

Polycyclic Aromatic Hydrocarbons in Superficial Sediments from Ghar El Melh Lagoon, Tunisia

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Abstract The concentrations of 17 polycyclic aromatic hydrocarbons (PAHs) were determined in 12 superficial sediments collected from the Ghar El Melh lagoon. Sediment samples were extracted by Soxhlet, and analyzed by Gas chromatography with flame ionisation detector (FID). PAH concentrations, ranged from 39.59 to 655.28 ng/g on a dry weight. Total PAH concentrations were not correlated with organic carbon (OC) content or grain size (% <63 μ m). Special PAH compound ratios, such as Ft/Py and Ft/Ft + Py were calculated to evaluate different hydrocarbon origins and showed that PAHs are derived from pyrolytic process.

Keywords Polycyclic aromatic hydrocarbons (PAHs) · Sediments · Ghar El Melh Lagoon · Tunisia · Origin indices

The polycyclic aromatic hydrocarbons (PAHs) are a group of persistent organic pollutants (POPs) that have been, for a long-time, focus of great attention by several scientific communities due to their impact on public health and the environment. PAHs are hydrophobic compounds that tend to adsorb onto suspended material and sediments. Hence, sediments can be considered a pollution reservoir and may be a source of contaminants to aquatic biota. As lipophilic compounds, they can easily cross lipid membranes and

have the potential to bioaccumulate in aquatic organisms. These compounds exhibit toxic, mutagenic, and carcinogenic effects or (anti)-estrogenic activities (Mastrangelo et al. 1996; Szeliga and Dipple 1998; Hirose et al. 2001; Mersch-Sundermann et al. 2001). These pollutants are known to enter aquatic environments through industrial discharges, petroleum spills, combustion of fossil fuels, automobile exhausts, and non-point sources such as urban runoff and atmospheric fall-out etc. (Neff 1979; Hoffman et al. 1984; Latimer and Quinn 1996; Simck et al. 1996).

Lagoons in northern Tunisia represent a major resource for people and aquatic organisms. One of the most important coastal lagoons is Ghar El Melh, situated in Northeastern Tunisia, on the Northwestern side of the Gulf of Tunis. This lagoon is 34 km² in size; it extends over a length of 7 km and a width of 4.5 km and limited by the outer geographic coordinates 37°07'N, 10°08'E and 37°11'N, 10°14'E. The lagoon has provided a variety of important services for human communities since Roman/Phoenician times. Currently about 10,000 people reside within its immediate catchments and much of this area is strongly agricultural. The construction of a new harbour in 1980 on the coast adjacent to the lagoon has made the area an ideal location for the development of fishing and commercial cultivation of marine organisms. The site also has intrinsic value for biodiversity.

A previous studies revealed the presence of residual polycyclic aromatic hydrocarbons in superficial sediment of Tunisia lagoons (Zrafi-Nouira et al. 2008; Trabelsi and Driss 2005; Zaghdien et al. 2007). To our knowledge, there are no studies of PAHs in sediments in Ghar El Melh lagoon, and as such, the aim of this study is to evaluate the levels of 17 PAHs with 16 nominated by the US Environmental Protection Agency (USEPA) as priority pollutants.

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Materials and Methods

A total of 12 surface sediments located by GPS were collected in November 2006. The sediment sample was placed in an aluminum container and frozen on dry ice, then transferred to the laboratory with no exposure to light. In the laboratory samples were defrosted, dried, then the sediments were passed through a stainless steel sieve (0.5 mm mesh) and stored at 4°C until analysis.

Acetone, n-hexane, and dichloromethane were purchased from Fisher (UK). The chlorodric acid, the phosphoric acid and the sulphuric acid were obtained from Biotechnica. Silica gel 60 (100–200 mesh size) was obtained from Merck (Darmstadt, Germany), and activated at 130°C for 20 h. The anhydrous sodium sulfate heated overnight at 150°C and the potassium dichromate were supplied from Fluka. All solvents used for sample processing and analyses were HPLC grade. The 17 PAH analytes employed in this research were: naphthalene (Naph), acenaphthene (Ace), acenaphthylene (Act), fluorene (Fl), phenanthrene (Phe), anthracene (An), fluoranthene (Ft), pyrene (Py), benz[a]anthracene (B[a]an), chrysene (Chy), benzo[b]fluoranthene (B[b]ft), benzo[k]fluoranthene (B[k]ft), benzo[a]pyrene (B[a]py), perylene (Pe), dibenzo[a,h]anthracene (D[a,h]an), benzo[ghi]perylene (B[ghi]pe), and indeno[1,2,3-cd]pyrene (Ind).

The analytical procedure used for PAHs was a modification of the method described by Koh et al. (2006). Briefly, 10 g of sediment samples were Soxhlet extracted for 12 h using 100 mL of hexane and acetone (1:1 v/v). Extracts were treated with acid-activated copper granules to remove sulphur and concentrated to 5 mL using a rotary evaporator. Concentrated extracts were passed through 5 g of activated silica gel packed in a glass column (10 mm i.d.) for fractionation. The extract is eluted with a sequence of 15 mL hexane (fraction 1 which was discarded) and 40 mL hexane: dichloromethane (8:2; fraction 2) which contained the PAHs. The eluate was concentrated to 0.5 mL in a micro-Kuderna-Danish evaporator under a gentle stream of nitrogen. Gas chromatography (GC) analysis was carried out using an Agilent 6890 Series apparatus operated by HP Chemstation software. Samples containing PAHs were injected into splitless mode onto SPB1 fused silica capillary column (30 m length 0.32 mm i.d., 0.25 µm film thickness) and subsequently analyzed by flame ionisation detector (FID). The oven temperature was held at 50°C for 1 min, then programmed at 20°C/min to 150°C and then at 8°C/min to 280°C and held at 280°C for 15 min. The injector was maintained at 280°C and the detector at 300°C. Azote was used as the carrier gas at a flow rate of 1.5 mL/min. PAHs were identified and quantified by comparison with known standards injected under the same conditions.

The whole analytical procedure was validated by analyzing EC-3 sediment reference materials from National Water Research Institute (Canada). The recoveries of studied PAHs in the extract using the same methodology were >80%. All samples were analyzed in triplicates, and the relative standard deviation ($n = 3$) was less than 20%.

The detection limits (LODs) of the method were described as three times signal versus noise value (S/N). For every set of twelve samples a procedural blank and a spiked sample with standards were run to check for the interference and cross-contamination. The detection limits using GC-FID were between 0.06 and 0.50 µg L⁻¹.

The organic carbon (OC) contents were measured by means of the method described by (BSI 1990). Briefly, 2 g dried sediment was treated with 10 mL of 1.000 N potassium dichromate followed by the rapid addition of 20 mL of concentrated H₂SO₄ containing 0.5 g silver sulphate, to precipitate chloride ions. Samples were allowed to cool uniformly to room temperature for 30 min (at 20°C), then the mixture was diluted by 200 mL of double-distilled water, and 10 mL of phosphoric acid was added. Finally, the excess dichromate was back titrated with iron (II) sulphate solution using barium diphenylamine sulphonate as an indicator.

The percentage of finer grain size fractions (% <63 µm) of each sediment sample was determined gravimetrically after wet sieving (Savinov et al. 2000).

Statistical treatment of data was performed using the STATISTICA 6.0 Analysis System, version 5. The Pearson correlation was used to analyze relationships between PAHs and both organic carbon percentage (%) and the pelite.

Results and Discussion

Table 1 shows quantitative PAH concentrations (dry weight basis), obtained at each station for each individual PAH. A wide range of sediment PAH concentrations was observed. Total PAHs in sediments from all stations ranged from 39.59 to 655.28 ng/g dry wt with a mean value of 223.16 ng/g dry wt. Fluoranthene and pyrene, two PAH with four fused aromatic rings were found almost in all stations.

Figure 1 shows the geographic distribution of PAH concentrations. In the whole of the lagoon system, the contents of total PAHs in the sediment vary from a site to another. This result reflects a zonation of the level of contamination.

The stations showing the highest total concentrations of PAHs were S2 and S7 with values of 484.39 and 655.28 ng/g dry wt respectively. The station S2 is located near the fishing harbour. The station S7 located in the west part of the lagoon is characterized by agricultural activities

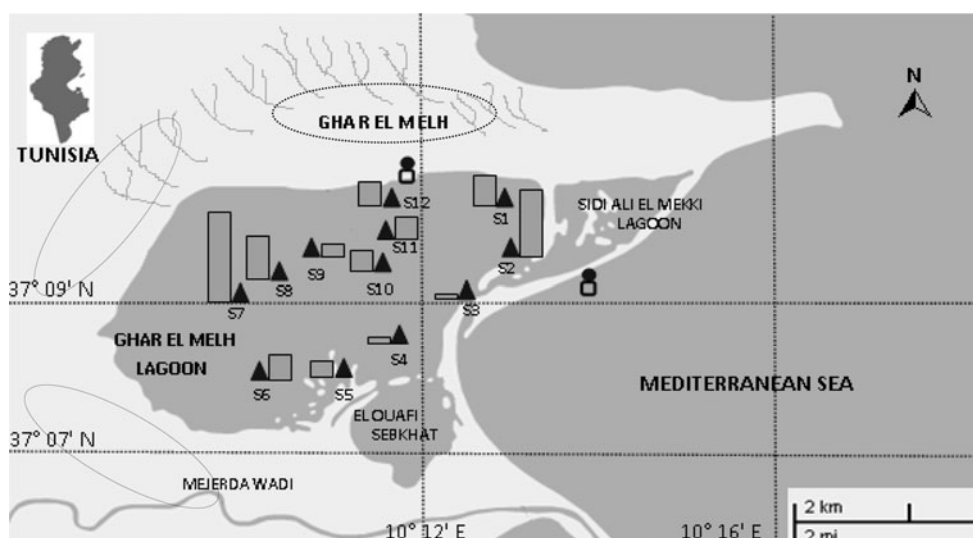
Table 1 Levels of PAHs (ng/g dry wt), percentage of organic carbon (OC), percentage of finer grain size fractions (% <63 μ m) and selected molecular ratios in surface sediments of the Ghar El Melh Lagoon

PAH	Sites											
	S1	S2	S3	S4	S5	S6	S7	S8	S9	S10	S11	S12
Naph	nd	nd	nd	nd	nd	10.58	nd	nd	nd	nd	nd	nd
Ace	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Act	nd	nd	nd	nd	nd	nd	nd	nd	6.16	nd	nd	nd
Fl	nd	28.8	nd	nd	nd	31.20	nd	nd	10.59	nd	nd	nd
Phe	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
An	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Ft	174.25	190.61	nd	nd	111.97	nd	386.88	142.17	204.57	77.73	88.58	118.71
Py	47.22	119.25	39.59	40.6	nd	143.10	126.41	78.23	96.26	nd	40.42	43.45
B(a)an	nd		nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Chy	nd	67.03	nd	nd	nd	nd	67.48	nd	nd	nd	nd	nd
B(b)ft	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
B(k)ft	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
B(a)py	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	21.39	nd
Pe	nd	78.7	nd	nd	nd	nd	74.51	nd	nd	11.52	nd	nd
Ind	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
D(a,h)an	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
B(g,h,i)pe	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
Tot PAHs	221.47	484.39	39.59	40.6	111.97	184.88	655.28	220.4	317.58	89.25	150.39	162.16
%OC	3.10	2.74	2.36	2.01	4.78	3.10	2.35	3.37	3.31	2.74	2.23	3.12
% <63 μ m	98.90	65.40	5.30	98.10	95.40	79.10	87.70	68.60	94.20	91.30	92.50	96.70
Ft/Py	3.69	1.59	nc	nc	nc	nc	3.06	1.81	2.12	nc	2.19	2.76
Ft/Ft + Py	0.78	0.61	nc	nc	nc	nc	0.75	0.64	0.68	nc	0.68	0.73
Pe/tPAHs (%)	nc	16.24	nc	nc	nc	nc	11.37	nc	nc	12.9	nc	nc

nd Not determined (below detectable limit)

nc Not calculated due to concentrations below the detection limits

Fig. 1 Spatial distribution of Σ PAH in surface sediment from Ghar El Melh Lagoon. Filled triangle sampling site, dotted oval populated area, oval agricultural area, shaded box Σ PAH (ng/g dry weight)



and water stagnation due to the weak currents; this explains the high level of PAHs in the sediments of this site.

The stations S1, S8 and S9 are classified as fairly contaminated with total PAH concentrations of 221.47, 220.4 and 317.58 ng/g dry wt respectively. The station S1 is near the old harbour and Ghar El Melh city where there are several local wastewater discharges. The two stations S8 and S9 are located in an area of the lagoon where pollution is affected by agricultural inputs.

In the other stations (S3, S4, S5, S6, S10, S11 and S12) the levels ranged from 39.59 (S3) to 184.88 ng/g dry wt (S6), with a mean value of 111.26 ng/g dry wt.

Relatively high concentrations of PAHs are observed in sediments from Ghar El Melh lagoon, supporting the hypothesis of certain point sources of contamination, the effects of which diminish with distance from the source. Indeed, the lagoon is exceedingly polluted due to human activities within the fishing harbour associated with artisan fishing, such as small boats maintenance and painting. Moreover, pollution could be attributed to the geological environment surrounding the lagoon such as anthropogenic input associated with erosion of upstream agricultural areas and setting out in the lagoon where water currents are generally low.

To assess potential environmental impacts of PAHs in sediments, the concentrations of PAHs can be compared with certain threshold values proposed by the US National Oceanic and Atmospheric Administration, e.g., the effects-range low (ER-L) (which is a sediment quality guideline below which the chemical concentration in the sediments would be expected to rarely have adverse biological effects) and the effects-range medium (ER-M) (a level above which adverse biological effects occur frequently).

The concentrations found for all stations ranged from 39.59 to 655.28 ng g⁻¹ dry wt which are below the ER-L (4,000 ng/g) and the ER-M (45,000 ng/g). This range represents a relatively clean environment.

Table 2 shows comparisons with other international sites. These comparisons demonstrate that levels of PAH in surface sediments of Ghar El Melh lagoon were

significantly lower compared to other Mediterranean sediments. The concentrations of total PAH in Ghar El Melh lagoon were generally lower compared to other lagoons such as Venice lagoon (La Rocca et al. 1996). The contamination level was similar to Bizerte lagoon (Trabelsi and Driss 2005) and four orders of magnitude higher than Cretan sea (Gogou et al. 2000). According to Baumard et al. (1998a), PAH levels can be characterized as low, moderate, high and very high when Σ PAH concentrations are 0–100, 100–1,000, 1,000–5,000 and >5,000 ng/g, respectively. On the basis of classification adapted by Baumard et al. (1998a), sediments from the Ghar El Melh lagoon can be considered low to moderately polluted with PAHs.

Three types of PAHs are commonly found in marine sediments: petrogenic, pyrogenic and biogenic PAHs. Petrogenic PAHs originate from unburned products with a smaller number of rings (two to three rings) and are more highly alkylated (Lee et al. 1977). Pyrogenic PAHs are formed in high temperature combustion processes such as industry emission, and have condensed ring structures (more than three rings); they are usually identified as the majority of PAH input from urban storm water runoff (Singh et al. 1993; Kucklick et al. 1997). The biogenic PAHs are retene originated from terrestrial conifer trees and perylene that is diagenetically formed from terrestrial precursors but it is also produced during pyrolytic processes (Baumard et al. 1998b; Guinan et al. 2001).

Several molecular ratios; such as phenanthrene/anthracene (Phe/An); fluoranthene/pyrene (Ft/Py); fluoranthene/fluoranthene + pyrene (Ft/Ft + Py) and benzo[a]anthracene/chrysene (B(a)an/Chy) have been developed for interpreting PAH compositions and inferring the possible sources (Budzinski et al. 1997; Zeng and Vista 1997; Yunker et al. 2002).

A fluoranthene/pyrene ratio of >1 and a fluoranthene/[fluoranthene + pyrene] ratio of >0.5 indicate that contamination by PAHs is due to combustion processes (Budzinski et al. 1997; Yunker et al. 2002). For all locations except stations S3, S4, S5, S6 and S10 (Table 1),

Table 2 Comparison of sediment PAH concentrations (ng/g dry wt) measured in this study with those in other countries

Sites	n	Range	References
Gelmik bay (Turkey)	14	50.8–13,482	Unlü and Alpar (2006)
Western Harbour, Alexandria (Egypt)	20	8–131,150	Mostafa et al. (2003)
Santander Bay (Northern Spain)	16	20–25,800	Viguri et al. (2002)
Sfax–Kerkennah coastal zone(Tunisia)	17	113–10,720	Zaghden et al. (2007)
Genova Punta Vagno, Italy (harbour)	16	5,201–26,247	Bertolotto et al. (2003)
Venice lagoon (Italy)	7	1.3–4,800	La Rocca et al. (1996)
Bizerte lagoon (Tunisia)	16	83.3–447	Trabelsi and Driss (2005)
Cretan Sea (Eastern Mediterranean)	26	14.7–161.5	Gogou et al. (2000)
Ghar El Melh Lagoon (Tunisia)	17	39.59–655.28	Present study

n: number of PAH compounds analysed in each study

where fluoranthene and/or pyrene were not determined, the data indicate that sediments for stations appeared to be contaminated by pyrolytic PAHs. A fluoranthene/[fluoranthene + pyrene] ratio <0.4 is generally characteristic of petrogenic sources (oil, diesel, coal), between 0.4 and 0.5 indicates liquid fossil fuel (crude oil and vehicle) combustion, whereas a ratio over 0.5 is generally found in kerosene, grass, coal and wood combustion samples and creosote (Budzinski et al. 1997; Yunker et al. 2002).

Perylene can be introduced in the aquatic environment by various processes: the incorporation of atmospheric particles (pyrolytic origin), of fossil fuels such as petroleum (petrogenic origin) or by in situ production by degradation of biogenic precursors (diagenetic origin). This PAH was found only in three stations: S2, S7 and S10. The relative perylene concentrations ranged from nd to 78.7 ng/g dry wt (Table 1). The percentage of the concentration of perylene relative to the sum of total PAHs in the sediments ranged from 11.37% to 16.24%. The low relative abundance of perylene in the study area argues for a dominant pyrolytic origin.

A number of factors determine the concentration and distribution of PAHs in marine environments. Due to their high hydrophobicity and thereby their low water solubility, the PAHs have a high affinity to organic particulate matter. These properties cause them to accumulate in the sediments. There is often a positive correlation between the total organic carbon content and levels of hydrophobic contaminants in sediments such a correlation also usually exists between the fractions of pelite (grain size less than 63 μm) and PAH levels in sediments (Budzinski et al. 1997; Bouloubassi et al. 2001).

OC contents ranged from 2.01% to 4.78% in the study area (Table 1). Organic matter contents of sediments appeared especially high ($>3\%$ of dry weight) in some locations (S1, S6, S8, S9 and S12). These stations are generally located in the extension of urban agglomeration. However, the highest concentration (4.78%) was recorded in station S5. This station is adjacent to El Ouafi Sebkhat, whereas OC contents are relatively low in the eastern part of the lagoon.

The pelite (fraction of the sediment having a grain size $<63\ \mu\text{m}$ (silt + clay)) is given in Table 1. The samples from Ghar El Meleh lagoon show large variations in sediments type, from very coarse to very fine grained. Except station S3, all sampled sediment had a predominantly mud-sand composition and after grain-size analysis, the mainly part is composed of particles below 63 μm . The station S3 is located in an area characterised by sandy sediments. This later station is localised near the old communication between this lagoon and the Mediterranean Sea.

Fine grain particles in sediment usually act as effective collectors and carriers of micropollutants from the water column to the sediments and thus elevate concentration of micropollutants in sediment. In Ghar El Meleh lagoon,

55–65% of the sediment by dry weight was represented by fine particles diameter 40 μm or less (SCET 1999). The abundance of fine particles were assumed to be due to anthropogenic input associated with erosion of upstream agricultural areas and setting out in the lagoon where water currents are generally low.

Correlation analysis shows negative correlations between total PAH concentrations/OC contents and the pelite/PAH levels. These results indicate that the observed distribution of PAHs was not governed by sedimentary characteristics such as OC contents and the fraction of pelite (grain size less than 63 μm), but, it might be due to the localised sources inputs (industrial or urban). However, positive and significant usual correlations ($R^2 = 0.70$) were observed between the sediment contents of fine fraction and those of OC contents (Johnsen et al. 1985). Indeed this association between fine matter and the organic particles is especially due to a common phenomenon in Mediterranean lagoons called flocculation.

This survey permitted to assess the degree of spatial contamination by trace PAHs in the superficial sediments of Ghar El Melh lagoon. Relative to other urbanized coastal areas, Ghar El Melh lagoon can be considered low to moderately contaminated. The main source of PAHs appears to be combustion processes.

These data provide insights into the chemical contamination of the lagoon which help to understand its ecosystem functioning and may be useful for its future management. While measured concentrations are below international limits (ER-L and ER-M), there is cause for concern since PAH concentrations in the sediment samples were elevated near sources of effluent. Furthermore, grab sampling collects the upper 3–5 cm of sediment and higher resolution sampling using only the upper 0.5 or 1 cm of most recent sediment could indicate a different pattern of contamination and would meet future investigation.

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