

*Full Length Research Paper*

# Physiochemical analysis and speciation of heavy metals in wastewater and sediment samples from the Suez province in Egypt

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The concentrations of dissolved and particulate forms of Cu, Zn, Pb, Cd, Cr, Ni, Co, Fe and Hg have been determined in wastewater of some factories, fertilizer plants, Attaqa power station, Suez oil processing and El-Nasr petroleum companies and a slaughter house. The concentrations of dissolved metals ranged between 8.02 to 98.90, 31.32 to 1024.50, 14.45 to 289.05, 1.87 to 36.40, 7.78 to 29.98, 3.07 to 45.20, 9.51 to 21.88, 104.91 to 478.63  $\mu\text{g/L}$  and 2.66 to 7.52  $\text{ng/L}$  for Cu, Zn, Pb, Cd, Cr, Ni, Co, Fe and Hg, respectively the particulate phases concentrations were high and ranged between 12.16 to 150.41, 389.41 to 1165.90, 14.86 to 254.75, 2.19 to 6.73, 3.36 to 36.29, 3.75 to 169.82, 12.68 to 29.15, 314.22 to 1335.50  $\mu\text{g/L}$  and 1.12 to 16.76  $\text{ng/L}$  for Cu, Zn, Pb, Cd, Cr, Ni, Co, Fe and Hg, respectively. The concentrations of heavy metals in the bottom sediments ranged from 22.30 to 86.70, 894.60 to 986.20, 18.90 to 41.20, 3.20 to 9.60, 17.30 to 28.50, 10.30 to 17.40, 14.50 to 27.20, 1750.20 to 2102.40 and 0.092 to 0.166  $\mu\text{g/g}$  (dry weight) for Cu, Zn, Pb, Cd, Cr, Ni, Co, Fe and Hg, respectively. The results indicate exchange and transport of heavy metals between the wastewater particulates and bottom sediments.

**Key words:** Distribution, heavy metals, suspended particulates, dissolved phases.

## INTRODUCTION

The pollution prevention act established a new national policy for environmental protection "that pollution should be prevented or reduced at the source whenever feasible". For example, in the past, it was emphasized that treatment of wastewater could be achieved after it was produced. Today, it is a must for the up stream movement in the manufacturing process to prevent the wastes from being generated in the first place. The rapid growth of industrialization in the south Suez City has significantly changed the environment of the region particularly the water quality of the western margins of the Suez Bay. That region receives considerable amount of drainage water from different sources. Such effluents amount to about  $5.4 \times 10^8 \text{ m}^3/\text{year}$  (Suez Governate, 1998) coming from oil refineries, fertilizer plant, power station, textile company, slaughter house and domestic

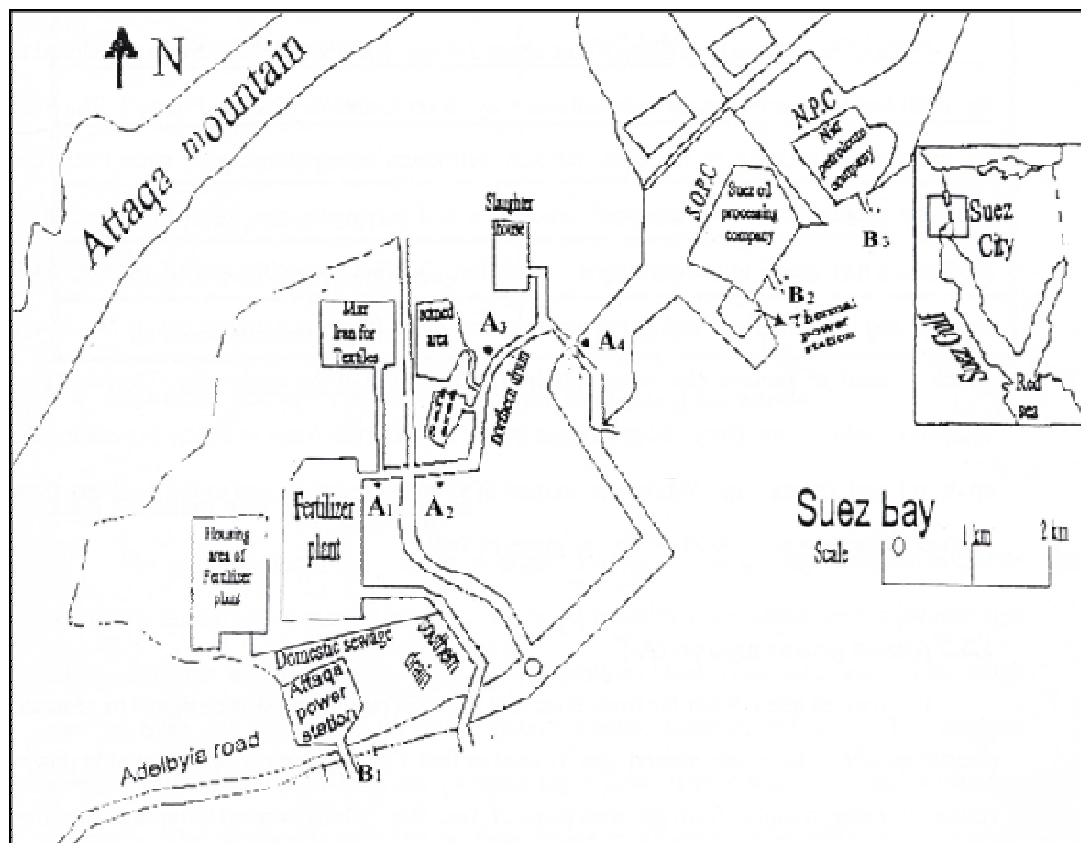
sewage. Such wastes contain varieties of contaminants including heavy metals as well as organic compound, and non-organic species. However, little is known about the distribution of heavy metals in the waste effluents of the area of the present investigation. The presence of Cd in wastes may cause carcinoma, mutation, and reproductive damage (Walks and Georing, 1990). Accumulation of Hg in the living organisms through diffusion in membranes causes damage to vital organs (Lindbergh et al., 1987).

The aim of the present work is: Study the classes of pollutants, which will increase progressively as a result of establishing new factories and harbors in the area under investigation, and Partitioning of some metals including Cu, Zn, Pb, Cd, Cr, Ni, Co, Fe, and Hg between the dissolved and suspended phases.

## MATERIALS AND METHODS

Water samples were collected bimonthly from seven locations in 2009 and sediments were collected from four locations in 2010

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**Figure 1.** Location of samples at the area of investigation. A<sub>1</sub> = Simple drain of fertilizer plant; A<sub>2</sub> = complex drain receiving mixture of the industry effluents of the fertilizer plant and the textiles company; A<sub>3</sub> = complicated drain receiving mixtures of effluents from both A<sub>1</sub> and A<sub>2</sub> as well as domestic sewage from colonies around them; A<sub>4</sub> = very complicated drain receiving mixtures of effluents of A<sub>1</sub>, A<sub>2</sub> and A<sub>3</sub> together with the remains of the slaughterhouse; B<sub>1</sub> = the drainage of Attaga power station; B<sub>2</sub> = the drainage of the Suez oil processing company and B<sub>3</sub> = the drainage of El-Nasr petroleum company.

(Figure 1). Location A<sub>1</sub> represents the simple drain of the fertilizer plant; while A<sub>2</sub> receives mixture of wastewater from the simple drain of the fertilizer plant and the textiles company; A<sub>3</sub> receives complicated effluents from the preceding two locations besides the domestic sewage from the colonies around the drains. Location A<sub>4</sub> is the collecting drain for the three others, in addition to the remnants of the slaughter house. The three other locations designated B<sub>1</sub> for Attaga power station drain, B<sub>2</sub> and B<sub>3</sub> are designated for Suez oil processing company and El-Nasr Petroleum Company drains, respectively. After collection of the samples, they were filtered through 0.45 µm fiber-glass paper (APHA et al., 2001). Residue (solid on filter) was kept for analysis while the filtrate (liquid) was treated with ammonium pyrrolidine dithiocarbamate (APDC) to complex the heavy metals after shaking well, methyl isobutyl ketone (MIBK) was used for extraction (Brooks et al., 1967). The particulates for each sample were digested in 3 ml of concentrated HNO<sub>3</sub> and 5 ml of H<sub>2</sub>O<sub>2</sub> (Tessier et al., 1979). The determinations of heavy metals were carried out using Perkin-Elmer 2380 atomic absorption spectrometer. For mercury measurement, the cold-vapor technique (Imran and Aboul-Enein, 2006) was used. Other parameters such as dissolved oxygen (DO), chemical oxygen demand (COD), biochemical oxygen demand (BOD), total dissolved solids (TDS), suspended matter (SM), total alkalinity (TA) and total hardness (TH) were determined using the standard methods cited in APHA et al. (2001).

## RESULTS AND DISCUSSION

The range and mean of some environmental parameters for the samples of wastewater collected from the drains of the investigated areas are listed in Table 1. The pH was in the alkaline range. A maximum pH of 11.36 was recorded at location A<sub>2</sub> (complex drain). This is attributed to the discharge of the alkaline industrial effluents from the textile company. In the same context, the use of soda lime as softener to remove the temporary hardness from the cooling water participated in increasing alkalinity in different drains. Another factor that may have increased the pollutants was the back washing of softener ponds and zeolite resin which were disposed into location A<sub>1</sub> causing an increase in the total hardness. The concentrations of DO in all effluents were considerably low. The major source of COD in the effluents at locations B<sub>2</sub> and B<sub>3</sub> was the disposal of petroleum hydrocarbons wastes. The levels of COD and BOD at locations B<sub>2</sub> and B<sub>3</sub> were above the permissible limits in law 4/94 (Ministry of Environment, 1994). High-suspended

**Table 1.** Range, mean and standard deviation of some environmental parameters of the investigated drains from Suez City in 2009.

Location	Parameter							
	pH	DO (mgO <sub>2</sub> /L)	COD (mgO <sub>2</sub> /L)	BOD (mgO <sub>2</sub> /L)	TDS (mg/L)	SM (mg/L)	TA (CaCO <sub>3</sub> ) mg/L	TH (CaCO <sub>3</sub> ) Mg/L
A <sub>1</sub>	7.2-10.16 (8.42±0.91)	2.2-3.8 (3.09±1.04)	121.5-145 (128.1±6.7)	95.6-110.5 (97.8±5.7)	1278-6501 (3340.2±24.6)	14.5-91.8 (46.2±9.67)	0.0- 680 (416.85±29.16)	200-1102 (429±36.74)
A <sub>2</sub>	7.85-11.36 (9.56±0.88)	2.0-3.2 (2.91±1.12)	148-178 (156.2±4.14)	125.8-152.6 (136.7±6.11)	1590-4805 (3158.9±29.82)	35.5-130 (81.4±8.56)	270-580 (478.72±21.66)	90-540 (288.5±32.95)
A <sub>3</sub>	6.89-11.02 (8.89±0.82)	1.34-2.54 (1.52±1.2)	180.2-215.5 (196.1±3.06)	148.7-189.5 (171.2±8.2)	1640-5940 (3825.6±21.74)	50.4-205.0 (119.2±7.12)	0.0-540.6 (395.22±25.17)	86.0-540.0 (331.6±24.18)
A <sub>4</sub>	7.40-9.50 (9.11±0.79)	1.0-1.6 (1.21±0.91)	281.5-490.0 (299.8±8.40)	216.6-429.5 (309.2±11.81)	1402.5-4102.1 (2610.6±22.67)	25.1-314.0 (136.7±6.33)	0.05-410.5 (336.6±20.98)	150.0-1004.0 (439.2±38.92)
B <sub>1</sub>	7.20-8.15 (8.14±0.80)	2.30-4.50 (3.40±0.86)	142.5-198.0 (169.6±10.2)	110.2-136.7 (121.30±12.55)	37952-41890 (40748±27.88)	20.5-48.1 (28.90±5.14)	0.0-336.7 (248.5±28.96)	2290.0-3540.0 (2990.6±21.5)
B <sub>2</sub>	8.02-8.25 (8.15±0.75)	1.02-1.95 (1.91±0.90)	295.5-504.2 (418.7±14.2)	237.4-504.6 (328.8±15.78)	40980-42215 (41588±21.68)	56.2-120.5 (86.3±9.18)	20.5-385.6 (267.6±26.11)	2040.0-3990.0 (3308.0±18.89)
B <sub>3</sub>	7.50-8.90 (8.22±0.96)	1.50-3.10 (2.38±1.16)	354.2-470.1 (408.2±11.6)	302.1-421.6 (356.8±16.81)	38810-41990 (40792±25.96)	51.20-90.50 (62.07±5.78)	8.5-402.5 (312.4±18.56)	2725.2-4899.2 (3850.9±30.28)
P. Limits Law 4/94	6-9	-	100	60	2000	60	-	-

DO = dissolved oxygen; COD = chemical oxygen demand; BOD = biological oxygen demand; TDS = total dissolved solids. SM = suspended matter; TA = total alkalinity and TH = total hardness.

solids reported at locations B<sub>2</sub> compared to that at location B<sub>3</sub> was mainly owed from dewaxing and coke distillation units as well as hydrodesulphurization section. Total hardness of effluents of drains A<sub>1</sub> to A<sub>4</sub> indicated that its source was originated from fresh surface water, since artesian wells of this area are of salty origin. The total hardness of effluents of drains B<sub>1</sub> to B<sub>3</sub> reveals its saline origin. In the same context, the concentration of the TDS can be related.

### Partitioning of heavy metals in effluents

#### Northern drains (A<sub>1</sub> to A<sub>4</sub>)

Table 2 shows the mean concentrations of the dissolved and particulate metals in the investigated drains. Generally, it can be safely stated that the decrease in the dissolved heavy metals concentration (A<sub>2</sub> to A<sub>4</sub>) is primarily related to the dilution effect of the disposed effluents.

The high concentration of the particulate iron as reported in effluents of locations A<sub>2</sub> to A<sub>4</sub> was the result of suitable alkalinity and scanty of the dissolved oxygen forming dissolved carbonate and or sulphide of ferrous iron. The decrease in the dissolved zinc in the effluents of A<sub>2</sub> was met with increase in particulates due to formation of zinc carbonate as the stable phase in that environment of pH 8.3 (Peter, 1982). This fact was extended to effluents of A<sub>3</sub> which in the

**Table 2.** Mean concentrations ( $\mu\text{g/L}$ ) of the heavy metals in the different drains from the Suez Bay in 2009.

Metal		Location							T. inflow water	P. limit law4/94
		A <sub>1</sub>	A <sub>2</sub>	A <sub>3</sub>	A <sub>4</sub>	B <sub>1</sub>	B <sub>2</sub>	B <sub>3</sub>		
Cu	D	98.90±6.36	54.2±4.23	18.5±1.98	41.78±2.57	8.02±1.12	11.5±2.01	19.28±1.29	21.00	1500
	P	150.41±8.14	36.18±2.25	79.11±2.15	33.66±1.48	12.16±0.68	23.32±1.1	22.56±1.75		
Zn	D	1024.50±21.14	148.77±7.95	235.6±10.54	132.5±8.11	85.88±6.04	31.32±3.16	280.11±10.68	47.00	5000
	P	951.75±18.20	1156.8±28.44	1096.8±25.33	907.48±24.15	389.41±14.65	698.67±18.72	625.09± 12.36		
Pb	D	41.08±7.56	22.55±2.19	14.86±2.04	72.19±6.1	14.45±1.78	204±12.11	186.88±5.91	25.00	500
	P	53.50±8.12	61.74±6.41	25.13±1.98	85.27±12.02	74.36±9.82	289.05±12.11	254.75± 8.86		
Cd	D	6.56±1.43	3.8±1.02	1.98±0.78	2.08±0.67	1.87±0.6	3.56±18.15	36.4±0.23	0.10	50
	P	3.48±0.98	2.19±0.65	4.66±0.54	3.5±0.44	6.73±0.27	2.22±0.5	4.35± 0.72		
Cr	D	22.42±1.40	15.78±1.34	24.36±0.77	14.77±3.26	20.5±0.95	29.98±1.88	7.78± 0.69	0.20	1000
	P	20.51±1.01	7.64±1.16	29.54±1.22	12.18±0.9	3.36±0.57	8.76±0.91	36.29± 2.11		
Ni	D	4.72±0.43	6.36±0.19	3.5±0.36	3.07±0.16	3.75±0.22	45.2±0.3	28.56±1.48	13.00	100
	P	7.15±1.11	9.08±0.54	12.11±0.37	10.22±0.99	7.63±0.41	152.64±3.98	169.82± 6.87		
Co	D	17.40±1.02	10.57±0.98	21.88±0.68	11.65±1.95	9.51±1.41	18.02±1.84	15.79± 0.6	0.15	2000
	P	26.03±1.21	12.68±1.16	26.36±2.04	13.47±2.41	14.08±2.11	29.15±1.16	21.32±0.76		
Fe	D	412.77±24.5	104.91±16.14	236.4± 18.33	238.36±17.72	240.16±20.56	271.25±19.33	478.63±22.64	440	1500
	P	839.68±36.78	1099.9±40.08	1335.5± 25.16	825.08±19.51	314.22±14.44	321.47±15.38	462.81±18.82		
Hg*	D	2.66±0.87	4.26±0.59	3.89± 0.29	5.78±0.3	7.21±0.14	7.52±0.4	6.33±0.28	0.40	5
	P	2.18±0.66	3.5±0.48	2.94± 0.36	1.12±0.18	11.54±0.25	16.76±0.54	5.14±0.59		

D = Dissolved; P = Particulate, - = ng/L; T = total; P. limit = permissible limit.

**Table 3.** Mean concentrations of dissolved heavy metals ( $\mu\text{g/L}$ ) in the effluents of the drains of Suez oil processing and El-Nasr petroleum companies during the periods 1992 to 1993 and 2009 to 2010.

Metals	Location			
	B <sub>2</sub>		B <sub>3</sub>	
	1992-1993 (Hamed, 1996)	2009-2010 Present study	1992-1993 (Hamed, 1996)	2009-2010 present study
Cu	3.20	11.5±2.01	9.40	19.28±1.29
Zn	3.40	31.32±3.16	11.50	280.11±10.68
Pb	1.18	289.05±12.11	14.70	186.88±5.91
Cd	0.03	3.56±18.15	0.20	36.40±0.23

particulates of zinc carbonate were higher than the dissolved form. Results of cadmium were more or less similar to those of zinc. This is quite reasonable, since both elements are similar in chemical properties. The results of copper reveal that the media of A<sub>2</sub> to A<sub>4</sub> enhanced the formation of particulates of copper compounds particularly basic salts of carbonate and sulphate. However, it is noteworthy to observe the high increase in concentration of the two forms of copper (dissolved and particulate); surely that is related to the discharged effluents from the fertilizer plant. Such effluents contain remains of copper released from the complex  $[\text{Cu}(\text{OAc})_2(\text{NH}_3)_2]$  which is added to remove the harmful effect of carbon monoxide on the production of nitrogen and hydrogen gases. Lead, nickel and cobalt results provide satisfactory correlation. However, lead and nickel particulates were more abundant than the dissolved forms.

Dissolved chromium salts were dominant, since the alkaline medium enhanced the formation of soluble chromate. It is clear that the concentration of mercury in the dissolved form was perceptible over that of the particulate form. This may be due to the use of mercury organic compounds in the irrigation water, as turf fungicides and cotton warm killer.

#### Attaqa power station and the petroleum refineries (B<sub>1</sub>- B<sub>3</sub>)

The characteristics of the drains B<sub>1</sub> to B<sub>3</sub> are quite different from that of the preceding drains (A<sub>1</sub> to A<sub>4</sub>). The effluents supply of drains B<sub>1</sub> to B<sub>3</sub> is primarily of salty source. A general outlook on Table 2 reveals that 80% of the analyzed samples contained high amounts of dissolved and particulate heavy metals compared to the inflow effluents. This is quite reasonable, taking into consideration the heavy wearing processes exerted on the internal ignition machines due to the evolution of sulphur gases, mainly sulphur dioxide and sulphur trioxide since sulphur and its compounds are of the constituents of the crude oil and natural coal in addition to metals such as chromium, vanadium, and nickel, which are usually associated with such natural materials; addition

of tetraethyl lead to improve the performance of gasoline in motor vehicles. Increase in mercury concentration may be related to the burning of natural coal in the power station of Attaqa, where mercury is emitted to the environment (Lindberg et al., 1987). The perceptible concentration of chromium may have originated from the use of its compounds to improve the efficiency of the cooling systems. Table 3 shows a comparison between some dissolved elements in the effluents of the drains B<sub>2</sub> and B<sub>3</sub> of the petroleum refineries. The results reveal a big gap between industrialization activities and environmental protection requirements. It seems that there is no way to treat effluents before disposal into the drains, since it was less than ten years the increase in pollutants reached many folds. However, it should be noted that the reported data of 1992 to 1993 are the available ones (Hamed, 1996).

#### Heavy metals in sediments

Table 4 indicates that the mean concentrations of Cu, Zn and Fe in sediments at location A<sub>1</sub> were greater than those detected in standard precipitate sediments. This may be attributed to the contamination of location A<sub>1</sub> with the industrial effluents discharged from the fertilizer plant. Also, the concentrations of Zn, Pb, Cd, Cr and Fe in the sediments increased from location A<sub>1</sub> to A<sub>2</sub>. Such increase may be related to the high pH of wastewater at location A<sub>2</sub> which enhances the coagulation and precipitation of iron oxy hydroxide to the sediments (Chao and Theobald, 1976). The increase in the contents of Zn, Pb and Cd are the results of the scavenging action (Heam, 1977) of Fe-oxides through adsorption process. Possible removal mechanisms of Cr from wastewater and accumulation of it in sediments was its precipitation as Cr (OH)<sub>3</sub> and /or sorption of Cr on the clay particles. The decrease of the mean content of Cu in the sediments from location A<sub>1</sub> to A<sub>2</sub> may be due to that the decrease in the content of Cu in the wastewater at A<sub>2</sub> (Table 2). The sources of heavy metals in the sediments at location A<sub>3</sub> were the industrial effluents which comes from the complex drain (A<sub>2</sub>) besides the raw domestic sewage discharged from different colonies around that drain

**Table 4.** Mean concentrations of some heavy metals ( $\mu\text{g/g}$ , dry weight) of sediments of the northern drains in 2003.

Metals	Locations				Standard precipitate (Cooper and Harris, 1974)
	A <sub>1</sub>	A <sub>2</sub>	A <sub>3</sub>	A <sub>4</sub>	
Cu	86.7	22.30	61.30	74.80	5.00
Zn	894.6	902.90	986.20	950.20	56.00
Pb	18.90	33.60	34.90	41.20	33.00
Cd	3.20	4.90	7.90	9.60	--
Cr	17.30	23.60	28.50	25.20	38.00
Ni	12.90	10.30	17.40	14.50	17.00
Co	19.10	14.50	27.20	22.40	--
Fe	1912.50	2102.40	1750.20	1781.6	1200.00
Hg	0.09	0.11	0.15	0.17	--

and atmospheric deposition mostly arising from dusts. Therefore, the mean concentrations of Cu, Zn, Pb, Cd, Cr, Ni, Co and Hg were increased in the sediments from location A<sub>2</sub> to A<sub>3</sub>, while the reverse is the state for Fe. This may be attributed to the increase in content of organic matter in sediments at location A<sub>3</sub> that can act as ion exchange surface and can absorb metal ions. The drop in the content of Fe at location A<sub>3</sub> is mainly owing to the precipitation of Fe(OH)<sub>3</sub> at location A<sub>2</sub> as a result of increase in the pH values. Table 4 shows that the mean concentrations of Cu, Pb, Cd, Fe and Hg increased in the sediments from location A<sub>3</sub> to A<sub>4</sub>, while Zn, Cr, Ni and Co decreased. This is most probably due to the increase in the sulphide concentration (2.21 mg/L) in the wastewater at location A<sub>4</sub> leading to formation of FeS after reduction of Fe<sup>3+</sup> to Fe<sup>2+</sup>. Thus scavenging of Cu, Pb and Cd up on the formation of iron sulphide should be higher taking into consideration the solubility product of this metal sulphide (Hogfeldt, 1982). The decrease in the contents of Zn, Cr, Ni and Co in the sediments at location A<sub>4</sub> is attributed to the decrease in the pH as well as, the addition of reductants like sulphides and organic matter to the sediments which leads to a change in the redox potential of the Fe(OH)<sub>3</sub>, and may cause the release of the metals adsorbed or associated with Fe(OH)<sub>3</sub> back to the aqueous phase. From a statistical point of view there was positive correlation of Fe and both Zn and Cu ( $r = 0.91$  and  $0.99$ , respectively,  $P < 0.05$ ) indicating intimate association between the three elements. There is weak correlation between Fe and Pb ( $r = 0.074$ ), most probably because its concentration was below the solubility product of its compounds in the sediments.

## Conclusion

It can be concluded that the concentration of Cu, Zn, Pb, Cd, Cr, Ni, Co, Fe and Hg in the dissolved phases was low as compared to the particulate phases. In the same context, the mixing of industrial effluents with domestic

sewage led to increase in the particulate phases. A clear relation was established between the concentration of Fe and that of Zn and Cu.

## RECOMMENDATIONS

Some proposals for wastewater treatment should be drawn from the study with regard to Aeration of hydrocarbon effluents, which discharges into the drains B<sub>2</sub> and B<sub>3</sub> to reduce the hydrocarbon contents; thus, the treatment of the acidic effluents of the fertilizer plant and alkaline wastes of the textile company in combined ponds to produce salts of economic value is therefore recommended. The treated effluents may be reused in cultivation of wooden trees in the area close to the industrial region. The domestic sewage of different colonies must connect to the network of the sewage treatment plant of the Suez City through pipelines instead of mixing it with the industrial effluents. The most effective treatment procedure for Cd and Pb removal is the use of ferric sulphate coagulant at pH > 8.0 plus excess of lime added to the effluents.

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