Levels of some heavy elements in water, sediments and two aquatic plants in Al-Garraf river at Shatra district/ southern Iraq

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Abstract. The present study was conducted in the Al-Garraf river in Thi-Qar governorate (October 2018 to March 2019) and aimed to investigate the pollution in the Al-Garraf river and the possibility of using some aquatic plants as bioindicator at three selected stations along the river. The study was achieved by measurement of air and water temperature, pH, and salinity, as well as the measurement of the concentration of some heavy elements (Cd, Pb, Ni, and Cu) in both dissolved and particulate phases in water and an exchangeable and residual phase in sediments and the tissues of two aquatic plants species, *Ceratophyllum demersum* L. and *Phragmites australis* (Can.). The mean concentrations of the heavy elements (Cd, Pb, Ni, and Cu) in the dissolved phase in water were (0.07, 1.19, 3.17, and 0.03µg/l) respectively, while their concentration in the particulate phase was (15.73, 24.71, 65.63 and 22.86 µg/g) respectively. The mean concentrations of the heavy metals in sediments in an exchangeable phase were 15.73, 24.71, 65.63, and 22.86 (µg/g), respectively, while in the residual phase were 0.07, 0.85, 74.62, and 14.00 (µg/g) respectively. Results confirmed increased mean concentrations of heavy metals in aquatic plants tissues compared to their means in water. The maximum mean of heavy metals was recorded in *Ceratophyllum demersum* L. The Bioconcentration factor (B.C.F) values were more than their values of Biosedimentation factor (B.S.F) in all aquatic species.

Key words: Heavy metals, Water, Sediments, Aquatic plants, Bioconcentration factor, Biosedimentation factor.

1. Introduction:

Water pollution as a result of human activities is significant for the environment of the Iraqi river. Water pollution in the river pollution, through human activities, oil spills from boats, and chemicals used in fishing, is the main problem in Iraq, and this led to the increased concentration of heavy metals in both water and sediment in the river area [1]. Aquatic plants play an essential role in removing pollutants from the wetlands [2][3]. They absorb pollutants from the aquatic environment and accumulate in their tissues or converted them to non-toxic materials [4]. The ability of plants to absorb pollutants varies among plants [5] and is affected by the proportion of pollutants in the aquatic environment [6]. Heavy Metals found in the Euphrates river, where high concentrations existed close to factories and high concentration density areas [7]. Another more recent study has found increased heavy metals in water in the Euphrates river in Thi-Qar due to anthropogenic activities [8]. Sediment is used as an indicator to assess heavy metals pollution. They are considered recipients and stores for these metals [9- 12]. Several studies investigate the contamination of heavy metals in sediment in the Shatt Al-Arab river [13-15]. Some researchers focused on aquatic plants as indicators for contamination by heavy metals [16-18]. This study aims to determine the concentration of heavy metals in water and sediment in the Al-Garaf river and to determine the viability of the selected plants (*Phragmitus australis* and *Ceratophyllum demersum*) to absorb these heavy metals, also to know the potential of using plants under the study in phyto remediation in future.
2. Materials and Methods

2.1. Description of the study area:
Tigris River is a branch passing through Kut dam and flowing into two major the former flows towards Mysan province, and the latter is penetrating Thi-Qar governorate towards Al-Shatra district. The river is distinguished by low gradient and sluggishly moving current, creating considerable sediment loads [19]. To conduct this study, three stations were selected near the Al-Shatra district (see Figure 1). The first station is located in the beginning of the entry of the Al-Garaf river in Al-Shatra district, the second station it about 10 km far from the first station. It is located near Al-Gun bridge, affected by domestic sewage, waste from land cultivation, and some private factories, while the third station was situated to the south of station 2 and Al-Shatra district. The distance between stations is about 10 km.

2.2. Sample collection
2.2.1. A-Water sample collection. Water samples were collected from three stations at the study area during the autumn and winter of 2018 and 2019. Water samples were collected using plastic bottles (polyethylene) with a capacity of 3 liters per sample. The bottles were washed before the sampling with a cleaning solution (nitric acid dilution), rinsed twice with deionized water, and dried to avoid any possible sample contamination.
2.2.2. Sediment sample collection. Sediment samples were collected using a sample collector (van ven grab sampler) at the same study area stations. Samples were placed in plastic containers and preserved in a cooling box until reaching the laboratory.
2.2.3. Plant sample collection. Plant samples were collected manually washed by river water for suspended solids. After that, they were saved in plastic bags until reaching the laboratory.

2.3. Physical and Chemical measurements:
The water and air temperature and water hydrogen ion concentration (PH) were measured directly in the field by a CyberScan series 600 waterproof portable meter, which was made in Singapore.
Water electrical conductivity was measured by using a CyberScan series 600 waterproof portable meters. The salinity was measured according to the equation: \( \text{Salinity} = \frac{\text{Ec} \times 0.64}{1000} \).

2.4. Extraction of heavy metals from water:
5 L of water was collected at each station. Samples were filtered using filter paper (0.45µm pore size). The filtered water was considered as dissolved, while the retained was particulate. Extraction of heavy metals in the dissolved phase was performed according to the [20] method. [21] The method was used to extract heavy metals in the particulate phase in water, where the filter paper was dried in an oven at 60°C for 6 hours until dry and weighted to get the values of the total particulate matter (PM). A specific weight taken from the particulate matter for digestion 0.5 gm of (PM) is taken for digestion by a 1:1 mixture of concentrated HCl and HNO₃ acids. After evaporation up to nearly dry, the digestion has further proceeded with a 1:1 mixture of concentrated HClO₄ and HF acids which were again evaporated up to nearly dry. The residue was dissolved in 20 ml of 0.5 N HCl, then made up to 25 ml with deionized water. All samples were stored in (25) ml Nalgene screw cap bottles and sealed for TMs analysis.

2.5. Extraction of heavy metals from Sediments:
Sediment samples were dried in an oven at 50°C for overnight. Finally, grinding in an agate mortar and sieved through <63 µm mesh sieve. The exchangeable trace metals were extracted using 20 ml (0.5 N) HCl overnight according to Chester and Voutsinou (1981). The solution was centrifuged at 5000 rpm for 20 minutes. The supernatant was filtered using pre-cleaned filter paper (Watman No.1). The filtrate was decanted into a 25 ml plastic volumetric flask. This step was repeated twice, and all supernatants were combined, and the final volume was made up to 25 ml with deionized water. All samples were then stored in naglen screw cap bottles (25 ml volume) and sealed for trace metals analysis [22].

The residue from the steps mentioned above was washed by 20 ml deionized water centrifuge for 30 minutes to clean the precipitate from acids. Then, the samples were digested with a mixture of 1:1 HCl: HNO₃ acids, evaporated up to near dryness with 80°C. The digestion was further proceeded with a 1:1 mixture of concentrated HClO₄ and HF acids digestion to get another. The residue was dissolved in 20 ml of (0.5 N) HCl and cooled for 10 minutes. The extractor was transferred into a 25 ml plastic volumetric flask, this step was repeated twice, and all supernatants were combined. Finally, the volume was reached 25 ml deionized water bottles and sealed for trace metals analysis [22].

2.6. Extraction of heavy metals from plants:
The determination of trace metals in different plants was done according to [23], where the digestion occurred by adding 10 ml (mixture 4 ml HCl and 1.5 HNO₃) to each sample, evaporated up to nearly dry on the hotplate at 80°C, then a mixture of concentrated HClO₄ and HF (1:1). Finally, the samples are transferred to a 25 ml volumetric flask, saved at room temperature. The volume is completed up to 25 ml by adding deionized water. All samples were stored in 25 ml plastic bottles. The trace metals were determined by using FAAS (M.S.C) as recommended by the.

2.7. Measuring of heavy metals
Concentrations of some heavy metals (Cadmium, Lead, Nickel, and copper) were measured by using the flame atomic absorption spectrophotometer. Model 210 VGP proved with hollow cathode lamps.

2.8. Calculation of Bio-Concentration factors (B.C.F) and Bio- Sedimentation factors (B.S.F)
The calculation of B.C.F and B.S.F for determine trace metals transfer among water, sediment and biota according to [24]:

\[
\text{B.C.F} = \frac{\text{[organism]}}{\text{[water]}} \\
\text{B.S.F} = \frac{\text{[organism]}}{\text{[sediment]}}
\]
2.9. Statistical analysis
All the analyses were conducted in triplicates for each station. The heavy metals values for the water, plants, and sediment were evaluated with the Two-way ANOVA considered to be statistically significant.

3. Results and Discussion

3.1. Physical and Chemical properties of water:
The values of all chemical and physical results, whilst the main and standard deviation for all locations of the study stations, are presented in (Table 1). The value of air temperature ranged from 10°C at station 1 in winter to 33°C at station 2 in autumn, while water temperature ranged from 7.3°C at station 1 in winter to 25°C at station 2 in Autumn. Slight fluctuations appeared in water pH values during the study period. The lowest value water pH was 7.19 in Autumn at station 1, which rose in winter to 7.44 at station 2. The water salinity was recorded at station 1 in the lowest value (0.43‰), while the highest value recorded was (0.85‰) at station 2 in Autumn. The current study showed that the highest water and air temperatures were recorded in Autumn compared with the lowest values in winter. In addition, the present data showed that water temperature was affected by changes in air temperature, and that was related, in part, to the shallowness of the water [25]. These results were confirmed by statistical analysis reflected in the significant correlation between air and water temperature (r=0.940, p<0.05). The finding of the current study was consistent with many previous studies in water bodies [26-28]. Salinity showed that during Autumn at station 2 the highest levels (0.85‰) were recorded due to an increase in evaporation rates and low water levels. However, the lowest salinity value was recorded in winter at station 1 due to an increase in rainfall and higher water levels. These results are supported by a range of literature [29-32], pH plays an essential role in solubility and hence mobility of heavy metals in the water column. A high pH value leads to increased heavy metals in the particulate phase or the sediment and a decreased concentration of heavy metals in the dissolved phase [33][34]. This is because the high pH does not lead to the dissolution of the hydroxide ion of metals, making the metals remain in the suspended materials [33]. This was confirmed by the present study results when the concentrations of heavy metals were higher in the sediment and particulate phase than in the dissolved phase.

| Factors       | Autumn | Winter | Mean | Standard division |
|---------------|--------|--------|------|-------------------|
|               | St.1   | St.2   | St.3 | St.1   | St.2   | St.3 |        |
| Water Temp.   | 24.2   | 25.2   | 25.1 | 7.3    | 10.1   | 8.1  | 16.66  | 2.12  |
| Air Temp.     | 30     | 33     | 32   | 10     | 12     | 11   | 21.33  | 1.45  |
| pH            | 7.19   | 7.22   | 7.2  | 7.40   | 7.44   | 7.42 | 7.31   | 0.21  |
| Salinity      | 0.78   | 0.85   | 0.79 | 0.43   | 0.60   | 0.60 | 0.67   | 0.03  |

3.2. Heavy Elements in Water:
The heavy element concentration in Al-Garraf river water through the time of the study was measured. The result illustrated the standard deviation for the seasonal changes of heavy elements in the dissolved and particulate phase. The concentration of heavy water elements was found in the following order: Ni>Pb>Cu>Cd, respectively. The concentration of heavy metals increased in winter more than autumn. Station 2 has more heavy elements than stations 1 and 3 (see Table 2). This may be due to the high population density near this station, and the municipal waste was directly discharged to the river by the main pipes without treatment. The effluents of municipal and industrial waste contain trace metals among their constituent [35]. These results could have happened as a result of the high activity from the population near the station. Also, there were many industrial discharges throughout the river without
treatment [35], especially in station 2. Interestingly, many trace metals concentration was greater in the particulate phase than the dissolved phase for all studied stations. The reason for increasing the concentration of heavy elements [36] had been found that the concentration of heavy elements is influenced by many issues like water discharge, seasonal plankton variations, chemical, environmental factors, for example (pH) and organisms that originate in sediment that results on increasing the concentrations of metals [37].

Table 2: Seasonal average values, ± standard deviation of eight heavy elements in the dissolved phase (µg/l) and particulate phase (µg/g) of water from Al-Garaf river.

| Season | Station | Cd Dis. ±SD | Pb Dis. ±SD | Ni Dis. ±SD | Cu Dis. ±SD |
|--------|---------|-------------|-------------|-------------|-------------|
| Autumn | 1       | 0.04±0.02   | 12.06±2.63  | 13.63±1.20  | 2.17±0.006  |
|        | 2       | 0.10±0.03   | 18.92±2.98  | 20.79±1.32  | 3.50±0.18   |
|        | 3       | 0.06±0.02   | 14.33±4.23  | 17.13±1.52  | 2.54±0.53   |
| Winter | 1       | 0.05±0.01   | 12.66±1.02  | 29.81±20.00 | 2.54±0.63   |
|        | 2       | 0.09±0.03   | 20.21±1.39  | 36.40±14.00 | 4.50±0.21   |
|        | 3       | 0.07±0.04   | 16.23±0.95  | 31.17±13.00 | 3.89±0.07   |
| Mean   |         | 0.07±0.025  | 15.73±2.2   | 24.71±8.50  | 3.17±0.27   |

St.: - station | Dis.: - dissolved phase | Part.: - particulate phase | ±SD: - standard deviation

3.3. heavy elements in sediment:
The concentration of heavy metal in sediment (µg/g) was measured. The concentration of these metals in sediments for both phases (exchangeable and residual) in all station were as follow: Ni > Cu > Pb > Cd (Table 3). The results showed that the high concentration of heavy elements was Ni, while Cd has the lowest concentration. Many studied for sediment showed a high concentration for heavy metal while the overlying water had less concentration [38]. Our result revealed that the amount of heavy metal in the residual phase more than in the exchangeable phase except Pb and Cd. This is due to the ability of the heavy metal to absorb a particular part of the sediment. Furthermore, the suspended matter with heavy metal tends to depose in the bottom and then absorbed by the organic matter and clay. All of these sources of heavy elements came from anthropogenic sources. Interestingly, station 2 has a high mean of heavy metals concentration, and this could due to wastewater discharges of leather facilities located nearby the river. In addition, increasing plant density in the river played an important role in increasing the heavy metals concentration in the sediments. Plant work to reduce the velocity of water flow, and this leads to the deposition of suspended matter containing heavy metals in the sediments. This was confirmed by the result of the current study, which found high concentrations of heavy metals in sediment compared with heavy metal concentrations in the dissolved phase. Statistical analysis showed significant differences between each of Nickel and Copper at p<0.01 and significant differences between seasons for Cadmium, Copper, and Nickel at p<0.05.

3.4. Heavy Elements in Plants:
Plants have been used as a good indicator of heavy element pollution due to absorb heavy metals from soils and sediments and accumulate them in their tissue [4]. Absorption of heavy metals varies depending on the plant species [5] and is affected by the bioavailability of the metals, the extent of their survival in the water [6], the PH, and the content of organic matter in the sediments [39].

In this study, there were differences in the heavy element concentrations among stations, seasons, and plant species (see Table 4). There were higher levels of heavy elements in plants in station 2 than in station 1.3. This is due to station 2 had exposed to different types of pollution such as sewage, oil spilled
from boats and chemicals used for fishing, while stations 1 and 3 were less polluted. The accumulation of heavy metals varied between the plants and between the plant parts (root, stem and leaves) [40]. The mean concentrations of heavy elements in *P. australis* were Cd (1.00µg/g), Cu (49.76µg/g), Ni (77.64µg/g) and Pb (1.10µg/g), while the mean concentrations of heavy metals in *C. demersum* were Cd (1.14µg/g), Cu (52.75µg/g), Ni (81.33µg/g) and Pb (1.27µg/g). The result showed that the *C. demersum* has the highest concentration of heavy metal than *P. australis*. Many factors affect the concentration of heavy metal in the plant, like soil texture, and depend on the evaluation of the plant, therefore; its found that *P. australis* has a low ability to absorption of heavy metal [41]. The results obtain find that the plant in station 2 had a high concentration of trace metal due to the huge density of the discharge wastewater. The higher concentration of metals in plants than in the dissolved phase is due to the higher proportion of organic matter in the sediment, which works to keep the heavy metals as long as possible and thus provide the opportunity for plants to absorb these metals, and also because of the high ability of plants to absorb these metals. The cause of the low concentration in the dissolved phase compared to other phases is due to the effect of various physical and chemical factors such as salinity, temperature, and PH, which leads to adhesion of metals with suspended materials, thereby reducing the concentrations of metals in the dissolved phase.

Finally, the results also showed the highest concentration of metals in all phases (water, sediments, and plants) were Nickel and Copper. This is probably due to the sources of pollution (sewage, oil spills, etc.) and the use of toxic chemicals in the process of fishing), which have high levels of these metals. There may be some contributions of these metals [1].

**Table 3:** Seasonal average values, ± standard deviation of eight heavy elements in the exchangeable and residual phase (µg/g) of sediment from Al-Garaf river.

| Season | Cd | Pb | Ni | Cu |
|--------|----|----|----|----|
| Autumn | Ex. ±SD | Res. ±SD | Ex. ±SD | Res. ±SD | Ex. ±SD | Res. ±SD | Ex. ±SD | Res. ±SD |
| 1 | 2.22±0.52 | 0.07±0.05 | 30.88±15.06 | 1.23±0.002 | 1.83±0.42 | 73.79±6.43 | 4.08±1.63 | 8.11±5.05 |
| 2 | 6.5±1.03 | 0.10±0.007 | 36.39±17.63 | 2.32±0.10 | 2.53±0.42 | 19.80±3.32 | 9.93±2.03 | 16.21±5.68 |
| 3 | 4.30±0.58 | 0.09±0.02 | 3.35±8.21 | 1.32±0.10 | 2.27±0.8 | 81.97±6.43 | 6.68±1.99 | 12.24±4.03 |
| Winter | Ex. ±SD | Res. ±SD | Ex. ±SD | Res. ±SD | Ex. ±SD | Res. ±SD | Ex. ±SD | Res. ±SD |
| 1 | 2.41±0.39 | 0.05±0.005 | 3.4±0.06 | 0.06±0.003 | 0.6±0.05 | 58.61±0.97 | 6.3±0.44 | 10.14±2.73 |
| 2 | 5.51±0.63 | 0.07±0.005 | 6.0±0.13 | 0.09±0.02 | 1.52±0.13 | 72.79±1.52 | 12.13±0.82 | 20.73±3.16 |
| 3 | 1.32±0.82 | 0.08±0.03 | 3.2±0.82 | 0.08±0.03 | 1.3±0.82 | 68.79±1.20 | 8.6±0.53 | 16.6±2.90 |
| Mean, ±SD | 4.05±0.61 | 1.92±0.012 | 17.32±7.13 | 0.85±0.04 | 1.6±0.59 | 74.62±3.31 | 7.9±1.24 | 14.0±3.92 |

Ex.: - Exchangeable phase Res.: - Residual phase St.: - station

**Table 4:** Concentration (means ±SD) of heavy elements in the plants µg/g dry weight at the study stations.

| Season | stations | *C. demersum* Mean±SD | *P. australis* Mean±SD |
|--------|----------|------------------------|------------------------|
|        | Heavy metals | Heavy metals | Heavy metals |
|        | Cd | Pb | Ni | Cu | Cd | Pb | Ni | Cu |
| Autumn | St.1 | 0.94±0.13 | 0.95±0.09 | 85.01±5.01 | 50.00±3.08 | 0.82±0.05 | 0.83±0.09 | 80.12±1.15 | 48.01±2.89 |
|        | St.2 | 2.02±0.83 | 2.23±0.95 | 87.03±6.03 | 59.83±2.19 | 1.78±0.77 | 1.9±0.83 | 83.30±1.22 | 54.10±4.00 |
|        | St.3 | 1.56±0.58 | 1.80±0.66 | 86.05±1.23 | 47.2±1.01 | 1.30±0.37 | 1.79±0.68 | 81.03±3.22 | 45.98±3.41 |
| Winter | St.1 | 0.61±0.07 | 0.43±0.4 | 74.10±1.50 | 51.10±6.01 | 0.51±0.04 | 0.19±0.03 | 72.30±3.01 | 49.2±4.01 |
|        | St.2 | 0.79±0.01 | 1.25±0.04 | 79.78±2.52 | 60.10±2.13 | 0.85±0.02 | 0.99±0.01 | 76.01±6.00 | 55.2±1.01 |
|        | St.3 | 0.99±0.02 | 1.00±0.05 | 75.80±2.00 | 48.3±1.04 | 0.79±0.03 | 0.97±0.06 | 73.10±4.01 | 46.0±2.01 |
3.5. Bio-Concentration (B.C.F) and Bio-Sedimentation (B.S.F) Factors:
The values of (B.C.F) in the study plants were higher than the values of (B.S.F) for all metals (see Table 5). The results show that the B.C.F values were more than B.S.F values. These results agreed with [15][17][26]. The B.C.F values of Cd, Cu, Ni, and Pb in C. demersum were more than P. austeralis. Metals in contaminated sediments are indirectly taken up by aquatic life (by partitioning of the metals into the ambient water, followed by their assimilation from the aqueous phase).

The C. demersum and P. austeralis up-taking these metals from the same source, the sediments considered as an archive for many organic materials that come from the death of organisms (plants and animals) [42-44], then accumulation in plants tissues.

Table 5: Bioconcentration factor (B.C.F) and Biosedimentation factor (B.S.F) in the tissues of two plant species.

| Metal | Metal concentration | C. demersum | P. austeralis |
|-------|----------------------|-------------|--------------|
|       | Dissolved water μg/l | Total sediment μg/g | Plant tissues | B.C.F | B.S.F | Plant tissues | B.C.F | B.S.F |
| Cd    | 0.07                 | 2.64        | 1.14         | 16.76 | 0.43  | 1.00         | 0.88  | 0.27  |
| Pb    | 1.19                 | 18.17       | 1.27         | 1.44  | 0.07  | 1.10         | 0.92  | 0.06  |
| Ni    | 3.17                 | 76.3        | 81.33        | 25.65 | 1.06  | 77.64        | 24.49 | 1.02  |
| Cu    | 0.033                | 21.95       | 52.75        | 2.19  | 2.04  | 49.76        | 1.51  | 1.26  |

4. Limitations of the Study
Due to the lack of time and resources, we were not able to test the effect of the plants that carried out the process of phytoremediation on the food chain, where the study can move to another level to experiment on two types of fish with different food habits of nutrition, where one is Herbivorous like Aspius vorax. The other is omnivorous like Cyprinus carpio, where the two kinds of fishes must breed on the plant subjected to the treatment process and test the ability of heavy metals transmission to the fishes through these plants, and measures the concentration of these metals in both kind of fishes.

5. Conclusions
1. The heavy metals concentration in the particulates phase is much more than the dissolved phase.
2. The heavy metals concentration in the residual phase is much more than the exchangeable phase except Cd, Pb elements can be attributed to anthropogenic sources.
3. Heavy metals concentrations were higher in station 2, and these, in turn, were higher than at stations 1 and 3.
4. The highest mean for heavy metals concentration was in the plants, followed by the sediment, then the water.
5. C. demersum accumulated heavy metals more than P. austeralis.
6. The B.C.F values for all elements were more than B.S.F values.

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