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## **Assessment of hydrocarbons and trace metals pollution in water and sediments of the Fertilizer Plant wastes in Khor Al-Zubair, Iraq**

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**Abstract** - The levels of petroleum hydrocarbons and trace elements were determined in water and sediment samples from the Fertilizer Plant in Khor Al-Zubair, southern Iraq, from three selected locations; ammonia unit, urea unit and water treatment plant. The mean concentrations of Total Petroleum Hydrocarbons (TPHs) in the water were 81.07 µg/l at ammonia unit, 153.69 µg/l at urea unit and 8.89 µg/l at water treatment plant. The mean values of TPHs in the sediment were 129.46µg/g at ammonia unit, 406.68 µg/g at urea unit and 48.97µg/g at water treatment plant. The range of trace metals concentrations (mg/l) for water samples were 480.13-620.12 for Fe, 280.23-400.26 for Mn, 3.28-10.23 for Cu, 18.23-31.28 for Zn, 3.26-9.23 for Pb, 0.23-1.05 for Cd and 6.25-10.21 for Co. The range of these measured metals concentrations in µg/g for sediment samples were 632.18-980.45 for Fe, 280.23-400.26 for Mn, 9.26-16.53 for Cu, 30.12-51.24 for Zn, 23.41-30.23 for Pb, 0.62-1.52 for Cd and 20.21-32.42 for Co. The results have been compared to other international and local values. The geochemical index values for studied metals excluded Fe and Cd in the sediments of the three stations exhibited class 2 and hence are moderately polluted. Station (2) was the highest in the geochemical index. All the sampling locations have an enrichment factors values greater than 5 except location (1) for Cd and location (3) for Zn. The sediments from the studied locations fall under the category of low for Fe, moderate for Zn and Cd except location (2) which showed a contamination factor (CF) of 4, marginally falls under considerable degree of contamination and considerable to very high contamination for the rest metals. All sediment samples with pollution index greater than 1 showed pollution caused by industrial processes of Fertilizer Plant.

**Key words:** Hydrocarbons, trace metals, sediments, water, Khor Al-Zubair.

### **Introduction**

The investigation in petroleum hydrocarbons and trace metals pollution is very important in Basrah city due to petroleum production and other activities in this region. The industrial effluents (liquids or solids) that are discharged to the surface or sea waters are the main reason behind water pollution.

These pollutants can be classified into three main groups: sewage water, industrial effluents and agricultural pollutants (Hassan *et al.*, 2011). The control of such pollution problems in the aquatic environment is very difficult because of the large number of input sources and their geographic dispersions. Contrary to popular views, evidence is accumulating to buttress the fact that petroleum hydrocarbon mixes with water and penetrates to the underlying sediments (Clinton *et al.*, 2009). The Sediments afford an integrated picture of the hydrophobic pollutant arriving at the river column. Sediments constitute a good record, and were be about the today's' state and the history of contaminated area, they can provide an integrated picture about the events that occurred in the water column. (Al-Saad *et al.*, 2009). Among the most dangerous pollutants are usually present in mercury (Hg), cadmium (Cd), silver (Ag), nickel (Ni) and lead (Pb) .. etc. (Heba *et al.*, 2007).

Pollution of the aquatic environment by inorganic chemicals has been considered a major threat to the aquatic organisms including fishes. The agricultural drainage water containing pesticides, fertilizers and effluents of industrial activities and runoffs in addition to sewage effluents supply the water bodies and sediment with huge quantities of inorganic anions and heavy metals (ECDG, 2002).

The purpose of this study was to estimate the levels of petroleum hydrocarbons and trace metal elements in water and sediments samples from Khor Al-Zubair of southern Iraq. The trace metals Fe, Mn, Cu, Zn, Pb, Cd and Co were included.

## Materials and Methods

The water and sediment samples were collected during 2000 from three different stations in Khor Al-Zubair near the discharge point of Fertilizer Factory. These stations were: (1) ammonia unit, (2) urea unit, and (3) water treatment unit. Figure (1) represents the location of Khor Al-Zubair. The samples were collected in glass bottles (three bottles for each station) and transferred to the laboratory with ice box.

Petroleum hydrocarbons were extracted from water samples by extraction with carbon tetrachloride solvent ( $\text{CCl}_4$ ) from water according to UNEP (1993). The samples were shaken several times, the extracts were dried by evaporation in rotary evaporator until dryness. Then to each sample, tetrachloride solvent is replaced by hexane solvent and then the Total Petroleum Hydrocarbons (TPHs) were estimated spectrofluorometrically by Ultraviolet Fluorescence (UVF) analysis.

The extraction and clean-up procedure for the determination of petroleum hydrocarbons in the sediment were made following Al-Saad (1995). Sediments were placed in a pre-extracted cellulose thimble and soxhlet extracted with 150 ml methanol: benzene (1:1) mixture for 24 hours. At the end of this time, the extract was transferred to a storage flask and the samples were further extracted with a fresh solvent.

The combined extracts were reduced in volume to a 10 ml in a rotary evaporator. It was then saponified for 2 hours with a solution of 4N KOH in (1:1) methanol: benzene. After extracting the unsaponified matter with hexane, the extract was dried over anhydrous sodium sulfate and concentrated by a stream of  $\text{N}_2$  for Ultraviolet Fluorescence (UVF) analysis.

Trace metals analysis was performed on the  $<63 \mu\text{m}$  fraction of the sediment has been separated by sieving after drying and grinding. According to Sturgeon *et al.* (1982), concentrated HCl and  $\text{HNO}_3$  (1:1) were added to each sample and evaporated to near dryness on a hotplate at  $80^\circ\text{C}$ , then a mixture of concentrated  $\text{HClO}_4$  and HF (1:1) were added. After heating to near dryness, 20 ml of 0.5 HCl were added and cooled for 10 min. The extraction was decanted into 25 ml plastic volumetric flask. This step was repeated twice and all supernatant were combined. Finally, samples were stored prior to trace metals analysis using a Pye-Unicam Atomic Absorption type SP9 Pye-Unicam.

The Total Organic Carbon (TOC) of the sediment was determined using the wet oxidation method. This method is described by El-Wakeel and Riley (1957).

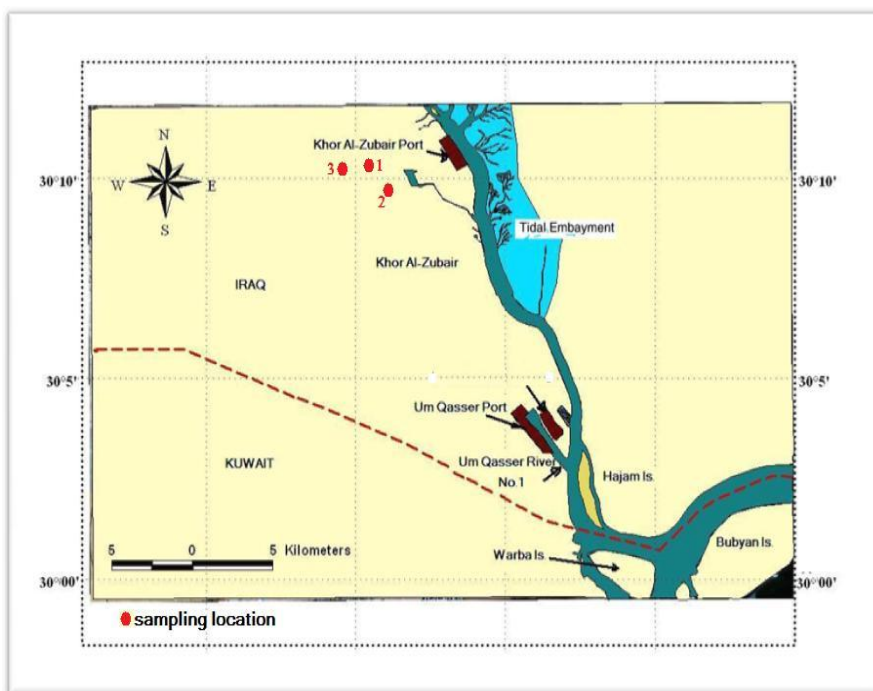


Figure 1. Location map showing the sampling location (Khor Al-Zubair).

## Results and Discussions

The mean concentrations of total petroleum hydrocarbons in water ranged from  $81.07 \mu\text{g/l}$  at station (1) to  $153.69 \mu\text{g/l}$  at station (2) and  $8.89 \mu\text{g/l}$  at station (3). In the sediments samples, the mean values ranged from  $129.46 \mu\text{g/g}$  at station (1) to  $406.68 \mu\text{g/g}$  at station (2) and  $48.97 \mu\text{g/g}$  at station (3). All results are shown in Table (1) and represented in Figures (2 and 3) for petroleum hydrocarbons in water and sediment, respectively.

Table 1. Range and mean concentrations of petroleum hydrocarbons in water and sediments in three locations.

station	Region	petroleum hydrocarbons in water ( $\mu\text{g/l}$ )		petroleum hydrocarbons in sediments ( $\mu\text{g/g}$ )	
		range	mean	range	mean
1	wastes of ammonia unit	80.06-82.03	81.07	128.03-130.26	129.46
2	wastes of urea unit	151.03-155.07	153.69	405.58-408.26	406.68
3	wastes of water treatment plant	8.81-9.02	8.89	47.31-50.23	48.97

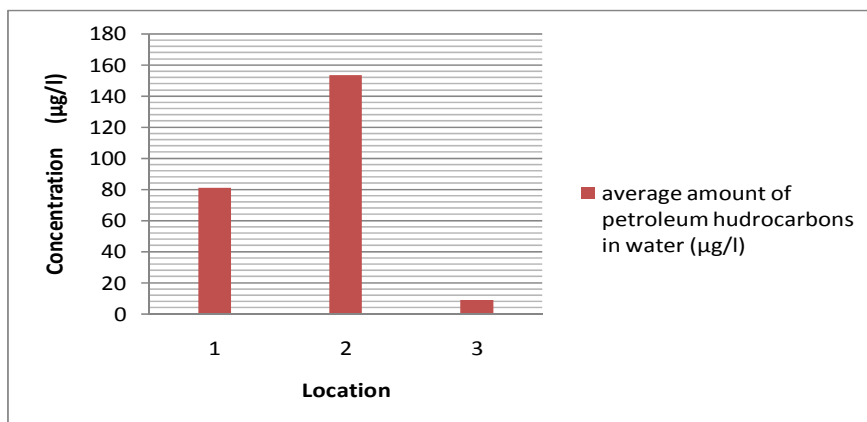


Figure 2. Mean concentrations ( $\mu\text{g/l}$ ) of petroleum hydrocarbons in water in three locations.

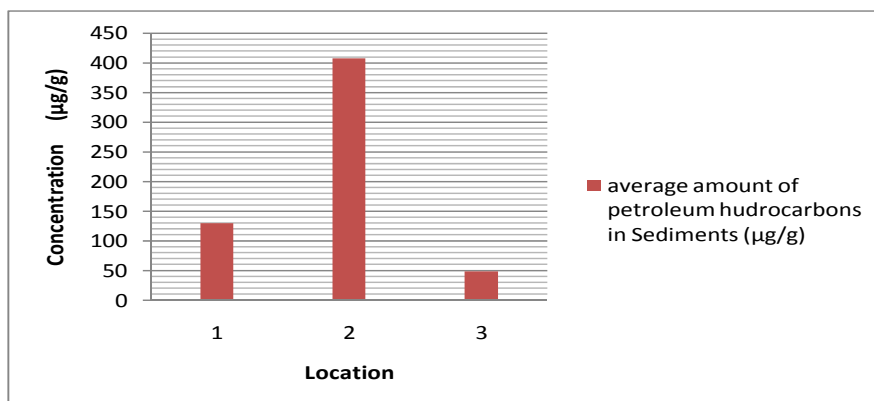


Figure 3. Mean concentrations in ( $\mu\text{g/g}$ ) of petroleum hydrocarbons in sediments in three locations.

The highest level of hydrocarbons concentrations in water and sediment were found at station (2). Al-Saad *et al.* (2009) estimated the hydrocarbons in water and sediment of the Marshland of southern Iraq and found that these concentrations were low. Al-Saad *et al.* (2006) studied the existence of hydrocarbons in Khor Al-Zubair and found that the concentrations were high. In this study and for the same area (Khor Al-Zubair), the effect of industrial location's wastes (in this case fertilizer company) took into account and hydrocarbons for waters and sediments have been estimated and found that these concentrations were also high compared with other studies. Tables (2 and 3) represent comparison of oil concentrations in waters and sediments in different parts of the World.

Table 2. Comparison of oil concentrations in waters in different parts of the Worlds.

Region	Concentration ( $\mu\text{g/l}$ )	Reference
Qatari beach	1.2-428	El-Samra <i>et al.</i> (1986)
Kuwait beach	2.1-3.6	
Antarctica	0.5-1.57	Bicego <i>et al.</i> (2003)
Baltic Sea	0.01-0.14	Pikkarainen & Lemponen (2005)
Khor Al-Zubair	36.8-478.2	Al-Saad <i>et al.</i> (2006)
Southern Iraq	0.012-0.037	Al-Saad <i>et al.</i> (2009)
Damietta Harbor, Egypt	14.5-65.5	Shereet (2009)
Niger Delta	19.3-27.9	Clinton <i>et al.</i> (2009)
Shatt Al-Arab river, Iraq	2.65-15.6	Al-Imarah <i>et al.</i> (2010)
The Gulf of Thailand	0.10-12.5	Wattayakorn (2012)
Brazil	0.0000344	Celino <i>et al.</i> (2012)
Khor Al-Zubair	8.92-153.57	The present study

Table 3. Comparison of oil concentrations in sediments in different parts of the world.

Region	Concentration ( $\mu\text{g/g}$ )	Reference
Kuwait beach	28	Fowler <i>et al.</i> (1993)
Oman beach	6-22	
San Francisco Estuary (Delta)	31	Oros & Ross (2004)
Khor Al-Zubair	2.07-38.5	Al-Saad <i>et al.</i> (2006)
The Aden Coast, Yemen	0.28-26.24	Bedair <i>et al.</i> (2006)
North west Spain	0.0009-0.422	Franco <i>et al.</i> (2006)
Malaysia	0.058-1.689	Sakari <i>et al.</i> (2008)
Southern Iraq	0.03-0.96	Al-Saad <i>et al.</i> (2009)
Niger Delta	336.16-436.72	Clinton <i>et al.</i> (2009)
Damietta Harbor, Egypt	0.16-4.16	Shereet (2009)
India	1.48-4.23	Veerasingam <i>et al.</i> (2010)
Shatt Al-Arab river, Iraq	7.373-24.415	Al-Imarah <i>et al.</i> (2010)
Bushehr Coastal Zone, Iran	0.844-4.972	Mahmoodi <i>et al.</i> (2012)
Khor Al-Zubair	48.396-407.47	The present study

Levels of trace metals (Fe, Mn, Cu, Zn, Pb, Cd and Co) in waters and sediments in Khor Al-Zubair has been estimated and given in Tables (4 and 5) for the three stations. The range of these measured metals in mg/l for water samples were 480.13-620.12 for Fe, 280.23-400.26 for Mn, 3.28-10.23 for Cu, 18.23-31.28 for Zn, 3.26-9.23 for Pb, 0.23-1.05 for Cd and 6.25-10.21 for Co. The range of these measured metals in  $\mu\text{g/g}$  for sediment samples were 632.18-980.45 for Fe, 280.23-400.26 for Mn, 9.26-16.53 for Cu, 30.12-51.24 for Zn, 23.41-30.23 for Pb, 0.62-1.52 for Cd and 20.21-32.42 for Co.

Table 4. Concentrations of trace metals in water samples (mg/l).

Station	Fe	Mn	Cu	Zn	Pb	Cd	Co
1	580	320	7.26	20	6.24	0.96	8.23
2	620	400	10.23	31	9.23	1.05	10.21
3	480	280	3.28	18	3.26	0.23	6.25

Table 5. Concentrations of trace metals in sediments ( $\mu\text{g/g}$ ).

Station	Fe	Mn	Cu	Zn	Pb	Cd	Co	TOC%
1	790	320	12	40	26	0.82	22	1.62
2	980	400	16	51	30	1.52	32	2.30
3	632	280	09	30	23	0.62	20	1.25

Estimation of Pb concentration is very important. The United State Environmental Protection Agency (USEPA, 1986) had classified this metal as hazardous and toxic to most forms of life. Pb metal concentration has not been estimated by Al-Edanee *et al.* (1991) and Al-Saad *et al.* (2007) for their studies in the same area (Khor Al-Zubair) to make a comparison. But due to other studies in sediments, the Pb concentrations were high. Cu is an important element for organism's life. The irregular existence for this metal is due to regeneration of organic matter which Cu forms soluble and insoluble metals. Mn is a metal that has a biological significance in aquatic environment; industrial activities represent the main reason behind this metal existence. As a comparison with other studies, there is no significant change observed in its concentrations. Co and Zn are significant metals for some metabolic activities in organisms. The results in Table (5), the concentration of Co is relatively low, but Zn is higher than that it was found in the history data.

The measurements of Total Organic Carbons (TOC%) were 1.62 % for station (1) and 2.30 % for station(2) and 1.25 % for station (3) as shown in Table (5). Table (6) represents a comparison of trace metal concentrations ( $\mu\text{g/g}$ ) in sediments in various estuaries, seas and oceans. TOC concentration is one of the main reasons that affect distribution of heavy metals as described by Al-Saad *et al.* (1996). The concentrations of TOC were the highest in station (2) among all stations and these concentrations in station (1) were higher than in station (3). Therefore, the concentrations of heavy metals in this study took the same context and the stations could be arranged according to that as station (2)> station (1)> station (3) in whole cases.

Al-Saad *et al.* (2007) showed that the sources of metals in Khor Al-Zubair due to industrial discharge, shipping activities, city run-off and atmospheric fall-out. For water and sediment samples, maximum levels of the whole metals were found at the wastes of urea unit (station 2).

*Geochemical index:*

According to Muller (1969), who classified the geochemical index for 6 degrees (Table 6), the geochemical index values for studied metals excluded Fe and Cd (Table 7) in the sediments of the three stations exhibited class 2 and hence are moderately polluted. Station (2) was the highest in the geochemical index. This can be indicated that all the sampling locations may face a severe trace metal contamination problem especially under the Khor Al-Zubair water condition.

Table 6. Geochemical index ( $I_{geo}$ ) classes with respect to sediment quality

Index	Class	Sediment Quality
0-0	0	Unpolluted
0-1	1	Unpolluted to moderately polluted
1-2	2	Moderately polluted
2-3	3	Moderately polluted to highly polluted
3-4	4	Highly polluted
4-5	5	Highly polluted to very highly polluted
5-6	<5	Very highly polluted

Table 7. The geochemical indexes of sediment for the studied locations.

Station	Fe	Mn	Cu	Zn	Pb	Cd	Co
1	-2.19	2.04	1.57	0.87	2.13	0.58	2.89
2	-1.88	2.36	1.99	1.22	2.33	1.47	3.43
3	-2.51	1.85	1.16	0.45	1.96	0.17	2.75

*Enrichment Factors:*

The enrichment factor (EF) is an opportune measure of geochemical trends and it is used for making comparisons between areas according to pollution degrees (Sinex and Helz, 1981). Zhang and Liu (2002) interpreted that a value of enrichment factor between 0.5 to 1.5 suggests that increasing in the concentration of metal may be related to crustal materials or natural weathering processes (Table 8). Table (9) represents the Enrichment factors values of some trace metals measured in the sediments of the studied locations according to the results of Al-Hashimi and Salman(1985) as a background data. All the sampling locations have enrichment factors values greater than 5 except station (1) for Cd and station (3) for Zn. According to earth crust level of metals, there is no enrichment for the Cu and Co and severe enrichment for Mn and minor to moderate enrichment for Zn, Pb and Cd (Table 10). Feng *et al.* (2004) recommended the enrichment factor values of less than 1 should be viewed with concern as they mean preferential release of these metals, making them bioavailable but if these values are greater than 1.5, they mean that the trace metal is delivered from



others sources suggesting environmental contamination by that particular trace element. If the enrichment values is high that is indicate an anthropogenic source of trace metals, mainly from activities such as industrialization, urbanization, deposition of industrial waste.

Table 8. Enrichment factors of some trace elements of the studied locations according to earth crust levels (David and Lide, 2005).

Stations	Mn	Cu	Zn	Pb	Cd	Co
1	24	0.61	3	3	3	0.16
2	24	0.65	3	3	5	0.13
3	26	0.52	3	4	2	0.20

Table 9. Values of enrichment factors for some trace metals in sediment according to history data.

Station	Mn	Cu	Zn	Pb	Cd	Co
1	19	14	9	19	5	32
2	21	17	15	27	20	43
3	18	11	5	17	6	37

Table 10. Degree of enrichment factors for trace element of the studied locations according to Acevedo-Figueroa *et al.* (2006).

EF value	Degree of enrichment
< 1	no enrichment
1 – 3	minor
3 – 5	moderate
5 – 10	Moderately severe
10 – 25	Severe
25 – 50	very severe
>50	extremely severe

#### Contamination factor (CF):

According to Hakanson (1980) (Table 11), sediment samples collected from Khor Al-Zubair fall under the category of low for Fe, moderate for Zn and Cd except location (2) which showed CF 4 marginally falls under considerable degree of contamination and considerable to very high contaminated for the rest metals. Degree of contamination was very high for all sample locations (Table 11).

Table 11. Contamination factor and contamination degree categories according to Hakanson (1980).

Contamination factor	Contamination degree	Status
CF<1	CD<7	Low degree of contamination
1<CF<3	7<CD<14	Moderate degree of contamination
3<CF<6	14<CD<28	Considerable degree of contamination
CF>6	CD >28	Very high degree of contamination

**Pollution index:**

The pollution index (PI) is a simple method to assess the extent of pollution by metals (Tomlinson *et al.*, 1980). It is based on the concentration factor (CF) of each metal in the sediment (Liu *et al.*, 2005) where values of PI=1 indicates heavy metal loads close to the background level, and values above 1 indicate pollution (Cabrera *et al.*, 1999). All sediment samples of the present locations had a pollution index greater than 1 (Table 12). So, the sediments in Khor Al-Zubair were heavily or moderately polluted.

Table 12. Contamination factor, contamination degree and pollution index for sediments of the studied locations.

Station	Fe	Mn	Cu	Zn	Pb	Cd	Co	Contamination degree	PI
1	0.33	6	4	3	7	2	11	34	445
2	0.41	8	6	3	8	4	16	45	1278
3	0.26	5	3	2	6	2	10	29	219

**Conclusion**

As a conclusion, the industrial discharge in station (2) was higher than that in the other stations besides reasons that mentioned in discussion. The recommendation is to find treatment methods for industrial wastes to decrease the pollution caused by petroleum hydrocarbons and heavy metals.

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## تقييم التلوث بالهيدروكربونات والعناصر النزرة في مياه ورواسب مخلفات معمل الأسمدة في خور الزبير العراق

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**المستخلص** - تم في هذه الدراسة حساب مستويات الهيدروكربونات والعناصر النزرة في النماذج المأخوذة من مياه ورواسب معمل الأسمدة في خور الزبير جنوبي العراق. أخذت النماذج من ثلاثة مواقع منتخبة في معمل الأسمدة وهي وحدة الأمونيا ووحدة اليوريا ووحدة معالجة المياه. وجد أن معدل تراكيز الهيدروكربونات للمياه كان 81.07 مايكروغرام/لتر عند وحدة الأمونيا و 153.69 مايكروغرام/لتر عند وحدة اليوريا و 8.89 مايكروغرام/لتر في وحدة معالجة المياه. تراوح معدل التراكيز في الرواسب بين 129.46 مايكروغرام/غرام عند وحدة الامونيا و 406.68 مايكروغرام/غرام عند وحدة اليوريا و 48.97 مايكروغرام/غرام في محطة معالجة المياه. تراكيز العناصر النزرة مقاسة بالمليغرام/لتر في النماذج المأخوذة من المياه كانت تتراوح بين 10.23 - 480.13 للحديد و 3.28 - 400.26 للمغنيز و 0.23 - 1.05 للنحاس و 18.23 - 31.28 للزنك و 3.26 - 9.23 للريصاص و 0.62 - 1.52 للكاديوم و 6.25 - 10.21 للكوبلت. بينما تراوحت تراكيز هذه العناصر مقاسة بالمايكروغرام/غرام للنماذج المأخوذة من الرواسب بين 980.45 - 280.23 للحديد و 9.26 - 16.53 للنحاس و 23.41 - 30.23 للريصاص و 0.62 - 1.52 للكاديوم و 32.42 - 32.42 للكوبلت. بينت النتائج التي تمت مقارنتها مع قيم عالمية ومحلية وجود تلوث بسبب المعالجات الصناعية لمعمل الأسمدة.