

DETERMINATION OF PERSISTENT ORGANIC POLLUTANTS IN WATER OF NEW DAMIETTA HARBOUR, EGYPT

TAREK OTHMAN SAID^{*1} AND MOHAMED ABDEL FATTAH HAMED²

*National Institute of Oceanography and Fisheries,
Alexandria branch¹ and Suez and Aqaba Gulfs branch², Egypt
E-mail: tareksaideg@yahoo.co.uk*

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ABSTRACT

Persistent organic pollutants (POPs) which include chlorinated pesticides and petroleum hydrocarbons were determined in water samples collected from Damietta Harbour during 2005. Concentration of total pesticides ranged from 0.02 to 0.07 ng/L and from 1.09 to 201.82 ng/L during winter and summer seasons, respectively. The increasing order of total pesticides was: THCHs < TC < total DDTs with concentrations ranged from 0.004-3.20 ng/L, 0.001-3.93 ng/L and 0.002-194.68 ng/L, respectively in the area of study. The average concentrations of total petroleum hydrocarbons were below the admissible environmental levels. They ranged from 2.17-72.89 µg/L and from 5.67-87.56 µg/L during January and July 2005, respectively. However, concentrations of PAHs were below the detection limit in the present study.

1. INTRODUCTION

Damietta Harbour is considered as semi closed water body affected mainly from loading/unloading operations, municipal and agricultural wastes resulting from Damietta governorate. POPs have a wide range of industrial, anthropogenic and agricultural applications. They include pesticides such as DDT (dichlorodiphenyltrichloroethane) and lindane (γ -HCH), in addition to petroleum hydrocarbons which are organic chemicals composed of fused benzene rings formed during incomplete combustion of coal, oil, petrol and wood (Laflamme and Hites, 1988; NRC, 1989).

It is generally agreed that, the pollution in near-shore waters of the Mediterranean Sea has reached a critical level. This is chiefly due to the total absence of control on toxic components (GESAMP, 1993). River runoff (through Rosetta and Damietta branches) has the direct effect of reducing the salinity of the surface layer of the Mediterranean sea, while

tidal currents have a considerable influence on the vertical mixing of shallow water near the coast (Said and Hamed, 2001).

Damietta Harbour is connected with Damietta branch of the River Nile through a connecting canal of 4.5 Km long, 90 m wide and 5 m deep. Untreated domestic wastewater with agricultural and industrial wastes are still released through a number of drainages and outfalls along the coastal area of study. Svobodova *et al.* (1993) stated that organochlorine pesticides act as nerve poisons and are highly toxic to fish (48-hour LC₅₀ <1.0 mg/L). Because of their chemical structure and their persistence, their use is now strictly controlled or banned (Nelson and Eisenreich, 2001). On the other hand, several polycyclic aromatic hydrocarbons (PAHs) are known to be potential human carcinogens these include; benzo[a]anthracene, chrysene, benzo[b]fluoranthene, benzo[k]fluoranthene, benzo[a]pyrene and benzo[ghi]perylene (IARC, 1991). PAHs are well known environmental pollutants at low

concentrations and included in the European Union and US Environmental Protection Agency (EPA) priority pollutant list due to their mutagenic and carcinogenic properties (Nieva-Cano *et al.*, 2001). They are generated by incomplete combustion of organic materials arising in part from natural combustion such as forest fires and volcanic eruptions (Cotham and Bidleman, 1995). The health hazard posed by these compounds has been studied extensively by several authors (Neff, 1985). The acute toxicity of PAHs to marine organisms increase as the molecular weight increases up to 202 (e.g., flouranthene and pyrene).

In order to understand more clearly the behaviour of these pollutants on a global scale and to prepare the future environmental policies, a baseline study is aimed to determine the occurrence of POPs in water of Damietta harbour.

2. MATERIALS AND METHODS

Fourty surface water samples were collected (1 m depth) from Damietta harbour during winter (January, 2005). However, nineteen water samples were collected during summer (July, 2005) using Niskin bottles covering about 40 Km². The studied areas were represented by three sectors: the navigational area; 4 Km², the eastern sector; 18 Km² and the western sector; 18 Km² (Fig. 1).

Water samples were extracted in the field, stored at 4°C and transported to the laboratory for PAHs analysis using well established techniques (UNEP/ IOC/ IAEA, 1991). 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 2 mL.

Total petroleum hydrocarbons were measured in water samples using the UVF Spectrofluorometer (Sequoia-Turner Model 450) at 360 nm excitation and 415 nm emission according to Parsons *et al.* (1985), where the analysis were performed as chrysene unit. A calibration curve was determined by analysing five separate concentrations (0.5, 1, 2, 4 and 6 mg/L) of chrysene using *n*-hexane as the solvent. Clean-up and fractionation was performed prior to gas chromatograph/flame ionization detector and electron capture detector (GC/FID/ECD).

The 1st mL of the extracted volume was passed through the silica column 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 sulphate. Elution was performed using 40 mL of hexane/dichloromethane (90:10) followed by 20 mL of hexane/dichloromethane (50:50) (which combined contain PAHs). Finally, eluted samples were concentrated under a gentle stream of purified nitrogen to about 0.2 mL, prior to be injected into GC/FID for PAHs analysis.

The 2nd mL of the extracted volume was passed through florisil column prepared by slurry packing 20 g of florisil, followed by 10 g of alumina and finally 1 g of anhydrous sodium sulphate. Elution was performed using 50 mL mixture containing 70% hexane and 30% dichloromethane for pesticide fractions. Finally, eluted samples were concentrated under a gentle stream of purified nitrogen to about 0.3 mL, prior to be injected into GC/ECD for pesticides analysis.

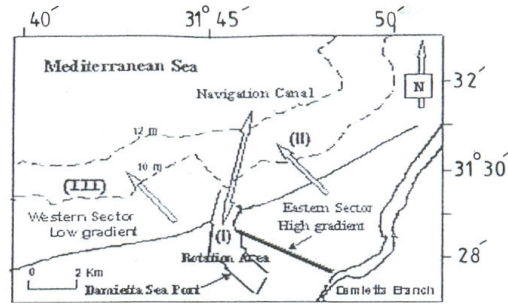


Fig. (1a): Sampling stations collected from Damietta Harbour during January 2005.

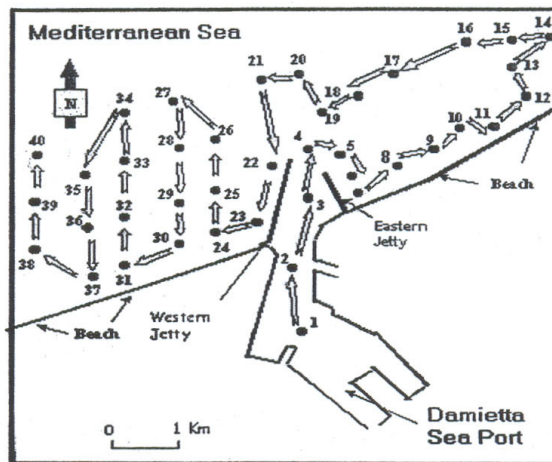


Fig. (1 b): Sampling stations collected from Damietta Harbour during January 2005.

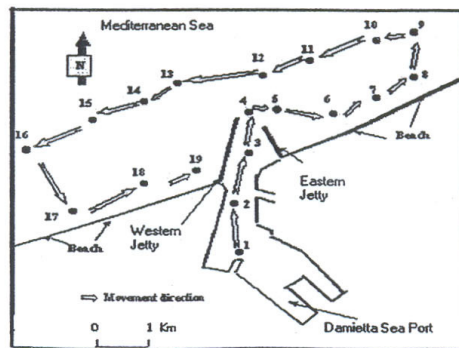


Fig. (1c): Sampling stations collected from Damietta Harbour during July 2005.

All samples were analysed by a Hewlett Packard 5890 series II GC gas chromatograph equipped with a flame ionization detector (FID) and electron capture detector. For hydrocarbons analysis the instrument was operated in splitless mode (3 μ L splitless injection) with the injection port maintained at 290°C and the detector maintained at 300°C. Samples were analysed on a fused silica capillary column HP-1; 100% dimethyl polysiloxane (30 m length, 0.32 mm i.d., 0.17 μ m film thickness). The oven temperature was programmed from 60-290°C, changing at a rate of 3°C/min and maintained at 290°C for 25 min. The carrier gas was nitrogen flowing at 1.2 mL/min. However, HP-5 capillary column (50m x 0.32mm x 0.52 μ m film thickness) with ⁶³Ni-electron capture detector (ECD) was used for pesticides analysis. The oven temperature was programmed from 90°C-140°C at rate of 5°C/min, maintained at 140°C for 1 min, then from 140°C – 250°C at rate of 3 °C /min, maintained at 250°C for 1 min, then from 250°C -300°C at rate of 20°C /min and maintained at 300°C for 1 min. the carrier gas was nitrogen flowing at 1.5 mL/min.

A stock solution containing the following PAHs was used for quantification of hydrocarbons: naphthalene, acenaphthylene, acenaphthene, 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 μ g/mL. The detection limit was approximately 0.01 μ g/mL for each PAH. For analytical reliability and recovery efficiency of the results, 6 analyses were conducted on PAH reference materials, HS-5 and 2974 (provided by EIMP-IAEA). The laboratory results showed recovery efficiency ranged from 92-111% with coefficient of variation (CV) 8-

14% for all studied pollutants (16 PAHs fractions).

Organochlorine pesticides were quantified from individually resolved peak areas with the corresponding peak areas of the external standards (POC mixture provided by IAEA). They includes: α , β and γ -Hexachlorocyclohexanes, Heptachlor, Aldrin, Heptachloroepoxide, Dieldrin, DDT and its isomer DDE. To control the analytical reliability and assure recovery efficiency and accuracy of the results, four analyses were conducted on organochlorine compounds reference material SRM-1974 provided by EIMP-IAEA. The laboratory results showed recovery efficiency ranged from 95-105% for pesticides reference material NO. 2974 with coefficient of variation of 15-20% for all pesticide congeners. All solvents were pesticide grade purchased from Merck and appropriate blanks (1,000 fold concentrates) were analyzed.

3. RESULTS AND DISCUSSION

Tables (1 and 2) show the residual concentration of organochlorine compounds determined in water samples collected from Damietta Harbour. The thorough sight on data of Tables (1, 2) indicate that p,p-DDT (2,2-bis(p-chlorophenyl)-1,1,1-trichloroethane) is the most dominant organochlorine compound during summer season (July, 2005). The maximum concentration was 190.52 ng/L recorded at location 31. The technical product of p,p-DDT contains usually up to 30% of its isomer o,p-DDT (UNEP, 1990), but our results illustrate the presence of a maximum of 2.23 ng/L (1.17%) of o,p-DDT isomer in water sample of location 31. DDT is generally used against a wide variety of agricultural and forest pests and against insect pests including vectors such as mosquito and tse-tse fly. In the environment, DDT can be degraded by solar radiation or metabolised in organisms. Dehydrochlorination of DDT gives its metabolite DDE. This proves the presence of

a maximum of 1.94 ng/L of p,p-DDE recorded also at location 31 (Table 2).

Heptachlor is the common name for 1,4,5,6,7,8,8-heptachloro-3a,4,7,7a-tetrahydro-4,7-methane-1H-indane. It is generally used as insecticide and also occurs technically as chlorodane. In the environment, it is degraded or metabolised and is more commonly found as its epoxide form; Heptachlorepoxyde (UNEP, 1990). This illustrates the presence of a mean value of 0.194 ng/L of heptachlor epoxide compared with a mean concentration of 0.151 ng/L for all locations during summer season in the area of study.

Aldrin is an alicyclic chlorinated hydrocarbon and is rapidly converted to the epoxide form; Dieldrin (GESAM, 1993). The presence of an average of 0.143 ng/L of Aldrin with a mean value of 0.122 ng/L of Dieldrin recorded during summer season declares that there is a renewal source of Aldrin in Damietta water.

HCH (hexachlorocyclohexane) is a fully chlorinated alicyclic compound. The most common isomers are α , β and γ -HCH. The γ isomer known as Lindane is one normally used as an agricultural pesticide. HCH is a reasonably stable compound and only under alkaline condition decomposes to yield trichlorobenzene. It is considered as one of the less persistence organochlorine pesticide. A maximum of 0.468 ng/L of γ -HCH was recorded at the western sector of Damietta harbour (location 31).

The data of Tables (1, 2) declared also that pesticides concentrations were higher in summer than winter reflecting higher agricultural and domestic activities in Damietta harbour during summer season. The order of contamination by pesticides showed a trend decreased from navigational sector > western sector > eastern sector; with a mean value of 73.35, 46.24 and 42.33 ng/L of total pesticides recorded respectively during summer.

Total HCHs were the major pollutant followed by total DDTs followed by total cyclodiens (TC) with an average value of

0.063, 0.022 and 0.014 ng/L respectively during winter (January, 2005); (Fig. 2). However, during summer season total DDTs are the major pollutant followed by total cyclodiens (TC) followed by total HCHs compounds with an average value of 50.29, 0.55 and 0.48 ng/L respectively (Fig. 3). This may clearly reflect the effect of higher water temperature for degradation of HCH compounds during summer more than that of winter.

The discovery of DDT and other chlorinated hydrocarbons in parts of the world's Oceans, shows that the mechanisms of global dispersion of some of these pollutants had to be by a more rapid mode than was possible by Oceanic turbulence and current systems. The trans-Atlantic atmospheric transport of DDT by the northeastern trade wind system was first deduced from observations by Risebrough *et al.* (2000). The presence of chlorinated compounds in the Sargasso Sea atmosphere and surface waters has been investigated more intensively by Bidleman and Olney (1974; 1975). Beside atmospheric deposition organochlorine compounds reach the marine environment through agricultural run-off, rivers and discharge of industrial and municipal wastes. Comparison of atmospheric and river input rates of organochlorine compounds to the world's Oceans was made recently by GESAMP (1993). They showed that pollution of the marine environment by these substances through the atmosphere is more important than river discharge. From another side, the potential effect of pesticidal pollutants carried by the Nile river and its associated canals and drainage systems were investigated by EL Sebae and EL Amayem (1979). Their results indicated the presence of some chlorinated pesticides in concentrations ranging from 340-950 ng/L in Mahmoudieh canal water and from 190-950 ng/L in slaughter waste water. From the above discussion, we can conclude that at open seas atmospheric fallout (rain water) is the major source of pollution by organochlorine compounds.

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However, in our investigated area; Damietta harbour is considered as semi enclosed body affected mainly from municipal and/or agricultural wastes coming mainly from Damietta governerate.

Figure 4 showed that the total petroleum hydrocarbons (THC) concentrations ranged from 2.17-72.89 with an average of 20.76 µg/L and from 5.67-87.56 with an average of 26.0 µg/L during winter and summer respectively in the area of study. These

concentrations are much lower than that given by Dunning and Major (1974); Lucas and Roux (1975); Mazmanidi *et al.* (1976). The THC concentration in seawater which can produce a harmful effect on the aquatic organisms is in the range of 50 µg/L in seawater. In addition, higher concentrations of THC recorded during summer season reflect increasing anthropogenic activities more than winter season in the area of study.

Table (1): Concentration (ng/L) of pesticides in surface water samples collected from Damietta harbour during winter, 2005

Chem. Name Sample No	Concentration, ng/L								
	2	5	10	15	20	25	30	35	40
Alpha-HCH	0.031	0.0012	0.0031	0.0097	0.0017	0.0011	0.0023	0.1472	0.1345
Beta-HCH	0.0068	0.0022	0.0068	0.0032	0.0019	0.0021	0.0012	0.0038	0.0675
Gamma-HCH	0.0166	0.0013	0.0017	0.0011	0.0039	0.0012	0.0011	0.0485	0.064
Total HCHs	0.0544	0.0048	0.0116	0.0139	0.0075	0.0044	0.0046	0.1995	0.266
Heptachlor	0.0027	ND	0.0027	0.0063	0.0097	ND	0.0018	0.0187	0.0277
Aldrin	0.0024	0.002	0.0024	ND	0.0018	ND	0.0014	ND	0.0056
HCP	0.0003	0.0003	0.0028	ND	0.0027	ND	ND	ND	ND
Dieldrin	0.0003	ND	0.0026	ND	0.0035	0.0009	0.0029	0.0028	0.0236
TECHNIQUE	0.0057	0.0023	0.0105	0.0063	0.0177	0.0009	0.0061	0.0215	0.057
<i>p,p'</i> -DDE	0.0013	0.0002	0.0013	ND	0.0084	0.0012	0.0079	0.0056	0.0055
<i>o,p'</i> -DDT	0.0052	0.0083	0.0052	ND	0.0055	0.003	ND	0.0016	0.0387
<i>p,p'</i> -DDT	0.0019	ND	0.0019	0.0022	0.0025	0.0085	0.002	0.0012	0.079
Total DDTs	0.0083	0.0085	0.0083	0.0022	0.0164	0.0127	0.0099	0.0084	0.1232
TP	0.0684	0.0156	0.0304	0.0224	0.0415	0.018	0.0207	0.2294	0.4462

TP: total pesticides, THCHs: total HCHs, TC: total cyclodienes, HCP =Heptachlorepoixide; ND: not detected.

Table 2. Concentration (ng/L) of pesticides in surface water samples collected from Damietta harbour during summer, 2005

Chem. Name	Concentration, ng/L																			
	1	2	3	4	5	7	8	10	23	24	25	26	27	29	30	31	32	33	34	
Sample No.	0.17	0.97	0.2	0.38	0.24	0.09	0.27	0.61	0.21	0.21	0.1	0.29	0.29	1.07	0.21	2.44	0.16	0.15	0.07	
Alpha-HCH	0.04	0.19	0.02	0.04	0.03	0.01	0.02	0.06	0.01	0.02	0.01	0.03	0.01	0.17	0.02	0.3	0.02	0.02	0.01	
Beta-HCH	0.01	0.25	0.01	0.04	0.02	0.01	0.01	0.07	0.01	0.02	0.01	0.01	0.01	0.17	0.04	0.47	0.02	0.04	0.01	
Gamma-HCH	0.23	0.23	0.23	0.46	0.29	0.11	0.29	0.74	0.24	0.25	0.12	0.33	0.31	1.41	0.28	3.2	0.19	0.21	0.08	
Total HCHs	0.13	0.44	0.08	0.2	0.07	0.03	0.12	0.18	0.1	0.13	0.03	0.08	0.11	0.32	0.06	0.65	0.07	0.05	0.02	
Heptachlor	0.07	0.32	0.04	0.16	0.05	0.04	0.05	0.12	0.05	0.07	0.06	0.06	0.04	0.29	0.05	1.13	0.05	0.04	0.01	
Aldrin	0.17	0.49	0.04	0.22	0.05	0.06	0.04	0.27	0.1	0.04	0.08	0.07	0.06	0.36	0.06	1.36	0.05	0.15	0.03	
HCP	0.04	0.23	0.03	0.11	0.03	0.03	0.02	0.16	0.03	0.03	0.04	0.03	0.02	0.56	0.08	0.8	0.03	0.04	0.02	
Dieldrin	0.42	0.42	0.2	0.69	0.2	0.15	0.23	0.73	0.28	0.27	0.21	0.24	0.24	1.52	0.25	3.93	0.2	0.27	0.08	
TC	0.09	0.71	0.07	0.31	0.06	0.05	0.07	0.68	0.12	0.06	0.13	0.12	0.05	0.29	0.21	1.94	0.07	0.26	0.02	
p,p'-DDE	0.16	0.64	0.14	0.31	0.08	0.04	0.16	0.23	0.17	0.15	0.04	0.13	0.14	0.87	0.13	2.23	0.07	0.08	0.01	
o,p'-DDT	69.79	147.4	58.83	14.97	54.5	6.03	78.99	21.83	60.95	91.95	0.59	42.28	79.2	14.33	6.88	190.5	1.45	3.1	0.88	
p,p'-DDT	70.05	148.7	59.03	15.59	54.64	6.13	79.22	22.74	61.24	92.16	0.75	42.52	79.39	15.49	7.22	194.7	1.59	3.43	0.92	
Total DDTs	70.69	149.4	59.46	16.75	55.14	6.39	79.73	24.21	61.76	92.68	1.09	43.1	79.93	18.42	7.74	201.8	1.98	3.92	1.08	
TP																				

TP: total pesticides, THCHs: total HCHs, TC: total cyclodienes, HCP: Heptachloroepoxide, ND: not detected.

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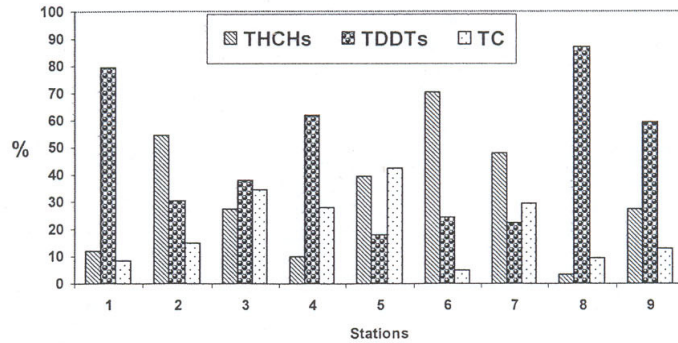


Fig. (2): % of concentrations of different chlorinated pesticides groups recorded in water samples collected from New Damietta harbour during January 2005.

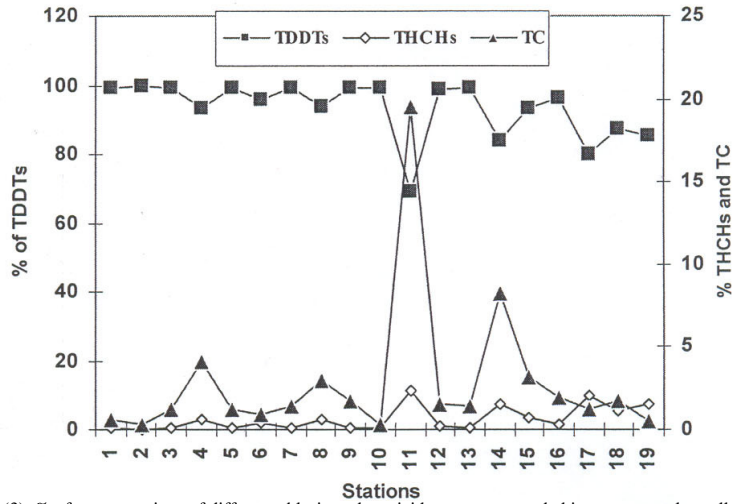


Fig. (3): % of concentrations of different chlorinated pesticides groups recorded in water samples collected from New Damietta harbour during July 2005.

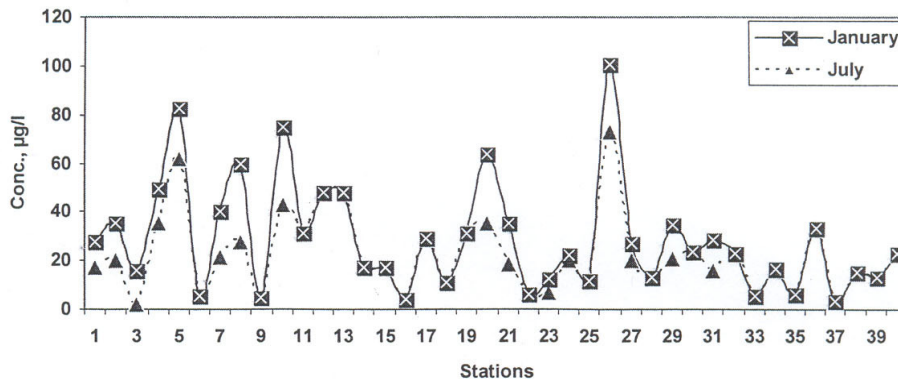


Fig. (4): Distribution of dissolved/dispersed petroleum hydrocarbons concentrations recorded in water samples collected from New Damietta Harbour during January and July 2005 using UVF technique.

The residual of 16 polynuclear aromatic hydrocarbons (naphthalene, acenaphthylene, acenaphthene, fluorene, phenanthrene, anthracene, fluoranthene, benz(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) were investigated in water of Damietta Harbour. No detection for PAHs was noticed in the area during July. This may reflect the effect of weathering degradation of PAHs via high temperature. However, very low concentrations of total PAHs was recorded during January. Benzo(k)fluoranthene and Benzo(a) pyrene were detected at locations 2 (navigation sector) with 2.6 and 1.24 µg/L, respectively. However, Benzo(ghi)perylene was detected at locations 2,10,15 and 35 with concentrations of 3.84, 9.76, 10.78, 6.06 and 10.74 µg/L, respectively during January. These values were much lower than that recorded in the Egyptian Red Sea coast which ranged from 9.73 µg/L to 164.03 µg/L with an average of 40.26 µg/L (Said and Hamed, 2005). The Arabian Gulf ranged from 0.3-455 µg/L with an average of 58.6 µg/L and that recorded in Saudi Arabia coasts ranged from 18.8-412 µg/L with an average of 147.2 µg/L (Awad, 1990).

4. CONCLUSIONS

The present study declared that, some stations have high concentrations more than 60 µg/L (stations: 5, 8, 10, 20 and 26; Fig. 4). However, the total average concentrations of petroleum hydrocarbons were below the

admissible environmental levels in the rest of the area of study. Since organochlorine compounds pose a potential health hazard, maximum permissible levels of toxic substances, recommended for the protection of aquatic biota, has been published. The environmental quality objective set by the European community (UNEP, 1990) is 10 ng/L of p,p-DDT and 25 ng/L of total DDTs. For HCH isomers, the permissible level should be set at 20 ng/L. In comparison to most other organochlorine pesticides, HCH isomers are relatively soluble in water and not therefore so markedly accumulated by marine organisms as DDTs compounds (GESAMP, 1993). Therefore, the present levels of HCHs (0.004-3.20 ng/L), presents no risk to marine organisms in the area of investigation. On the contrary, it is notable that the results of all DDT isomers exceeded the limited acceptable levels markedly during summer season. For Heptachlor, 10-100 ng/L is acceptable for all marine species (UNEP, 1990). This value was not exceeded in Damietta Harbour and ranged from <DL to 1.361 ng/L.

Thus land based activities mainly agricultural and municipal wastes are the major source of pollution by organochlorine compounds occurred markedly during summer season in area.

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