Egyptian Journal of Aquatic Biology & Fisheries
Zoology Department, Faculty of Science,
Ain Shams University, Cairo, Egypt.
ISSN 1110 – 6131
Vol. 27(4): 33 – 48 (2023)
www.ejabf.journals.ekb.eg

The Lead Removal from Industrial Wastewater Using Two-Stages Poly-Urethane Column for Further Use in Aquaculture

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ARTICLE INFO

Article History:
Received: May 26, 2023
Accepted: June 23, 2023
Online: July 9, 2023

Keywords:
Wastewater treatment,
Lead removal,
Polyurethane,
Two-stages column

ABSTRACT

Lead (Pb) pollution adversely affects the growth and welfare status of various aquatic animals. Thus, the search for treating agents having the capability to remove Pb from the polluted wastewater is worthwhile. Hence, the two-stage polyurethane foam (PU) column was applied for lead (Pb) removal from industrial wastewater. The foam was packed into a column for easier use. The effect of different factors such as the weight of foam, the contact time and the pH degree were studied. Results revealed that the lead removal percent (LRP) was the best at pH 5 and 105 minutes for 4g PU/0.01 moles lead. The highest LRP reached 88% after the first stage and 98.6% after the second stage. The foam was used for Pb removal from a real sample of industrial wastewater. The LRP range was 62.1% - 88.2% for the tested samples after the first stage and 85.6% - 98.6% after the second stage.

INTRODUCTION

Due to the growing aquaculture sector, water needs are increasing day by day. Indiscriminate discharge of raw and partially treated industrial effluents into the aquatic ecosystem leads to the deterioration of the environment. The main source of heavy metals pollution in aquatic ecosystems comes from mining activities, industrial effluents and anthropogenic activities among others **(Guowei *et al.*, 2021)**. These discharges generally contain many heavy metals (HM), forming a global pollution problem, especially in aquatic ecosystems **(Rajeshkumar *et al.*, 2018; Al-Halani *et al.*, 2022; Monier *et al.*, 2023)**. HM including lead (Pb) toxicity adversely affects the survival, growth, innate immunity, and hepatic antioxidant indices of aquatic animals inhabiting the environment **(Abdel-Tawwab *et al.*, 2017a, b, c; Hamed *et al.*, 2022)**. Hence, it is necessary to preserve water resources with acceptable water quality for aquaculture industry.

Some heavy metals including lead (Pb) are abundant in industrial wastewater, with a significant negative impact on aquatic ecosystems, groundwater, agricultural production and human health **(Wang *et al.*, 2020)**. Heavy metals are not biodegradable;

therefore, they could be accumulated in organs of aquatic animals to levels above the accepted limits, causing devastating effects on their health since they stay in their bodies for a very long period **(Hu *et al*., 2014; Huang, 2018)**. They may cause serious health-related diseases **(Li *et al*., 2017**). For example, too much zinc (Zn) may cause health problems like skin irritations, vomiting and stomach cramps **(Meligi, 2007; Bourliva *et al*., 2015)**. High concentration of nickel (Ni) causes cancer of lungs and kidney **(Iqhrammullah *et al.*, 2020)**. Consequently, the rejection water from these processes needs a treatment step before discarding them to reduce the adverse effects of pollutants to less toxicity forms **(Hahladakis *et al*., 2013; Omran *et al.*, 2023)**.

Lead (Pb) is a very toxic metal even at low concentration, accordingly Pb-contaminated wastewater must be treated before being discharged to the environment **(Azad *et al*., 2016; Zou *et al*., 2016)**. The polluted water containing Pb causes human health disorders and/or damage in stomach, kidney and brain **(Bayuo *et al*., 2019)**. The allowed limit of Pb to be discharged into inland water bodies and marine coastal regions according to the WHO are 1.0 and 2.0 ppm, respectively **(Emenike *et al*., 2016)**. The permitted Pb levels in drinking water are 0.01 ppm according to **WHO (1985)** and 0.015 ppm according to **USEPA (2023)**. These levels should be followed up by the authorities to safeguard the human health. In addition, the methods of Pb treatment should be modified and improved by extra studies for simplification and increasing efficiency. In this aspect, several methods were applied for the Pb removal from wastewater.

Lead and many other heavy metals were removed from aqueous solutions by pomegranate peel **(El-Ashtoukhy *et al*., 2008)**. They may be eliminated through a variety of processes, such as, biosorption **(Bayuo *et al*., 2022)**, chemical precipitation, electrodialysis, photocatalysis and adsorption **(Crini *et al.*, 2019; Omran *et al*., 2023)**. **Zhu *et al*. (2017)** used silica gel for the removal of Pb and cadmium (Cd). Zeolite is also capable of decontaminating Hg (II) and Pb (II) ions from aqueous media **(Kobayashi *et al*., 2020)**. In this context, employed zeolite A4 was used for Cd (II) and Pb (II) decontamination in water **(Makki, 2014)**; while, **Abdel-Tawwab *et al*. (2017c)** used rice straw, sugarcane bagasse, and maize stalks in removing Pb, Cd, copper (Cu) and Zn from wastwater. On the othr hand, several researches applied poly-urethane (PU) for the removal of heavy metals from wastewater **(Prakash *et al*., 2011)**. **Gama *et al*. (2006)** applied chitosan impregnated PU for Cu and Ni removal. Using PU foam/Me-BTANC for the removal of Pb and Cd, **Zhang and Banks (2006)** compared between PU immobilized sphagnum moss, seaweed, sunflower waste and maize for the removal of Cu, Pb, Zn and Ni from wastewater. Moreover, **Shamsi *et al*. (2017)** used PU modified with bis (2-ethylhexyl) phosphoric acid for the removal of Zn, Pb and Cd. Furthermore, **Jiříček *et al.* (2017)** applied nanofiber PU for water desalination.

Although the above methods used PU for wastewater treatment, they might have used hazardous chemicals for the preparation of the PU or attached some chemicals to improve its properties. While, in the present study, simply PU was applied as balls of its pure state without introducing any chemicals neither treated by any hazardous chemicals for the removal of lead from wastewater. The two-stage process of adsorption was introduced as a simple way to obtain pure water. In addition, the impact of different parameters on the adsorption efficiency was addressed. Therefore, the present study was managed to investigate the ability of PU foam for catching Pb from polluted wastewater based on its adsorption properties. This included studies for the best performance condition and introducing an original high efficiency two-stages column.

MATERIALS AND METHODS

1. Chemicals and preparations

Distilled water was used to prepare every aqueous solution. Washed sand (purchased from local store building staff) was used as column filler. The used chemicals were of an analytical grade type. Lead nitrate ($Pb(NO_3)_2$), nitric acid (HNO_3), ethylene diamine tetra acetic disodium salt ($EDTANa_2$), sodium citrate ($Na_2C_6H_5O_7$), potassium nitrate (KNO_3), potassium dihydrogen phosphate (KH_2PO_4), disodium hydrogen phosphate dihydrate ($Na_2HPO_4.2H_2O$) were purchased (ALPHA CHEMIKA, Cairo, Egypt). Sodium tetraborate decahydrate ($Na_2B_4O_7.10H_2O$) was purchased (Sigma-Aldrich, USA). Ammonium hydroxide (NH_4OH), ammonium oxalate ($(NH_4)_2C_2O_4$), sodium hydroxide (NaOH) were purchased (Adwic Chemical Co, Cairo, Egypt). Sodium nitrate ($NaNO_3$), murexide, methyl orange and citric acid were purchased from El Nasr Pharma, Abo Zaable, Egypt. Hydrochloric acid (HCl), sodium chloride (NaCl), zinc sulphate heptahydrate ($ZnSO_4.7H_2O$), magnesium sulphate pentahydrate ($MgSO_4.5H_2O$) were purchased from Sigma-Aldrich, and Erichrome Black T (EBT) was bought from Sigma-Aldrich, USA. The stock lead solutions were prepared and standardized against standard 0.01 M Na_2-EDTA by back titration using EBT indicator.

The polyurethane foam was examined by (SEM) device (National Research Centre, Cairo, Egypt). The pH was measured by Adwa pH, meter model 1030, Romania, fitted with a combination glass-electrode. The low concentrations of lead solutions were measured by an atomic absorption spectrometry (Hollo Cathode Lamps and SAVANTA AA Lamps and a Perkin-Elmer AA lamps (Central lab Faculty of Veterinary Medicine, Zagazig University, Zagazig, Egypt). A glass column was designed at glass workshop, Ain Shams University, Cairo, Egypt.

2. Analytical procedures

The idea of measuring the different effects depends on measuring the concentration of lead in the solution before and after adsorption **(Zareh *et al*., 2022)**. Then, the efficiency of the (PU) was calculated as shown in the aforementioned equations (1 and 2). The efficiency of the (PU) was calculated by using the following equations:

The adsorbed “removed” amount of lead (Pb_{ads}) = Pb_f (mg/L) - Pb_i (mg/L) ……… (1)

The lead removal percentage (LRP) = 100 (Pb_{ads} / (Pb_i) ……………………….……..(2)

Where, Pb_{ads} is the amount of the adsorbed lead; Pb_f the quantity of lead remained in solution after adsorption (mg/L), and Pb_i is the initial concentration (mg/L) before adsorption.

3. Adsorption isotherms

The kinetics of adsorption data are most commonly described by the isotherms of **Freundlich (1906)** and **Langmui (1918)**. They were applied to detect the relation between lead adsorption at different concentration and different value of PU foam masses. The best results were measured by linear degradation. The adsorption factors were obtained from the intercept and slope of the linear tracts of these models. The highest degree of adsorption of PU foam surface was calculated by Langmuir equation (4).

$q_e = [q_{max}\ K_L C_e] / [1+K_L C_a]$ ……. (4)

The linear Langmuir model is expressed as given in equation (5).

$1/q_e= [1/q_{max}] + [1/q_{max} KL] [1/C_e]$ ……. (5)

Where, "C_e" is the remaining concentration solute at equilibrium (mg/L); q_e is the amount of solute adsorbed per unit weight of solid adsorbent at equilibrium (mg/g), and q_{max} and K_L are Langmuir constants related to the adsorption capacity and energy of adsorption, respectively. These values can be obtained from the plot of ($1/q_e$) against ($1/C_e$).

The linear plot ($1/q_e$) versus ($1/C_e$) shows that adsorption follows Langmuir isotherm. The q_o and b were calculated from the slope and intercept of the linear plots. The magnitude of a dimensionless constant R_L was used to determine the quality of Langmuir adsorption isotherm (the separation factor) that can be calculated by the following equation:

$R_L = 1/(1 + C_o k_L)$ ……….. (6)

The parameter RL indicates the shape of the isotherm accordingly:

Value of R_L	**Type of isotherm**
0 < RL > 1	Favorable
$R_L > 1$	Unfavorable
$R_L = 1$	Linear
$R_L = 0$	Irreversible

The calculated R_L value was 0.77, which means that the Langmuir isotherm is a favorable process for Cu on PU foam. The Freundlich isotherm model was applied for the adsorption of lead from water by PU foam. The model of nonlinear equation is expressed using equation 7, as follows:

$\log q_e= K_f + (1/n) \log C_e$ ……… (7)

Where, (K_f) is the Freundlich constant, and "n" is the adsorption constant for Freundlich in L/mg.

4. Effect of weight of the PU adsorbent

Different weights (1, 1.5, 2, 2.5, 3, 3.5, 4, 4.5, 5, 5.5 and 6g) were studied for the proposed adsorbent (PU) at pH 3.5. The initial concentration of lead solution was 10^{-2} mg/L. The sample was let in contact with adsorbent for 1 hour. For each case, the remained lead amount was determined. Then, the lead removal percentage (LRP) was calculated as shown in the aforementioned equations (1 and 2).

5. Effect of contact time

The contact time effect was studied for lead withdrawal. Various time periods (30, 45, 90, 105 and 120 minutes) were tried. The pH value was fixed at 4, and the adsorbent weight was 4g. Then, the percentage of removed lead was calculated as before.

6. Impact of pH degree

The effect of pH degree was studied for lead removal. Several pH values 2, 3, 4, 5, 6, 7 were tried when the lead concentration was 10^{-2} and contact time was 1 hour. For each case, the amount of the remained lead was determined. Then, the removed Pb amount and the removal% were calculated as shown in the above equations (1 and 2).

7. Effect of Pb concentrations

Different initial Pb concentrations (10^{-2}, 10^{-3}, 10^{-4}, 10^{-5} M) were applied to evaluate the impact of initial lead amount on the efficiency of the PU adsorbent. The pH value was adjusted at 4, and the adsorbent weight was 4g. The lead concentrations were measured by atomic adsorption spectrometer. The lead removal% was calculated as shown in the above equations of 1 and 2.

8. Preparation of a column for Pb removal by using PU

The proposed PU adsorbent was packed into a glass column. This column was designed especially for this work at Glass workshop, Ain Shams University, Cairo, Egypt. It was made from Pyrex glass with a height of 50cm and a diameter of 3cm; it was fitted with sintered glass G4 at its bottom end. It had a tap to adjust the flow rate of the solution. The column was packed with 2.5g of PU adsorbent.

9. Application of the column for Pb removal from Pb solutions

The proposed column was applied for the removal of lead from aqueous solutions. The column was applied for different concentrations of lead (10^{-2},10^{-3}, 10^{-4} and 10^{-5} M), at pH 4 at a rate of flow of 1mL/ min. The concentration of the remained lead in tested samples was measured by AAS.

10. Application of the column for Pb-removal from industrial wastewater

Wastewater samples were obtained from the discharge places of Cairo Company for Dyeing, El Nabwya Company for Dyeing (Zagazig, Sharqia Governorate, Egypt), Farm Frist (Cairo, Egypt), Factory Kaha for Preserved Food (10th of Ramadan City, Egypt) and the Egyptian Company for Prestressed Concrete (ECPC) (Concrete Factory, 10th of Ramadan, Sharqia Governorate, Egypt). A volume of 100 mL from each wastewater sample was transferred to a 100mL beaker, and 1.0mL of HCl was added to the sample. Then, 30mL of this HCl-treated sample were injected into the column subsequently. The flow rate of the lead solution through the column was adjusted at 1mL/ min. The samples were analyzed for lead before and after treatment by the proposed column. The LRP was evaluated using equations 1 and 2.

RESULTS AND DISCUSSION

1. PU characterization

Polyurethane (PU), traditionally and most frequently, polymers are created via the interaction of a di- or tri-isocyanate with a polyol. Polyurethanes are categorized as consecutive copolymers because they have two different types of monomers that polymerize one after the other. Polyurethane acts as an adsorbent, which performed a significant part in removing contaminants from polluted water. PU is a great adsorbent due to its valuable properties like mechanical stability, high specific surface area, high cation exchange capacity and layered structure. Additionally, it shows high selectivity for heavy metals.

The chemical structure of poly urethane is shown in Fig. (1). The scan electronic microscope (SEM) photos of the used polyurethane foam showed information about the nature of the foam surface (Fig. 2). Fig. (3) shows the EDX analytical results that proved the presence of hydrogen, carbon and oxygen. The PU foam size used in the current study has spherical shape with a diameter of 0.55cm, representing a volume of 0.091cm^3. The UP density was 0.017g/ cm^3, and its surface area was 0.96cm^2.

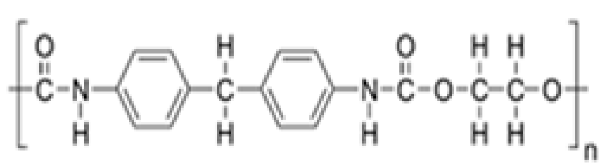
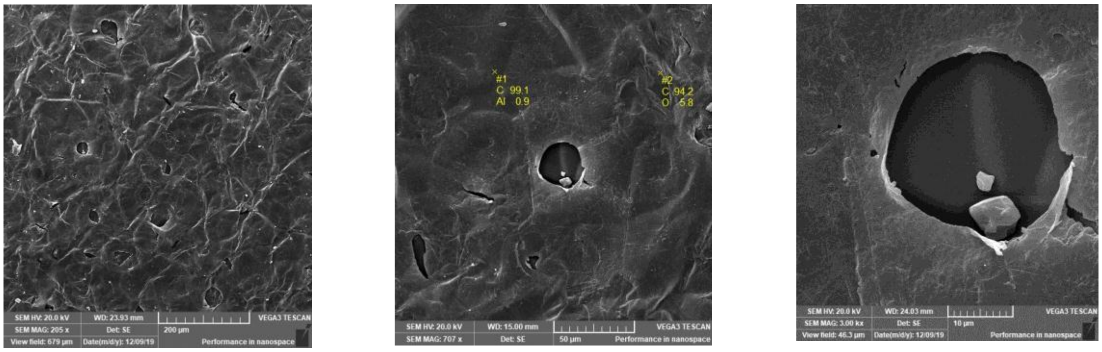


Fig. 1. Structure formula of polyurethane (PU)



A B C

Fig. 2. SEM image by TESACN, VEGA3, for PU surface at SEM HV:20KV and 205x(a), 707x (b), 3.0 Kx (c)

Fig. 3. EDX-analysis for Poly-urethane foam

2. Effect of weight of PU-adsorbent

Experiments were conducted for 1, 1.5, 2, 2.5, 3, 3.5, 4, 4.5, 5, 5.5 and 6g of poly-urethane. Each was soaked with a solution of 10^{-2} M of lead for one hour at pH 3. The concentration of the remained lead in the tested samples decreased by rising the adsorbent weight of PU. Subsequently, the LRP increase was caused by the increase of the exposed adsorbent surface area to the solution and existence of more adsorption sites with the same amount of lead cation. Fig. (4) shows the relation between the removal % and the amount of adsorbent. The highest value of removal% of lead ions was determined when the adsorbent weight was at 4g, while the lowest value removal (l%) of lead was at 1.0g.

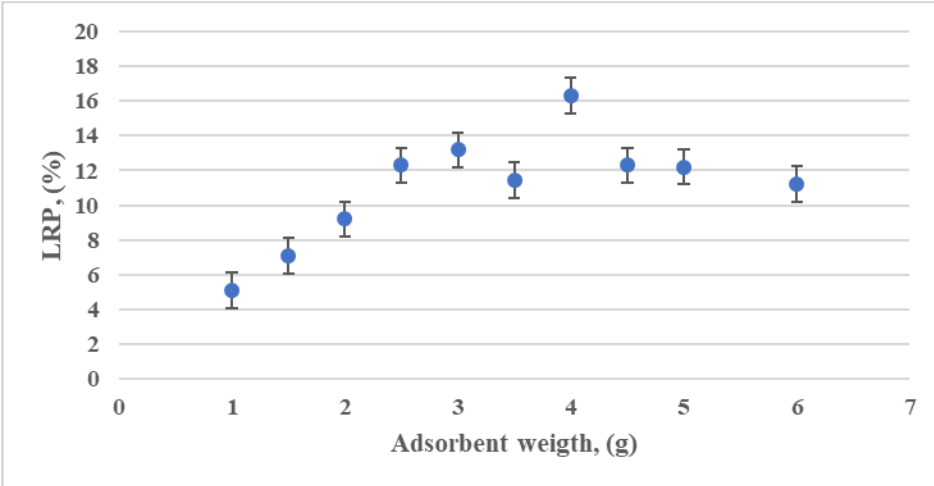


Fig. 4. Influence of PU foam weight on the Pb removal % (LRP)

3. Influence of exposure time

The contact time between the polyurethane foam and lead solution showed that the percent of removal increased by increasing the time till 105 minutes. Then, it obviously decreased at 120 minutes. Other factors were made constant (adsorbent weight 4g and pH 3). The starting amount of lead in the solution was 2.07 g/L. The increase in Pb-removal% was due to the longer time providing more possibility for adsorption on the surface of the foam as shown in Fig. (5). Increasing the Pb exposure time in the PU column is accompanied with an increase in the number of transported ions. Therefore, it is expected that metal ion removal will increase with time and with increasing voltage **(Senem & Belgin, 2023)**. The decrease after 105 minutes was due to the probability of the physical nature of the equilibrium **(Zareh *et al*., 2022)**.

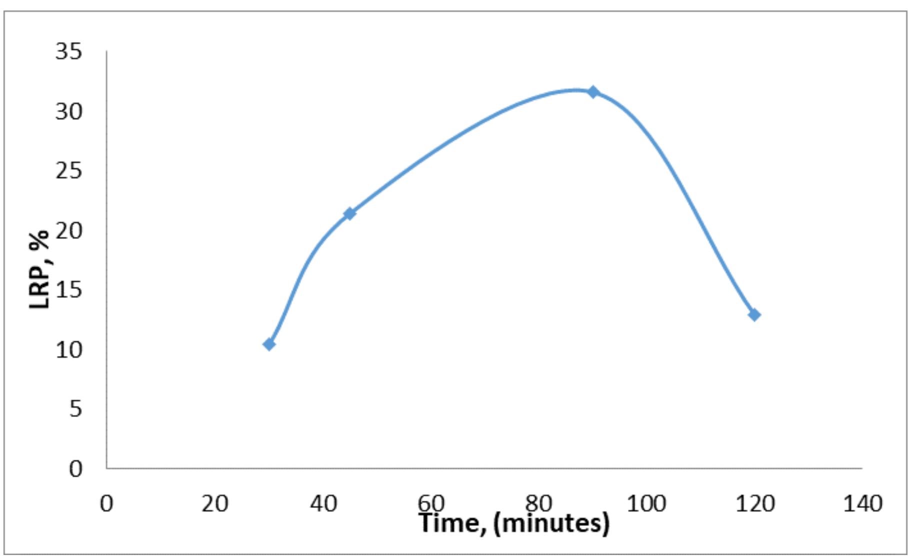


Fig. 5. The impact of time on LRP by using PU

4. Effect of pH degree

The pH is a crucial parameter for the solubility of the lead in solutions. However, low pH value solution indicates acidic media, and a competitive transfer can be expected across the membrane between the metal cations and protons **(Gering & Scamehorn, 1988)**. While, at higher pH, many metals may be precipitated. Therefore, choosing the appropriate pH range in the metal removal process in the PU column has great importance to achieve the greatest Pb removal operating efficiency. For this reason, in our study, the Pb adsorption was tested at different pH values (2, 3, 4, 5, 6 and 7). Whereas, other factors (adsorbent weight 4 g, contact time 105 min) were made constant. Fig. (6) shows the change of Pb removal at different pH values. The Pb-removal efficiency increased by increasing the pH value till 5. After that (pH 6 and 7), the Pb removal% decreased to percentages of 45 and 36 for the mentioned pH values, respectively. This is due to the competition from the OH^- increased concentration which forms $Pb(OH)_2$ (Fig. 6).

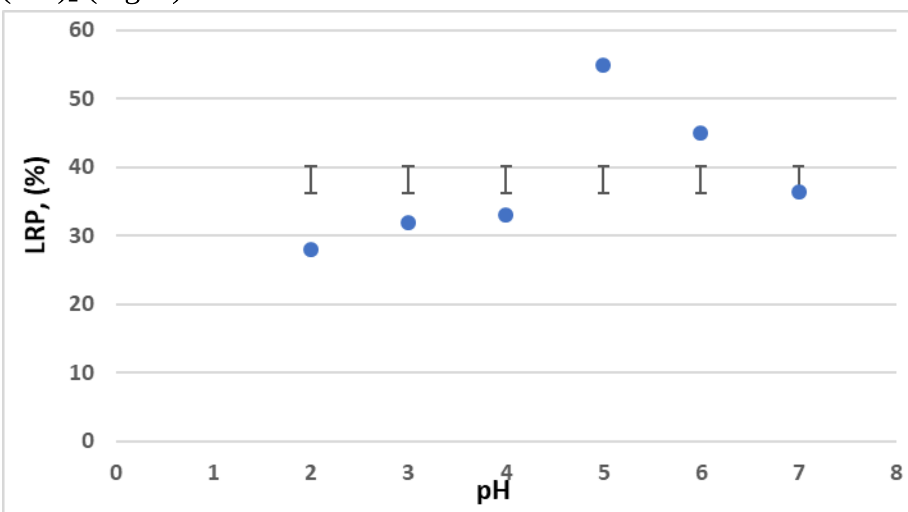


Fig. 6. Influence of changing pH on adsorption of lead by poly-urethane (PU)

At low pH, the lowest Pb removal value was found, which is related to the increase in acidity and the amount of hydrogen ions. Moreover, the Pb^{2+} competes with the H^+ ion at low pH values, and the rate of Pb removal is reduced, and it is easier to pass through the membrane as the pH decreases **(Kreysa *et al*., 2014; Li *et al*., 2021)**. In contrast to our results, **Omran *et al*. (2023)** reported that the Cu removal decreased after reaching the optimum value of pH 4.

5. Impact of initial Pb levels

The influence of initial levels of Pb in the solution were applied for a weight of adsorbent of 4g, pH 5 and the contact time of 105 minutes. Different Pb levels (254.5, 278.7 and 291.5 ppm) were tried. The removed lead amount was measured by AAS. The obtained LRP values are presented in Table (1). Results displayed the decrease in the Pb removal efficiency by the increase of the initial Pb amount in solution. This can be explained due to the more the Pb amount is competing for the adsorbed Pb on the PU-surface. In a similar study using an electrodialysis unit, **Omran *et al*. (2023)** reported a decrease in Cu removal efficiency at low initial Cu levels and visa versa; they reported that the increase in current efficiency and energy consumption are observed as the concentration increases. With increasing the metals concentration, excess ions can create resistance causing pollution. In this case, the ED cell will have an adverse effect, resulting in accumulation and stratification in the membranes, and the removal efficiency will decrease **(Sadrzadeh & Mohammadi, 2008)**. In the study of **Senem and Belgin (2023)** on Cr (VI) and Ni (II) removal by the electrodialysis method with a batch recycle, it is seen that the removal efficiency is much higher at low concentrations, regardless of the type of ion.

Table 1: The effect of Pb levels on LRP value by using 4g of the PU adsorbent

Sample No.	Initial Pb level (ppm)	LRP (%)
1	2.9	89.8
2	8.5	54.1
3	291.5	27.8
4	278.7	15.6
5	254.4	3.1

6. Application of PU column for spiked Pb water samples

The proposed column was used to remove different concentrations of lead. The results gave suitable values of lead removal%. At high initial Pb level (2.07 g/L), the removal% was small (7.3%). On the other hand, for 8.5 x10^{-3} - 3.5 x10^{-3} g/L M, the LRP range was 54.1% - 40.6%. For lower Pb level (2.9x10^{-3} M), an excellent LRP value (89.8%) was found (Table 2).

Table 2. Lead removal % by using the proposed PU column

Sample No.	Lead before (g/L)	First stage, (LRP %)	Second stage, (LRP %)
1	2.07	7.3	14.1
2	8.5x10^{-3}	54.1	79.6
3	3.5x10^{-3}	40.6	63.8
4	2.9x10^{-3}	89.8	98.9

When the two-stage column was applied, high LRP was achieved in case of 2.9ppm of initial Pb level. The 79.6 LRP was recorded for 8.5ppm of Pb amount. This technique was based on passing the raw water into two successive similar columns. Fig. (7) shows the schematic diagram of the proposed water treatment system.

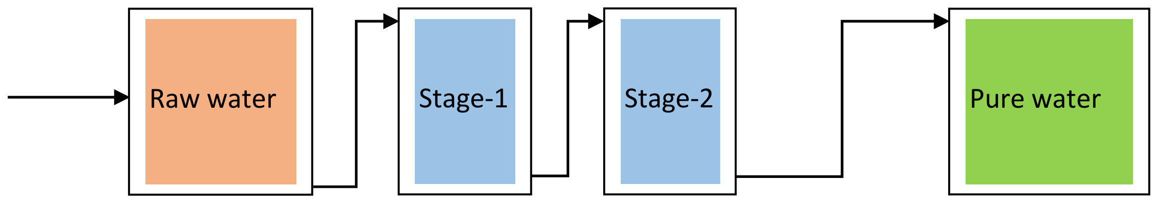


Fig. 7. Schematic diagram of the proposed two- stage water treatment system by using the proposed PU column

7. Application of PU column for industrial wastewater treatment

Several samples were obtained from 6 different factories. They were put in contact with the PU foam for 1h after filtration and measuring the concentration. Then, the LRP was determined as shown in Table (3). It could be recognized that the foam exhibited excellent results for all wastewater samples from the chosen companies (88.2% and 62%) after the first stage. Upon applying the second stage, the LRP reached 88.2% - 98.6%. For all samples, the pH was adjusted at 5, with the rate of flow of 1mL/ min.

Table 3. Application of PU for lead removal from industrial wastewater samples

Sample No.	Name of company	Initial Pb amount (ppm)	LRP first stage (%)	LRP second stage (%)
1	El-Nabwya company for dyeing	0.071	76.6	94.5
2	Farm Frist	0.026	65.7	88.2
4	Factory Kaha for Preserved Food	0.176	79.6	95.8
5	ECPC factory	0.1457	88.2	98.6
6	Cairo Company for Dyeing	0.0517	62.1	85.6

CONCLUSION

The PU succeeded to remove Pb^{+2} in the rejection water from different factories. It was found that the pure poly urethane foam was able to remove Pb^{+2} from wastewater, without addition of hazardous chemicals to the foam. The adsorption increases with increasing the amount of initial Pb^{+2}. The PU foam was packed to a column and applied for the first time for easier processing. The second stage column technique was effective for the lead removal. The lab experiments clarified that PU foam could be used to accomplish a good removal of harmful heavy metals with high amounts in industrial wastewater. Nevertheless, further studies should be done to evaluate the effects of produced water after the PU foam treatment on fish welfare.

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Arabic summary

إزالة الرصاص من مياة الصرف الصناعى على مرحلتين بأستخدام عمود البولى يوريثان للاستخدام في تربية الأحياء المائية

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نظر للتوسع فى الاستزراع السمكى فى الاونة الاخيره تتزايد الاحتياجات المائية كما ان تزايد الانشطة البشرية والزراعية قد تصل العناصر الثقيلة و منها عنصر الرصاص الى البيئة المائية مما دفع الباحثون للبحث عن طرق عملية لازالة و تقليل سمية عنصر الرصاص فى مياه الصرف لاستخدامها فى اغراض استزراع الاحياء المائية. تم تطبيق رغوة البولي يوريثان ذات المرحلتين (PU) من أجل إزالة الرصاص بكفاءة عالية من مياه الصرف الصناعي. تم تعبئة الرغوة في عمود لسهولة الاستخدام. وقد تم دراسة تأثير العوامل المختلفة مثل وزن الرغوة ووقت التلامس وعامل الأس الهيدروجيني ووجود أملاح أخرى. أظهرت النتائج أن نسبة إزالة الرصاص(LRP) جم 0.01/ كانت الأفضل عند درجة الحموضة 5 ، 105 دقيقة ، لـ 4 مول من الرصاص. بلغت أعلى نسبة إزالة للرصاص88 (LRP) ٪ بعد المرحلة الأولى و 98.6٪ بعد المرحلة الثانية. تم استخدام الرغوة لإزالة الرصاص من عينة حقيقية من مياه الصرف الصناعي. تراوح نطاق LRP بين 62.1 - 88.2٪ للعينات المختبرة بعد المرحلة الأولى و 85.6-98.6٪ بعد المرحلة الثانية.