604	0.880	0.839	0.657	0.655	1.228
C18	3.330	0.822	0.781	1.016	0.468	0.951	0.228	0.045	0.955
Phytane	6.448	1.691	1.038	2.540	0.855	1.232	0.466	0.073	1.793
C19	17.912	5.118	2.101	6.108	1.970	2.731	1.238	0.325	4.688
C20	13.528	3.529	1.778	5.042	1.609	1.992	0.950	0.632	3.632
C21	9.830	1.195	4.059	4.474	3.156	3.957	0.468	2.564	3.713
C22	6.365	7.524	1.854	3.285	0.976	0.444	0.673	1.392	2.814
C23	6.657	4.716	0.913	0.169	0.126	0.173	0.931	0.223	1.739
C24	6.187	4.818	0.761	2.831	0.148	0.052	1.155	0.229	2.023
C40	3.458	3.434	4.023	1.452	1.962	1.017	2.859	1.188	2.424
∑ALIP	88.885	50.103	20.291	41.308	14.223	15.623	12.259	8.680	31.421
∑n-alkanes	81.37	45.71	17.83	37.164	12.488	13.522	11.136	7.952	28.40

Were: 1 and 2 El-hamama sites, 3 and 4 El-Haniea sites, 5 and 6 Susa sites, 7 and 8 Ras ahilal sites

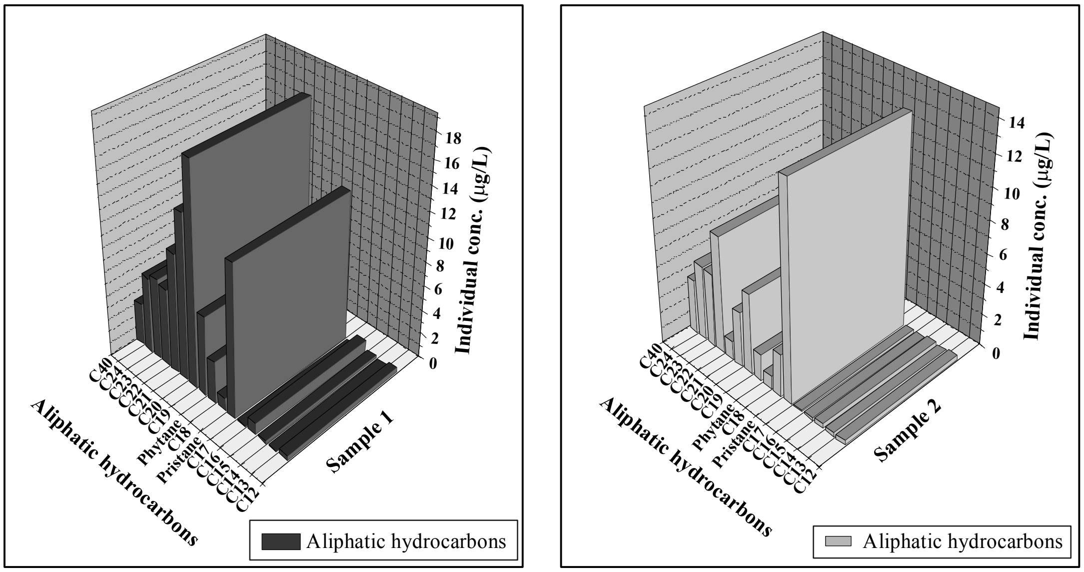

Fig. 2: The individual concentrations (µg/L) of aliphatic hydrocarbons for water samples (1 and 2)

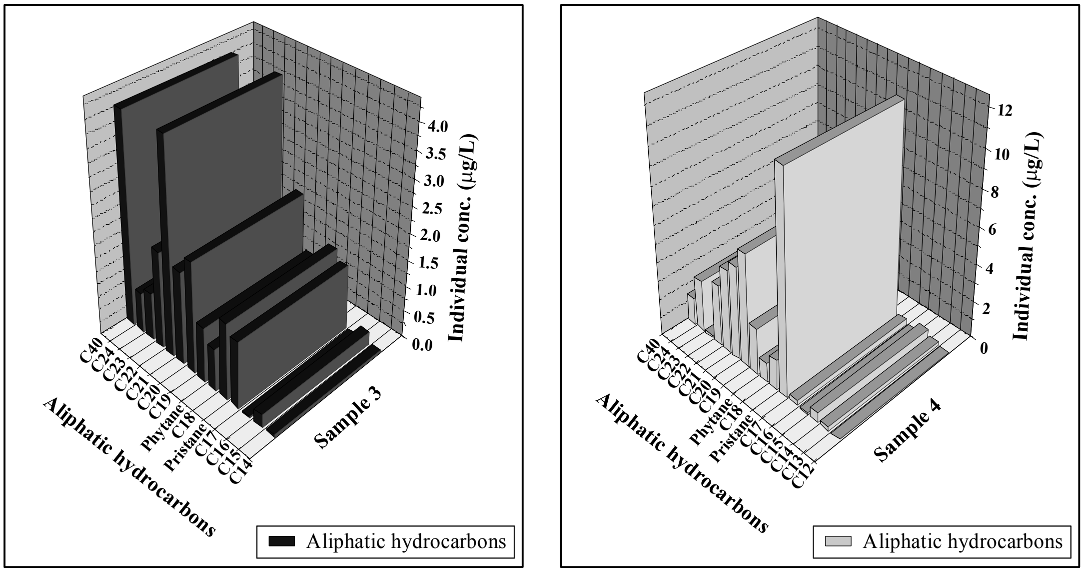

Fig. 3: The individual concentrations (µg/L) of aliphatic hydrocarbons for water samples (3 and 4)

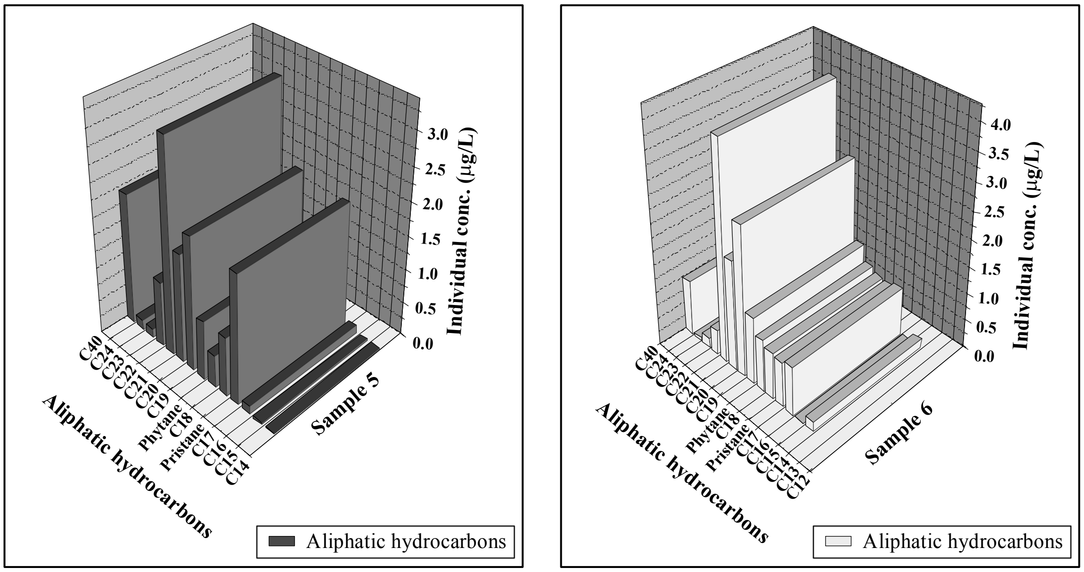

Fig. 4: The individual concentrations (µg/L) of aliphatic hydrocarbons for water samples (5 and 6)

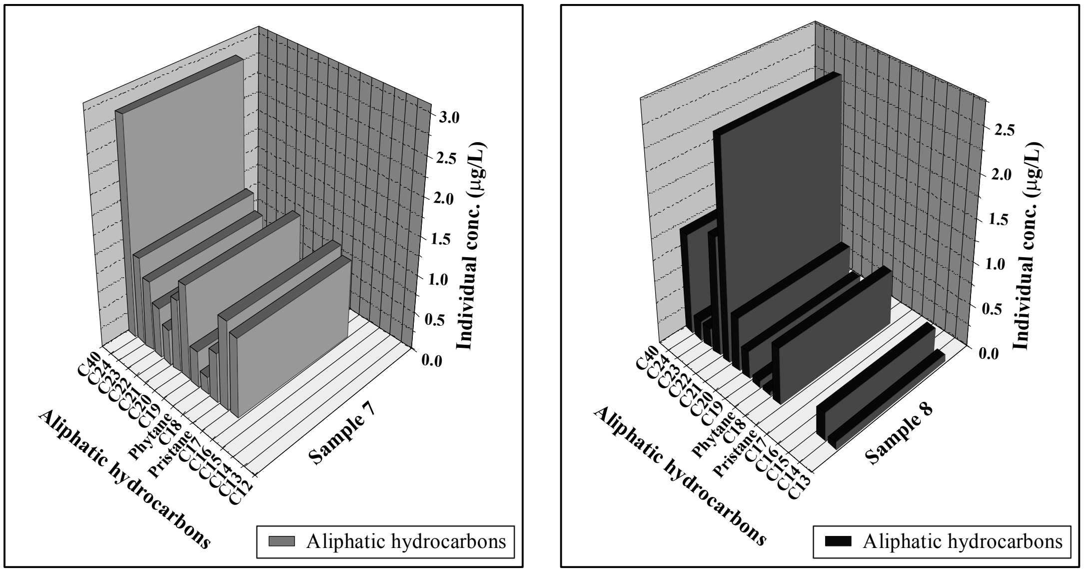

Fig. 5: The individual concentrations (µg/L) of aliphatic hydrocarbons for water samples (7 and 8)

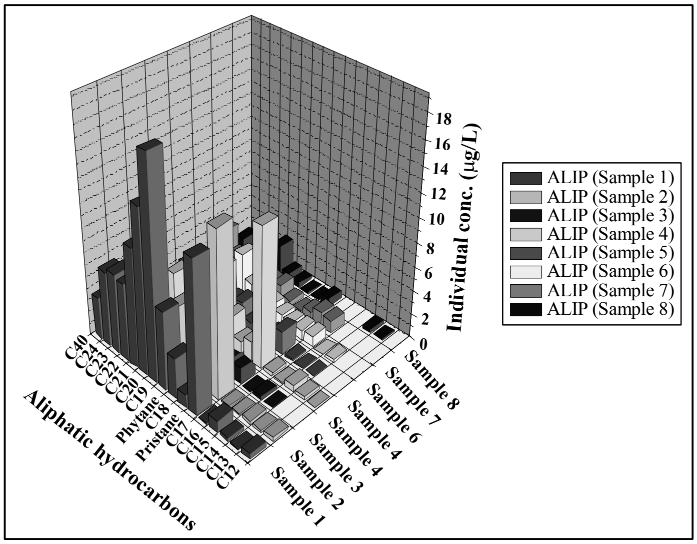

Fig. 6: The individual concentrations (µg/L) of aliphatic hydrocarbons for water samples from (1 to 8)

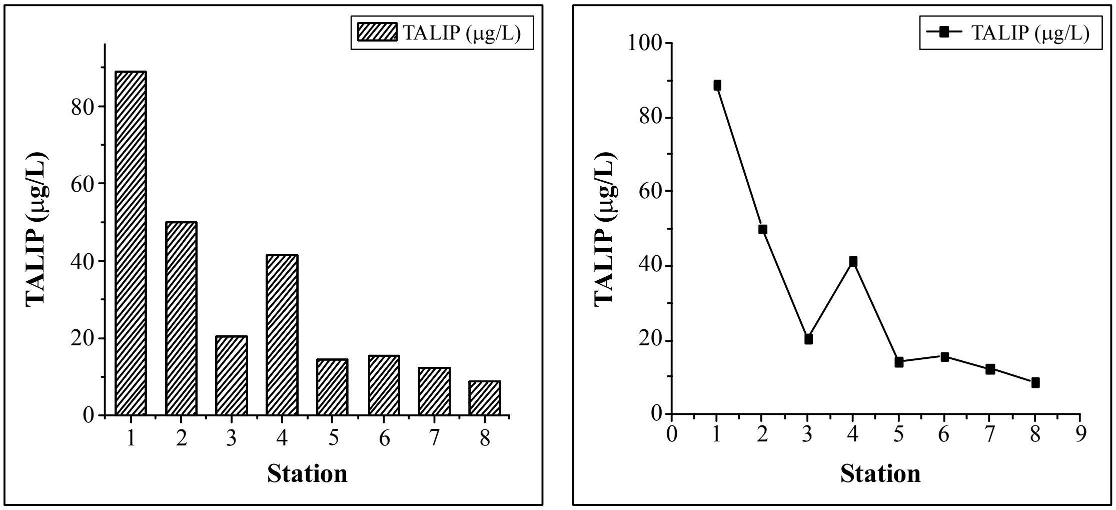

Fig. 7: The TALIP concentrations (µg/L) for water samples and stations

It was reported that, the isoprenoid hydrocarbons, pristane (Pr) (2,6.10,14-telramethylpentadecane) and phytane (Ph) (2,6,10,14-tetramethylhexadecane), are products of geological alteration of phytol and other isoprenoidyl natural products, and are not primary constituents of most terrestrial biota[7,8]. Pr and Ph are prresented in most petroleum oils in a ratio of Pr/Ph < 1, so the detection of these two components is often used as good indicators of petroleum contamination. However, a high concentration of pr alone can be derived from zooplankton[9]. In uncontaminated waters, the ratio Pr/Ph is > 1, typically between 3 and 5[10]. Table 2 showed that pr/ph was < 1 for stations (1,4,5,6) indicating mainly petrogenic hydrocarbons inputs. High pr/ph ratio was >1 for stations (2, 3, 7, 8) reflecting biogenic origin.

Tables 2, indicating mainly biogenic origins of the AL-gable AL-akder coast. The predominance of even carbon numbered as C_{16} and C_{18}. Could be explained as a consequence of microbial activity[11]. The odd/even carbon ratio, or Carbon Predominance Index (CPI) > 1, reflects that the major source of n-alkanes is terrestrial, mostly aquatic plants, while a CPI approaching unity indicates great inputs from aquatic organisms[12]. The present study showed that 24% of stations recorded CPI < 1 which may reflect microbial activity.

However, about 76% of stations recorded have CPI > 1 reflecting terrestrial source from aquatic organisms result from flourishing of zooplankton as biogenic inputs of aliphatic hydrocarbons (Table 2). Also, it is noted that the ratio of (n-C_{17}/pr) in most water samples was > 1 indicating contributions from biogenic origins except sites (3, 6, 8) was < 1 indicating hydrocarbons contribution from petrogenic sources (Tables 2). The ratio of (n-C_{18}/ph) in all stations was < 1 indicating hydrocarbons contribution from petrogenic sources (Table 2).

Table 2: Total aliphatic hydrocarbons concentrations (µg/L) with diagnostic ratios of water samples

Ratio station	TALIP (µg/L)	Total n-alkanes (µg/L)	(C_{17}/pr)	(C_{18}/ph)	CPI	Pr/Ph
1	88.885	81.37	11.5538	0.5164	1.42	0.1654
2	50.103	45.71	4.4914	0.4861	1.19	1.5978
3	20.291	17.83	0.8812	0.7524	0.92	1.3709
4	41.308	37.164	7.2493	0.4	1.56	0.6314
5	14.223	12.488	2.1625	0.5473	1.35	0.9943
6	15.623	13.522	0.9845	0.7719	1.31	0.6810
7	12.259	11.136	1.7777	0.4892	0.51	1.4098
8	8.660	7.952	Zero	0.6143	0.67	8.9726

In general, that meaning indicate that hydrocarbons origin mixed from petrogenic sources and biogenic origins in sites (1, 2, 4, 5, 7) (Table 2).

Sediment samples

Total organic carbon (TOC %)

The compositions and structures of the organic carbon in the sediment are varying due to its origin and geological history in the marine and aquatic environment. Phytoplankton and zooplankton are the most abundant source of the organic material in the sediments[13]. The organic Carbon content of the sediment is a result of contribution of teragenous materials and the decomposition of plants and animals by the action of bacteria[14].

The values of organic Carbon, (Table 3) and (Fig. 8), were 1.6% 1.42, 2.6% and 1.15 at El-Hamama, El- Haniea, Susa and Ras alhilal, respectively.

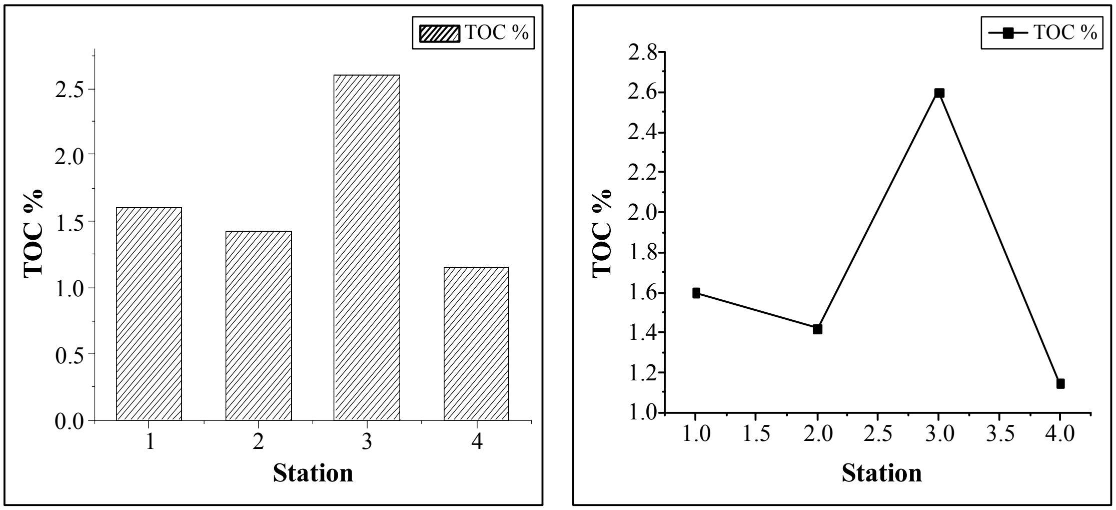

Fig. 8: The concentrations of (TOC %) of the studied sediment

The high TOC% content at Susa area is mainly attributed to discharging of sewage and domestic wastes from out lets and/or susa harbour. Accumulation of organic carbon in sediments is affected by the size of the basin, the wide of the continental platform, the lootom relief, and other morphological features. Physical (temperature, light, oxygen, etc.) and biological characteristics (competition among species, reproduction) are critical factors in the establishment and maintenance of the species inhabiting a sediment.

The quantitative distribution of organic matter content in the sediment depends principally on some factors:

- The allochthonous organic load entering into the waters with sewage and industrial wastes.
- The autochthonous organic production of the sediments.
- The decomposition of organic matter.
- Particle composition of the sediments.

Carbonate: (CO_3^{-2} %)

The total carbonate content is certainly the most important environmental factor and one of the equilibrium systems in the marine environment. It plays an important role in constructing the shells in all species. $CaCO_3$ precipitation is controlled by photosynthesis. Although many calcite rich sediments may have largely allergenic source of carbonate, many other lacustrine carbonate sediments are truly androgenic. Their principal constituents have been precipitated directly from the water column[15].

The concentrations of carbonate in the sediments, Table 3 and Fig. 9, were 66.2, 68.08, 72.8 and 70.55% at El-Hamama, El-Haniea, Susa and Ras alhilal, respectively. The higher value of carbonate is attributed to the aquatic plants and phytoplankton applied to extract CO_2, and thus promote precipitation of carbonate with the increase of pH. A gradual increase in carbonate content from east to west side is recorded. The higher values are probably due to biogenic precipitation of organize by aquatic organisms building their calcareous shells[16], and/or due to the calcium rich water where $CaCO_3$ is precipitated with increase of pH during photosynthesis.

Table 3: The concentrations of total organic carbon and the carbonate content of the studied sediment

Ratio Station	TOC %	CO_3%
1	1.6	66.2
2	1.42	68.08
3	2.6	72.8
4	1.15	70.55

Were: 1 El-hamama sites, 2 El-Haniea sites, 3 Susa sites, 4 Ras ahilal sites

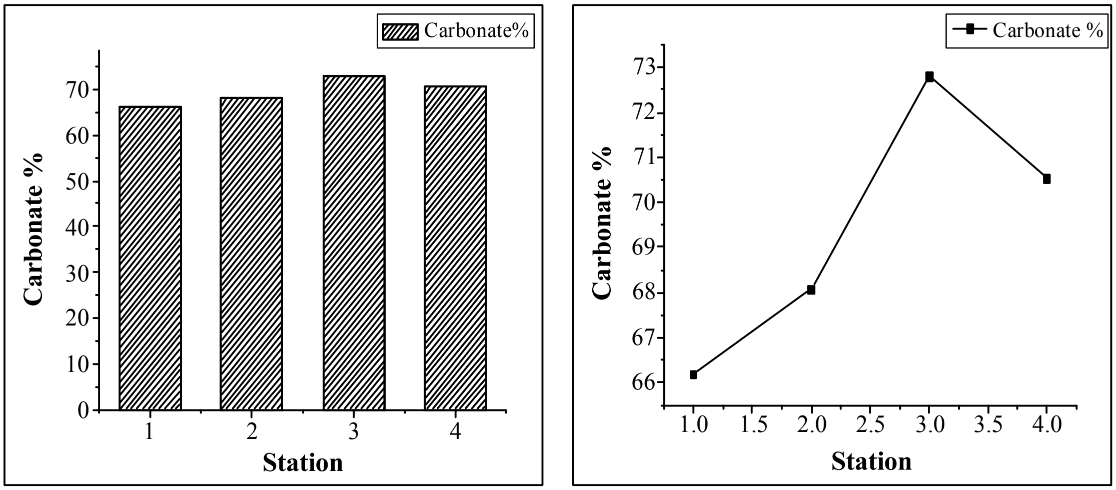

Fig. 9: The concentrations of (carbonate %) of the studied sediment

The distribution of carbonate is a function of the hydrodynamic regime and the magnitude of physical energy which might affect the degree of breakdown of skeletal materials and their subsequent redistribution[17]. Generally, the content of carbonate depends on the type of sediments. However, the sediments of investigated area are mainly calcite and magnesium calcite, to explain the high carbonate content[15].

Aliphatic hydrocarbons of the sediment samples

Table 4 gives the total concentrations of the n-alkanes ranging from C_{12} to C_{40} diagnostic criteria useful for the identification of natural or anthropogenic origins and granulometric parameters for the sediments. The concentrations below their limits of detection were given a value of zero for the calculation. In the present study. The total n-alkanes varied from 26.313 to 1069.183 ng/g (dry weight). Higher concentrations occurred at sites susa port. Lower concentrations were found for samples from sites EL-hamama and Ras alhilal and EL-haniea, Figs. 10-12 show the individual concentrations of aliphatic hydrocarbons in sediment samples.

Table 4: The concentrations (ng/g) of aliphatic hydrocarbons of sediment samples

Station name	Concentration ng/g				Average
	1	2	3	4	
C12	—	—	80.996	3.622	42.309
C13	2.790	—	21.060		11.925

Cont…

Station name	**Concentration ng/g**				**Average**
	1	**2**	**3**	**4**	
C14	14.900	3.990	8.189	1.140	7.055
C15	0.000	—	16.580	—	8.290
C16	46.940	—	3.802	—	25.371
C17	31.799	—	159.700	—	95.750
Pristane	22.300	13.230	81.840	15.260	33.158
C18	6.446	1.965	10.010	1.367	4.947
Phytane	6.647	5.224	40.040	3.088	13.750
C19	7.734	1.100	97.470	1.701	27.001
C20	27.310	1.865	26.830	18.150	18.539
C21	5.323	1.854	28.810	9.605	11.398
C22	18.450	3.537	36.300	5.120	15.852
C23	13.750	5.409	103.630	5.406	32.049
C24	33.710	1.680	20.036	1.233	14.165
C40	163.988	4.912	455.770	—	208.223
∑ALIP	402.087	44.767	1191.063	65.692	425.902
∑n-alkanes	373.14	26.313	1069.183	47.344	378.995

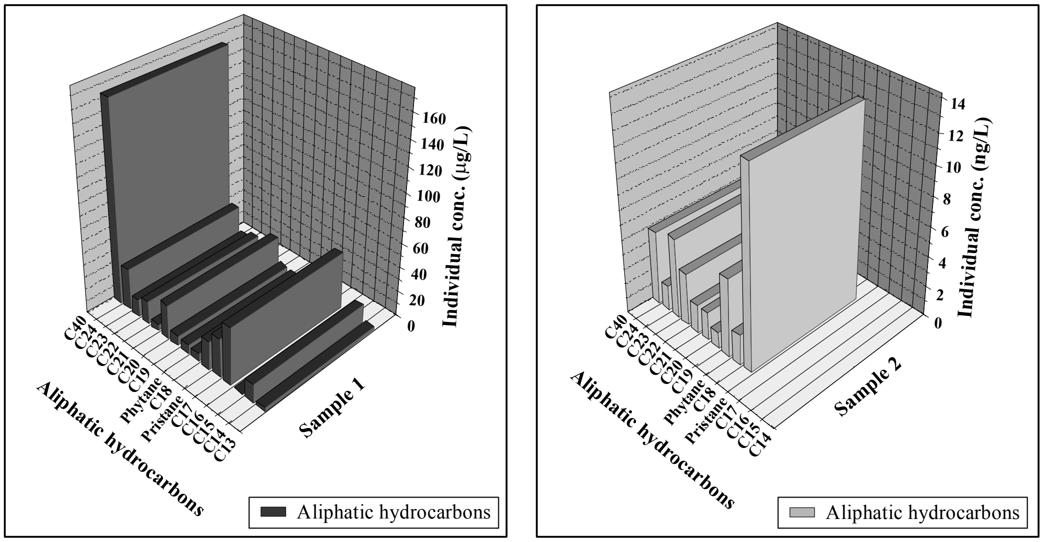

Fig. 10: The individual concentrations (ng/g) of aliphatic hydrocarbons for sediment samples (1 and 2)

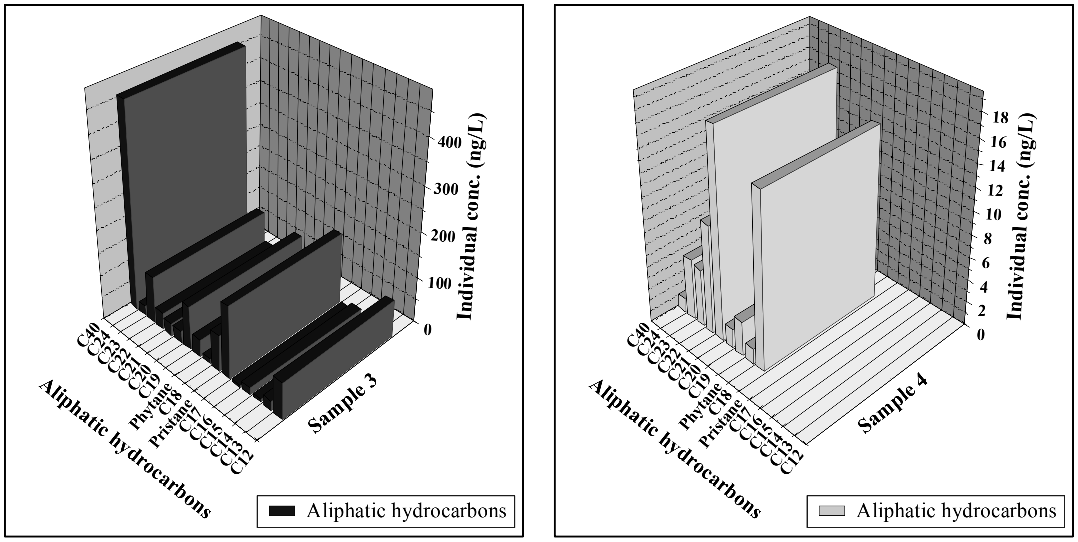

Fig. 11: The individual concentrations (ng/g) of aliphatic hydrocarbons for sediment samples (3 and 4)

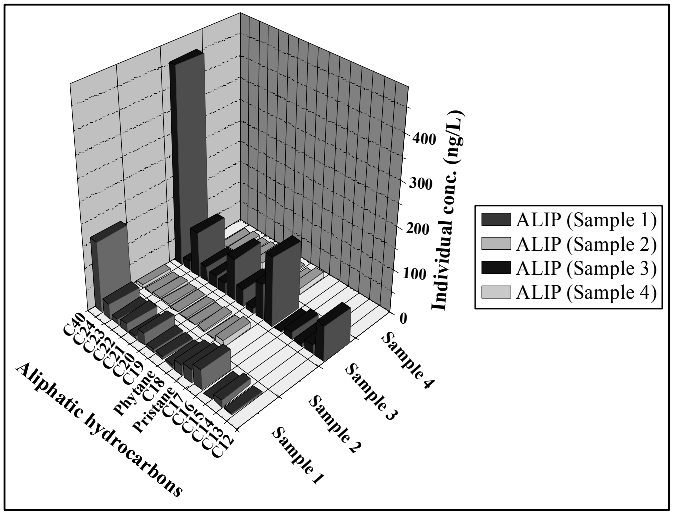

Fig. 12: The individual concentrations (ng/g) of aliphatic hydrocarbons for sediment samples from (1 to 4)

The average total n-alkanes concentrations was 378.995 ng/g (dry weight) (Table 4). The concentration of TALIP in sediments (Table 4) varied from 44.767 to 1191.063 ng/g with an average of 425.902 ng/g (dry wt.), This value was less than the recorded level for clean urban sites in Scotland, UK; with an average value of 3003 ng/g (wet weight)[18]. However it is less than the recorded level for Black sea, which was ranged from 1200 to 24000 ng/g of sediment[19]. The high concentrations was recorded at site (Susa region) (Fig. 13).

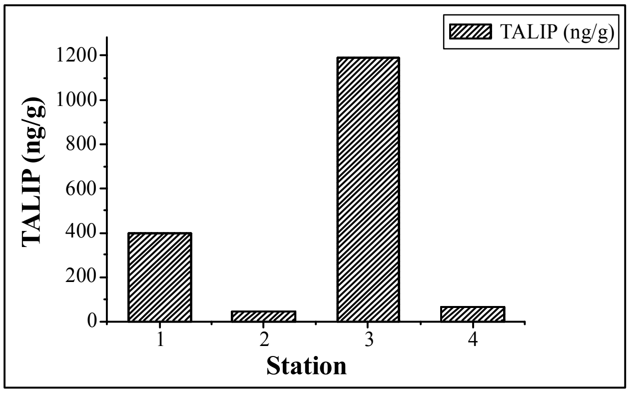

Fig. 13: The relationship between the TALIP concentrations (ng/g) for sediment samples and stations

It was stated that, Pr and Ph are products of geological alteration of phytol and other isoprenoidyl natural products, and are not primary constituents of most terrestrial biota[8]. Pr and Ph are presented in most petroleum oils in a ratio of Pr/Ph < 1, so the detection of these two components is often used as good indicators of petroleum contamination. However, a high concentration of pristane alone can be derived from zooplankton[20]. In uncontaminated sediments, the ratio Pr/Ph is > 1. Typically between 3 and 5[10].

Table 5 showed that Pr/Ph was > 1 for all stations reflecting biogenic origin. The present study showed that all stations recorded CPI < 1 which may reflect microbial activity. The ratio of (n-C_{17}/pr) in sites (El-Hamama, susa port) was > 1 that reflecting to biogenic origins and the ratio of (n-C_{18}/ph) in all sites was < 1 that reflecting to petrogenic sources (Table 5).

Table 5: Total aliphatic hydrocarbons (ng/g) and some diagnostic ratios of sediment samples

Ratio station	TALIP (µg/L)	Total n-alkanes (µg/L)	(C_{17}/pr)	(C_{18}/ph)	CPI	Pr/Ph
1	204.087	373.14	1.4259	0.9697	0.19	3.3554
2	44.767	26.313	Zero	0.3761	0.46	2.5325
3	1191.063	1069.183	1.9513	0.25	0.66	2.0439
4	66.692	47.344	Zero	0.4426	0.54	4.9417

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