



Accumulation of some heavy metals in the vegetative parts of *Phragmites australis*, which grow in polluted soil by oily wastewater discharged from Salah Al-Din oil refineries, Iraq

Noor Sabah Al-Hammdani¹  , Ibrahim Omar Saaed²  , Rana Hashim Aloush³  

¹ Department of Biology, College of Education for Pure Science, Tikrit University, Tikrit, Iraq

^{2,3} Department of Biology, College of Science, Tikrit University, Tikrit, Iraq

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ABSTRACT

Environmental problems have consistently garnered significant attention from the scientific community. Pollution, particularly heavy metal contamination, represents a critical environmental concern with profound implications for human health. This study investigates the accumulation of lead (Pb), copper (Cu), cadmium (Cd), and nickel (Ni) in the roots, stems, and leaves of *Phragmites australis* plants grown in soils contaminated with these metals. The results showed that the highest concentration for (Pb, Cu, Cd and Ni) in roots which reached 0.771 ppm, 0.832 ppm, 0.094ppm and 0.062 ppm respectively while the lowest concentration was in the leaves of the plant which reached 0.378 ppm, 0.428 ppm, 0.064 ppm and 0.035 ppm for (Pb, Cu, Cd, and Ni) Respectively, results also indicated a direct correlation between the concentration of heavy metals in soil and in Plant Parts.

Keywords: Heavy metals, *Phragmites australis*.

Name: Noor Sabah Al-Hammdani

E-mail: Noor.Saed@tu.edu.iq



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تراكم بعض العناصر الثقيلة في الأجزاء الخضرية لنبات *Phragmites australis* النامية في التربة الملوثة بمياه الفضلات النفطية المصروفة من مصافي نفط صلاح الدين-العراق

نور صباح الحمداني¹، إبراهيم عمر سعيد²، رنا هاشم علوش²

¹ قسم علوم الحياة، كلية التربية للعلوم الصرفة، جامعة تكريت، تكريت، العراق

² قسم علوم الحياة، كلية العلوم، جامعة تكريت، تكريت، العراق

الملخص

تحظى المشكلات البيئية دائماً باهتمام كبير من العلماء، إذ يعد التلوث أحد الاهتمامات البيئية الحرجة التي تشكل تهديدات خطيرة على صحة الإنسان، وتأتي المعادن الثقيلة على رأس قائمة المواد السامة البيئية، وتركز هذه الدراسة على وجود المعادن الثقيلة، الرصاص (Pb) والنحاس (Cu) والكاديوم (Cd) والنيكل (Ni) في جذور وسيقان وأوراق النبات الذي ينمو في التربة الملوثة بهذه المعادن، أظهرت النتائج أن أعلى تركيز لـ (Ni, Cd, Pb, Cu) كانت في الجذور إذ بلغت 0.771ppm, 0.832 ppm, 0.094ppm و 0.062 ppm على التوالي بينما كان أقل تركيز في أوراق النبات بلغ 0.378 ppm, 0.428 ppm, 0.064 ppm, 0.035 ppm. لعناصر (Pb, Cu, Cd, و Ni) على التوالي. كما أشارت النتائج إلى وجود علاقة طردية بين تركيز العناصر الثقيلة في التربة وفي أجزاء النبات.

INTRODUCTION

Oil has been refined for various uses for at least 1000 years. An Arab handbook Written around 865 A.P. when AL-Razi described distillation of (naphtha) for use in lamps and thus the beginning of oil refining ⁽¹⁾, Petroleum industries have increased the threat of oil pollution to environment and their discharges in to natural environment create a major ecological problem throughout the world, the concentration of oil in effluents from different industries sources is found to be as high as 40000 mg/l ⁽²⁾, the wastewater considered to be one of most significant source of pollution around the world and has detrimental effects on natural ecosystem and water sources because wastewater contain residues of poisons, heavy metals, chemical and physical pollutants ⁽³⁾, Iraq has 15 oil refineries, Salah al- Din complex which has a production capacity of 311 thousand barrels per day and produced about 500 thousand of oily wastewater, Oily wastewater is defined as a wastewater that consist of fat, oil and grease coupled with a variety of dissolved substances (organic and nonorganic) in

suspension at high concentration ⁽⁴⁾, Electrical Conductivity (EC) is an important chemical parameters to describe the quality of the wastewater and the soluble salts and soil salinity and can be used for monitoring soil quality changes due to wastewater application information of soil ⁽⁵⁾, the Total Dissolved Substances (TDS) are the top of the parameters list of water quality that requires investigation for planning and management, especially for irrigation and drinking purposes, Chemical Oxygen Demand (COD) are indicator for evaluating the quality of wastewater, in most typical petroleum refinery wastewater samples, the average reported values of COD can be up to 600mg/l ⁽⁶⁾ Oil contamination will change the chemical properties of the soil such as pH, total organic carbon, soil minerals nutrients such as sodium, potassium and nitrate of soil, thus indirectly affecting the growth and development of plants and microorganisms ⁽⁷⁻⁹⁾ showed adversity influencing effect of heavy metals on the agriculture ecosystem (plant and soil) and human health ⁽¹⁰⁾, Numerous studies informed that

Cu toxicity could significantly inhibit soil microbes⁽¹¹⁾, The lead (Pb) has been listed as a hazardous heavy metal pollutant due to its high toxicity, the effects of Pb on soil several such as reducing soil nutrients, microbial diversity and soil fertility⁽¹²⁾, the studied indicated that the soil pH and cation exchange capacity where the important parameters influenced by Pb accumulation⁽¹³⁾, many sensitive plants growing in sites with heavy metals exposure showed altered metabolism .growth reduction and reduce biomass production and reduce yield^(14, 15) the combination of⁽⁶⁾, *Pragmatism austrias* are well known hyper accumulator emerged plants, they are capable to accumulate metals, Cu, Cd, Cr, Ni, and Fe⁽¹⁶⁾.

MATERIALS AND METHODS

During the Second Gulf War in 1991, the Salah Al-Din oil refineries and many tanks and wastewater treatment stations were destroyed. These historical events caused heavy oil pollution in the areas surrounding the oil refineries and contaminated the surrounding zones. This research came, the length of the wastewater canal which transports the oil refinery's effluent to the Tigris River is about one kilometer, this distance was divided into four equal sites and the beginning of each site was considered a sampling site, the first site at the beginning of the canal and the last site at the river⁽¹⁷⁾.

Wastewater samples were collected from five sites using 5 L plastic bottles, and soil samples were collected monthly from the bottom of the wastewater canal, with each sample weighing 1 kg. This process was conducted once at the same five locations. Chemical and physical analyses were conducted to determine the properties of wastewater and soil. The concentration of heavy metals in the soil was analyzed by digesting a 0.5 g sample with a 2:1 mixture of nitric and perchloric acids. Before analysis, the soil sediments were diluted 1:10 to ensure accurate measurement, then filtered and brought to 50 mL with deionized distilled water, and then analyzed using an AA202 atomic absorption

spectrometer. Plant samples were cleaned and dried at 70°C for 48 hours. Subsequently, 0.5g of each sample was digested in a 2:1 mixture of nitric acid/perchloric acid, and the resulting solution was diluted to 50mL using deionized water. Elemental concentrations were determined using Atomic Absorption Spectrophotometry (Spectrolab AA202), and sulfate and phosphate concentrations were quantified using UV spectrophotometry (JASCO V-530). Oil residues were extracted with carbon tetrachloride, partitioned with 0.1N hydrochloric acid, and quantified with methyl orange indicator at 50°C. Sediment samples were dried at 70°C for 24 hours before analysis. All analyses were performed in triplicate. Water samples were filtered using a 0.45µm filter and stored at 4°C. They were then digested in a 2:1 mixture of nitric acid/perchloric acid at 120°C. Total elemental analysis was performed in triplicate. Statistical analysis was conducted using SPSS software⁽¹⁸⁾.



Fig. 1: Illustrative diagram of the channel of the wastewater carrier that discharged from the North Refineries Company in Biji city /Iraq

RESULTS AND DISCUSSION

The results of chemical and physical properties of oily wastewater samples appeared in [table 1](#), the results showed that the values of the pH ranged

between 6.7 in site (1) and 7.3 in site (5) with significant difference according to Duncans test at $P < 0.05$ as for the values of EC it has ranged between 756 - 1484 $\mu\text{S}/\text{cm}$ in site (5) and site (1) respectively with significant difference according to Dunkin test at $P < 0.05$. the site (1) recorded values for PO_4 , SO_4 , TSS, TDS, COD and BOD_5 of 1.65, 258, 1610, 764, 1865 and 684 mg/l respectively, there was a gradual decreases as we moved from the first site towards the fifth site recording values of 0.42, 144, 614, 454, 512 and 486 mg/l respectively with significant differences according to Duncans test at $P < 0.05$ as for the values of heavy metals Pb, Cu, Cd and Ni, the first site recorded the highest values reaching 0.68, 0.18, 0.05 and 0.16 ppm respectively with a significant difference according to Dunkin test at $P < 0.05$. In contrast, the remaining sites recorded decreases of 0.40, 0.08, 0.02 and 0.08, respectively⁽¹⁹⁾, showed that the EC values ranged between 295-405 $\mu\text{S}/\text{cm}$ as far as the total dissolved solids (TDS), their average was 182.5 mg/l, and pH values ranged between 6.8-7.95, while the values of (COD) were 85- 108 mg/l. While showed that the

EC values ranged between 590 – 3492 $\mu\text{S}/\text{cm}$ and the pH values ranged between 7.02-7.85 and the SO_4 concentration ranged between 49.67 – 796.27 ppm, the concentration of oil residues reached at the first site 1242 ppm, then the concentration gradually decreased with a significant difference reached at the fifth site 544 ppm this may be due to the deposition of these materials in the soil layers or there absorption by the plants and storage in their tissues or because of their dilution with river water, on the other hand the concentration of Pb, Cu, Cd, Ni, was the highest in the first site and showed a slight difference in the concentration of the heavy metals at last site, the concentration of the Pb ranged from 0.68 – 0.40 ppm, 0.18-0.08 ppm for Cu, 0.046-0.022 ppm for Cd. And. 16-0.08 ppm for Ni, and the Cu was the highest concentration, while the Cd was the lowest concentration⁽²⁰⁾ showed that the concentration of Pb in the soil was 163 ppm, which was higher than the normal concentration, while the concentration of Cu, 42 ppm, was lower than the normal values.

Table 1: The physical and chemical properties of the wastewater that is discharged from Salah Al-Din petroleum refineries

Parameters	St1	St2	St3	St4	St5	average
EC $\mu\text{S}/\text{cm}$	1484 a	1160 b	844 c	864 c	756 d	1022
TSS	1610 a	1354 b	1182 c	856 d	614 e	1123
pH	6.8 b	6.7 b	7.2 a	7.2 a	7.3 a	7.0
SO_4 mg/l	258a	212 b	188 c	162 cd	144 d	193
PO_4 mg/l	1.56 a	1.42 a	0.86 b	0.48 c	0.42 c	0.95
TDS mg/l	764 a	532 b	518 b	466 c	454 c	547
COD mg/l	1865 a	1216 b	944 c	856 d	512 e	1079
BOD mg/l	684 a	512 b	526 b	432 d	486 c	528
Oil residues ppm	1242 a	1024 b	866 c	682 d	544 e	872
Pb ppm	0.68 a	0.52 b	0.44 cd	0.48 bc	0.40 d	0.50
Cu ppm	0.18 a	0.14 a	0.08 b	0.08 b	0.08 b	0.11
Cd ppm	0.05 a	0.04 a	0.02 b	0.02 b	0.02 b	0.3
Ni ppm	0.16 a	0.12 ab	0.14 a	0.12ab	0.08 b	0.12

Table 2 presents the chemical and physical properties of the soil samples collected from the same five sites at the bottom of the wastewater

Channel, which carries the wastewater from the wastewater treatment Stations to the Tigris River. The results showed that the pH values were

neutralized with an average of 7.4. In contrast, the concentration of other parameters(EC, PO₄, SO₄, oil residues) are highest in the first site recorded 842 μ S/cm, 1316, 346, 432 mg/l respectively and decreased dramatically until the fifth site to recorded 466 μ S/cm, 318, 142, 0.86 mg/l with significant difference according to Duncans test at P <0.05 level,, on the other hand the concentration of Pb, Cu, Cd, Ni was highest in the first site. The concentrations decreased. The highest concentrations of lead (Pb), copper (Cu), cadmium (Cd), and nickel (Ni) were observed at the first site,

with concentrations declining gradually toward the last site. Specifically, Pb concentrations varied from 82.24 to 0.48 ppm (dry weight), Cu from 3.12 to 0.34 ppm, Cd from 0.48 to 0.06 ppm, and Ni from 2.84 to 0.32 ppm. Statistical analysis using Duncan's test revealed a significant decrease in pollutant concentrations at the P < 0.05 level. This reduction is likely due to the role of reed-bed roots as hosts for aerobic bacteria and to the plants' ability to absorb atmospheric oxygen, which they transfer to their roots to oxidize organic matter and reduce pollutants^(21, 22).

Table 2: The physical and chemical properties of the polluted sediment by wastewater discharged from Salah Al-Din petroleum refineries

Parameters	St 1	St 2	St 3	St 4	St 5	average
pH	7.6 a	7.6 a	7.2 b	7,4 ab	7.2 b	7.4
EC	842 a	664 b	618 c	432 d	466 d	604
Po ₄ mg/l	1316 a	1224 b	642 c	538 d	318 e	808
SO ₄ mg/l	346 a	218 b	232 b	156 c	142 c	219
Oil residue mg/l	432 a	116 b	12 c	2.6 d	0.86 d	113
Pb ppm	2.24 a	0.82 b	0.64 bc	0.46 c	0.48 c	0.93
Cu ppm	3.12 a	0.88 b	0.72 b	0.38 c	0.34 c	1.08
Cd ppm	0.48 a	0.22 b	0.16 b	0.062 c	0.06 c	0.19
Ni ppm	0.94 a	0.62 b	0.34 c	0.36 c	0.32 c	0.51

Table 3 shows the concentrations of heavy metals in the roots, stems, and leaves of *Phragmites australis* that grow in the wastewater channel. The results indicate higher concentrations of Pb, Cu, Cd, and Ni. Were found in the *Phragmitis australis* which growing at the first site compared to their concentration at the other four sites with a significant difference according to Duncans test at P<0.05 level and the concentrations of four heavy metals were higher in the roots of the plants compared to those found in stem and leaves, the concentration of Pb , Cu , Cd ,Ni in the roots of the plants which grow in the first site (0.771 , 0.832 ,

0.094 , 0.062 ppm) on a dry weight respectively while their concentration in the roots of plant which grow in the fifth sites 0.216 , 0.316 ,0.022 , 0.020 respectively with significant difference according to Duncans test at P<0.05 level , the results also indicated a direct correlation between the concentration of heavy metals in the bottom sediments and in the plant parts, these results agree with what ^(23, 24) indicated about the accumulation of some heavy metals such as Pb , Cd , Ni , in the tissues of plants growing in polluted soils and that the highest concentrations found in the roots of plants .

Table 3: The concentration of heavy metals in the roots, stems, and leaves of *Phragmites australis* grown in sediments polluted by wastewater

Heavy metals concentration ppm	Plant parts	St1	St2	St3	St4	St5
Pb	roots	0.771 a	0.416 b	0.312 c	0.116 e	0.216 d
	steams	0.466 a	0.286 b	0.156 c	0.082 d	0.046 d
	leaves	0.378 a	0.242 b	0.184 c	0.088 d	0.062 d
Cu	roots	0.832 a	0.418 b	0.426 b	0.242 d	0.316 c
	steams	0.642 a	0.432 b	0.318 c	0.328 c	0.242 d
	leaves	0.428 a	0.422 a	0.0316 b	0.246 c	0.312 b
Cd	roots	0.094 a	0.052 b	0.044 b	0.024 c	0.022 c
	steams	0.063 a	0.044 b	0.038 b	0.016 c	0.020 c
	leaves	0.064 a	0.042 b	0.032 b	0.020 c	0.016 c
Ni	roots	0.062 a	0.034 b	0.022 c	0.018 c	0.020 c
	steams	0.033 a	0.028 a	0.018 b	0.020 b	0.000 c
	leaves	0.035 a	0.032 a	0.024 b	0.018 b	0.000 c

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