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Scientific paper

Enhanced Adsorption of Lead (II) Ions from Aqueous Solution by a Chemically Modified Polyurethane

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Abstract

Heavy metal pollution is a major threat to living systems due to increase in the industrial development worldwide. In this study, the adsorption of lead (II) ions by chemically modified polyurethane was reported. Polyurethane (PU) was chemically modified by sulphonation and chlorination to obtain sulphonated PU (SPU) and chlorinated PU (CPU). The adsorption parameters such as pH, contact time, adsorbent loading and initial metal ion concentration were optimized in batch experiments for both the adsorbents. Maximum Pb (II) ion adsorption of 90 and 85% was observed for SPU and CPU respectively at optimal conditions. Isotherms results showed that the equilibrium data was fitted with Freundlich isotherm and followed multilayer adsorption mechanism. Adsorption of Pb (II) ions by both SPU and CPU followed pseudo second order kinetics. The outcome of this study showed that chemical modification of PU is effective for efficient removal of Pb (II) ions from effluent.

Keywords: lead; isotherm; chemical modification; polyurethane, kinetics.

1. Introduction

Heavy metal pollution in the environment was mainly caused by the industrial waste discharge to water bodies.[1] Unlike organic pollutants, heavy metal ions are stable and persistent environmental contaminants which are non biodegradable. Water contaminated by toxic metal ions remains a serious public health problem for human health.[2] Unique properties of lead like high ductility, flexibility, softness, resistance to corrosion and low melting point have resulted in its widespread usage in different industries like ceramics, plastics, automobiles, paint, etc. This in turn has led to a manifold rise in the occurrence of free lead in biological systems and environment. Human exposure to lead occurs through various sources like battery recycling, coal combustion, leaded gasoline and industrial processes etc. Lead toxicity is particularly insidious hazard with the potential of causing irreversible health effects. It was known to interfere with numerous body functions and it primarily affects hematopoietic, central nervous, renal and hepatic system producing serious disorders.[3] Chronic toxicity, on the other hand was much more common and occurs at blood Lead levels of about 40–60 μg/dL. It could be much more severe, if not treated in time resulted by encephalopathy, persistent vomiting, delirium, lethargy, convulsions and coma.[4,5]

Several methods commonly employed for Pb (II) removal from aqueous solution include biosorption,[6–7] nanofiltration,[8] ion exchange[9] and reverse osmosis.[10] But these methods have the disadvantages like less removal efficiency, require high energy, generation of toxic end products which need further treatments made these processes costly for heavy metal removal at lower concentration. Adsorption is an effective technique with many advantages like convenience, simplicity, efficiency including cost-effectiveness and minimization of secondary wastes. Polyurethane foam (PU) is a cheaper and thermodynamically favorable adsorbent material for heavy metal removal from industrial wastewaters.[11] Previously, PU was reported as an effective

adsorbent for removal of nickel, mercury, cadmium etc.[12–14] The porosity and high surface of PU make it suitable as efficient adsorbent for heavy metal removal.[15] Mangaleshwaran et al. reported that the efficiency of adsorption can be enhanced by chemical modification of PU.[12]

The aim of the present study was to evaluate the adsorption efficiency of chemically modified polyurethane for Pb (II) ion removal from aqueous solution. The batch optimization of adsorption parameters (pH, contact time, adsorbent loading and initial metal ion concentration) was investigated. Adsorption isotherms and kinetics of Pb (II) ion adsorption were also reported.

2. Experimental

2. 1. Synthesis and Chemical Modification of PU

25 mL of toluene diisocyanate (Merck, India) and 25 mL of tetra methylene ether glycol (Merck, India) was added in acidic condition and form homogenous mixture to initiate polymerization. In the polymerization reaction, initially toluene diisocyanate in acidic medium reacts initially with free available H^+ ions and then reacts with tetra methylene ether glycol to form PU. After completion of foaming, solid structured open cellular PU was cut into small cubes (1 cm) and used for further studies. The synthesized PUF was chemically modified by two methods (i) sulphonation and (ii) chlorination. To obtain sulphonated PU (SPU), 25 mL of 4N H_2SO_4 (Merck, India) added to 2 g of PUF and agitated for 45 min at 60 °C at 100 rpm. After this treatment, the cubes were dried at 105–110 °C for 3 h. In chlorination reaction, 50 mL of 0.5% bleaching powder (Sigma Aldrich, India) solution was added to 2 g of PUF and agitated for 45 min at 60 °C at 100 rpm. After this step, the cubes were dried at 105–110 °C for 3 hrs to get chlorinated PU (CPU) [12]. The chemically modified SPU and CPU were used for further Pb (II) ion adsorption in batch mode.

2. 2. Preparation of Pb (II) Adsorbate Solution

Pb (II) stock solution was prepared by dissolving 1.6 grams of lead nitrate in a 1000 mL volumetric flask and diluted using double distilled water to get a 1000 (mg/L) concentration. The sample solution concentrations 10-50 (mg/L) were prepared from stock solution by dilution using double distilled water and the pH of the samples were adjusted using 0.1N HCl and 0.1N NaOH.

2. 3. Batch Adsorption Studies Using SPU and CPU

The adsorption efficiency of SPU and CPU were studied in a batch mode by varying the adsorption parameters such as pH (2–6), contact time (5–150 min), adsorbent dosage (0.25–3g/50 mL) and initial adsorbate concentration (10–50 mg/L). At the end of each batch experiment, remaining Pb (II) ion concentration was determined by measuring absorbance in UV-Vis spectrophotometer at 520 nm as per IS 3025 (Part 47): 2003 procedure. The adsorption efficiency was calculated using following Eq.1

$$\text{Pb(II)adsorption efficiency} = = \frac{(Initial\ absorbance - final\ absorbance)}{Initial\ absorbance} x\ 100 \quad (1)$$

2. 4. Desorption and Reusability Studies

For desorption studies, NaOH was chosen as regenerant since the adsorption was carried out in acidic environment. 50 mL of 0.2, 0.4, 0.6, 0.8 and 1.0N NaOH was taken in 250 mL Erlenmeyer flask and 1 g of adsorbents (SPU, CPU after adsorption) was added separately in each flask, agitated at 100 rpm, 120 minutes for Pb (II) ions desorption. After desorption, the adsorbents were taken out and dried. The dried adsorbents were employed for adsorption of 50 mL of Pb (II) solution (10 mg/L) and their removal efficiency was calculated by measuring the absorbance after adsorption. A plot between maximum removal efficiency and concentration of regenerant was plotted and optimum dose of regenerant was selected. The adsorption and desorption process were repeated for 5 cycles and the Pb (II) removal efficiency were calculated.

3. Results and Discussion

3. 1. Batch Adsorption Studies Using SPU and CPU on Pb (II) ion Adorption

Batch experiments were carried out to study the factors affecting the adsorption process such as pH, contact time, adsorbent dosage and initial adsorbate concentration on Pb (II) ion adsorption by SPU and CPU.

3. 1. 1 Effect of pH

In order to study the effect of pH on Pb (II) adsorption, experiments were carried out for SPU and CPU at the pH range (2 to 6) keeping the contact time constant as 150 min. For SPU, the Pb (II) ion adsorption efficiency was observed increase with increase in pH from 2 to 4 and above pH 4, Pb (II) ion removal efficiency was declined (Fig.1a). The maximum Pb (II) ion removal efficiency of 90 % was obtained at pH 4 for SPU. Similar to SPU, the maximum Pb (II) adsorption efficiency (79%) was obtained at pH 4 for CPU whereas the adsorption efficiency was observed lesser than SPU (Fig.1a). The optimal pH for maximum Pb (II) absorption by both the adsorbent was found to be 4

and it was correlated with the results published previously.[16–17] The results revealed that the optimum pH at which the maximum Pb (II) ions removed by SPU and CPU were in the acidic range. At acidic pH, H^+ ions compete with Pb (II) ions at the adsorbent surface which would hinder Pb (II) ions reaching the bonding sites of adsorbate caused by the repulsive forces. At higher pH > 6, the Pb (II) ions get precipitated due to hydroxide anions forming lead hydroxide precipitate. This hydroxylated form metals can also compete with metal ions at the active sites of the adsorbent thereby decreasing the adsorption.[16]

3. 1. 2 Effect of Contact Time

The effect of contact time on the Pb (II) ion removal efficiency using SPU and CPU at optimized pH 4 was represented in Fig.1b. The Pb (II) ion removal efficiency of SPU was increased from 18 to 90% as the contact time was increased from 5 to 90 min and beyond 90 min, the adsorption of Pb (II) ion attained equilibrium. Khan et al.[18] obtained 87.6 % of Pb (II) ion removal using 0.1 g/L of multiwalled carbon nanotubes with the contact time of 90 min which was comparable to the present study. For CPU, Pb (II) ion adsorption efficiency was initially rapid and increased till 120 min. Further increase in contact time beyond 120 min to 180 min showed that adsorption attained equilibrium. The maximum Pb (II) ion removal efficiency (80%) by CPU was observed at 120 min. Nordiana and Siti[16] also reported similar contact time for maximum Pb (II) ions removal using activated charcoal and peanut shell. The Pb (II) ion removal efficiency by SPU and CPU increases rapidly during the initial stage which may be due to that adsorbent sites at the surface were empty and the adsorbate concentration gradient was high. Later, the adsorbate uptake rate was decreased mainly due to the unavailability of adsorption sites in the adsorbent surface.[19]

3. 1. 3 Effect of Adsorbent Dosage

Absorbent dosage is a critical parameter in the adsorption process to identify the optimal amount of adsorbent required for maximum adsorbate removal. The effect of adsorbent dosage (0.25–3 g/50 mL) on the adsorption of Pb (II) ion by SPU and CPU were studied at pH 4 and contact time 90 min and 120 min respectively. Fig.1c represents the Pb (II) ion removal efficiency by SPU and CPU at different adsorbent dosage. The optimal adsorbent dosage of SPU and CPU was found to be 0.75 g in 50 mL and 1 g in 50 mL and

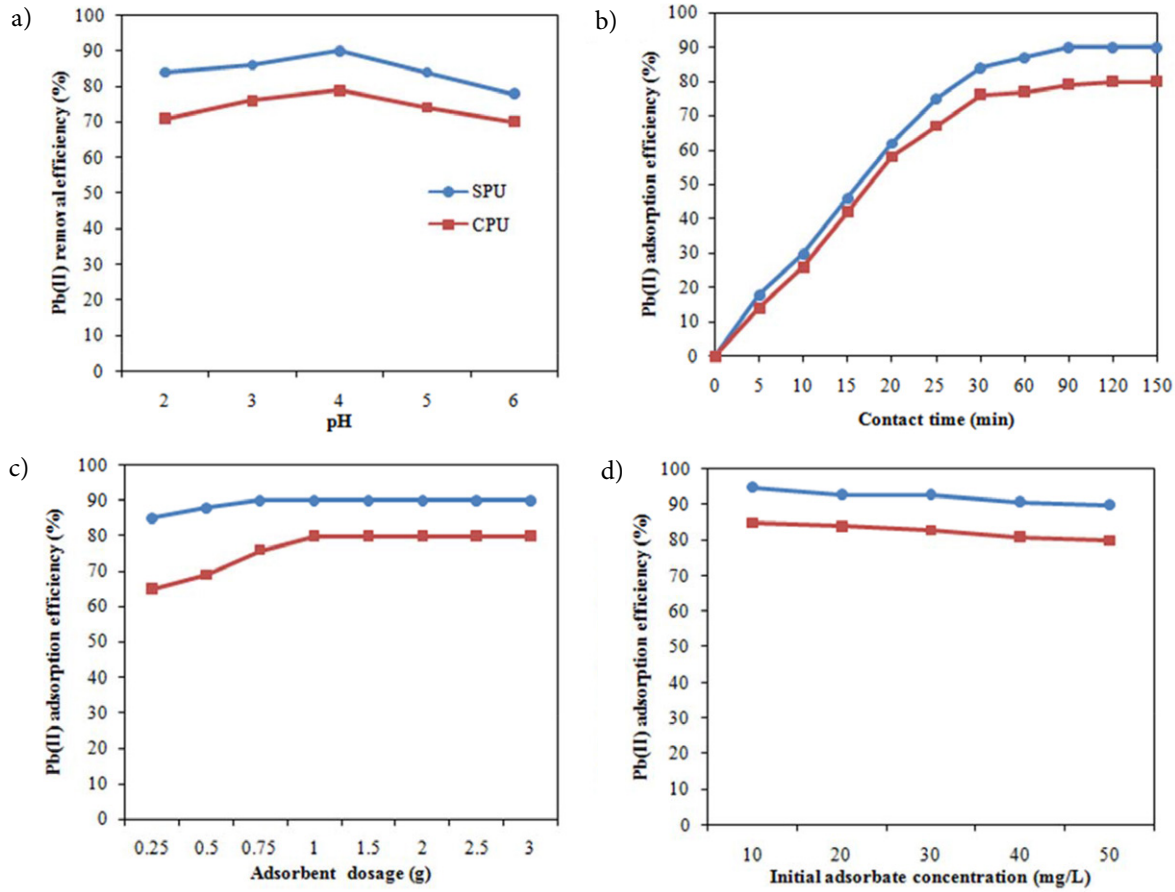

Figure 1. Effect of (a) pH, (b) contact time, (c) adsorbent dosage and (d) initial adsorbate concentration on Pb (II) ion removal efficiency using SPU and CPU

the maximum Pb (II) ion removal of 90% and 80% respectively. The increase in removal efficiency with the increase of adsorbent dosage was mainly attributed to the presence of more vacant active sites in the adsorbent surface.[20]

3. 1. 4 Effect of Initial Adsorbate Concentration

Experiments were carried out to study the effect of initial adsorbate concentration (10–50 mg/L) on Pb (II) adsorption by SPU and CPU with optimized pH 4, optimized contact time (SPU 90 min, CPU 120 min) and optimized adsorbent dosage (SPU 0.75 g/50 mL, CPU 1 g/50 mL).The trend of Pb (II) removal efficiency by SPU and CPU was shown in Fig.1d.At the initial Pb (II) ion concentration of 10 (mg/L), higher Pb (II) removal efficiency was expected than the other studied concentrations for both SPU and CPU. At lower concentration, the numbers of Pb (II) ions available in the solution are less as compared to the available sites on the adsorbent. However, at higher concentrations, the available sites for adsorption become fewer, and the percentage removal of lead ions depends on the initial concentration.[21]

3. 2. Adsorption Isotherms

The mechanism of Pb (II) ion adsorption onto the adsorbent surface was studied by fitting equilibrium data with Langmuir[22] and Freundlich[23] isotherm models represented in Eq.2 and 3.

$$\frac{C_e}{q_e} = \frac{C_e}{q_0} + \frac{1}{q_0 b} \quad (2)$$

$$\ln q_e = \ln K + \frac{1}{n \ln C_e} \quad (3)$$

where *q*e (mg/g) is metal adsorbed per mass of adsorbent, q_0 (mg/g) is maximum adsorption capacity, *b* (L/mg) adsorption energy constant, *C*e (mg/L) is equilibrium metal ion concentration, *K* and *n* were Freundlich isotherm constants.

Langmuir and Freundlich plots for SPU and CPU on Pb (II) adsorption were represented in Fig. 2a–d and the estimated isotherm model constant values were given in Table.1. Based on the R^2 value, both the adsorbents fitted well with both the isotherm models and are highly correlated with Freundlich model than Langmuir model (Table.1). The characteristic equilibrium separation factor R_L of Langmuir isotherm SPU and CPU were calculated as 0.299 and 0.640 respectively, which indicates the favorable adsorption of Pb (II) ion on the adsorbent.

The maximum adsorption capacity (q_0) for SPU and CPU were estimated from Langmuir plot as 5.435 and 5.495 mg/g respectively and the obtained results was compared with other adsorbents reported in the previous studies on Pb (II) ion adsorption (Table.2).The Freundlich isotherm

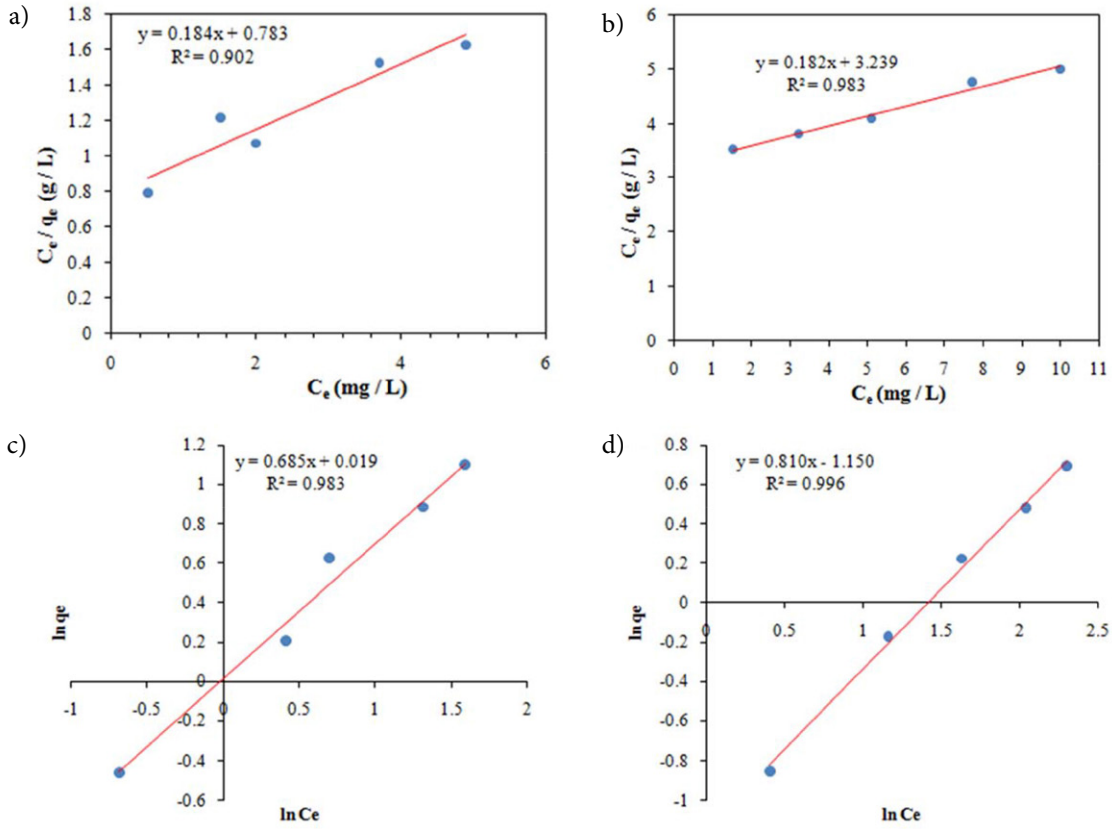

Figure 2. (a &b) Langmuir isotherms and (c & d) Freundlich isotherm for the adsorbents SPU and CPU.

Table 1. Estimated constants for Langmuir and Freundlich isotherm models for the adsorption of Pb (II) ion using SPU and CPU

Adsorbent	Langmuir model				Freundlich model		
	b (L / mg)	q_0 (mg / g)	$R_L = \frac{1}{1+bC_0}$	R^2	K_f (mg/g)	n (L / mg)	R^2
SPU	0.235	5.435	0.299	0.902	1.019	1.460	0.983
CPU	0.056	5.495	0.640	0.983	0.316	1.235	0.996

parameter 'n' measures adsorption intensity of adsorbent and the value of 'n' between 1 to 10 indicates favorable multilayer adsorption. Based on the obtained 'n' values for SPU and CPU, this study revealed that the adsorption of Pb (II) ion for SPU and CPU favored by multilayer mechanism.

3. 3. Adsorption Kinetics

The adsorption data were used to fit the pseudo first order[24] and pseudo second order[25] kinetic models to predict the controlling mechanism of adsorption.

Table 2. Comparison of Langmuir isotherm model parameters for Pb (II) ion adsorption using various adsorbents

Absorbent	Langmuir isotherm model parameters			Reference
	q_o (mg / g)	b (L / mg)	R^2	
Sesame leaf activated carbon	279.860	0.123	0.994	Liu et al.[31]
CS-Fe_2O_3 nanocomposite	214.92	0.077	0.991	Ahmad and Mirza,[32]
2,2'- Ethylenedithio diethanol immobilized amberlite XAD 16	107.52	11.625	0.999	Khalil et al. [33]
Maize green algae activated carbon	24.154	0.350	0.982	Suresh and Chandrasekaran, [21]
CPU	5.495	0.056	0.983	Present study
SPU	5.435	0.235	0.902	Present study
Maize leaf activated carbon	3.713	0.627	0.998	Uzma, [17]
Chitosan -G- Polyacrylonitrile	3.080	0.019	0.984	Shanmugapriya et al.[34]

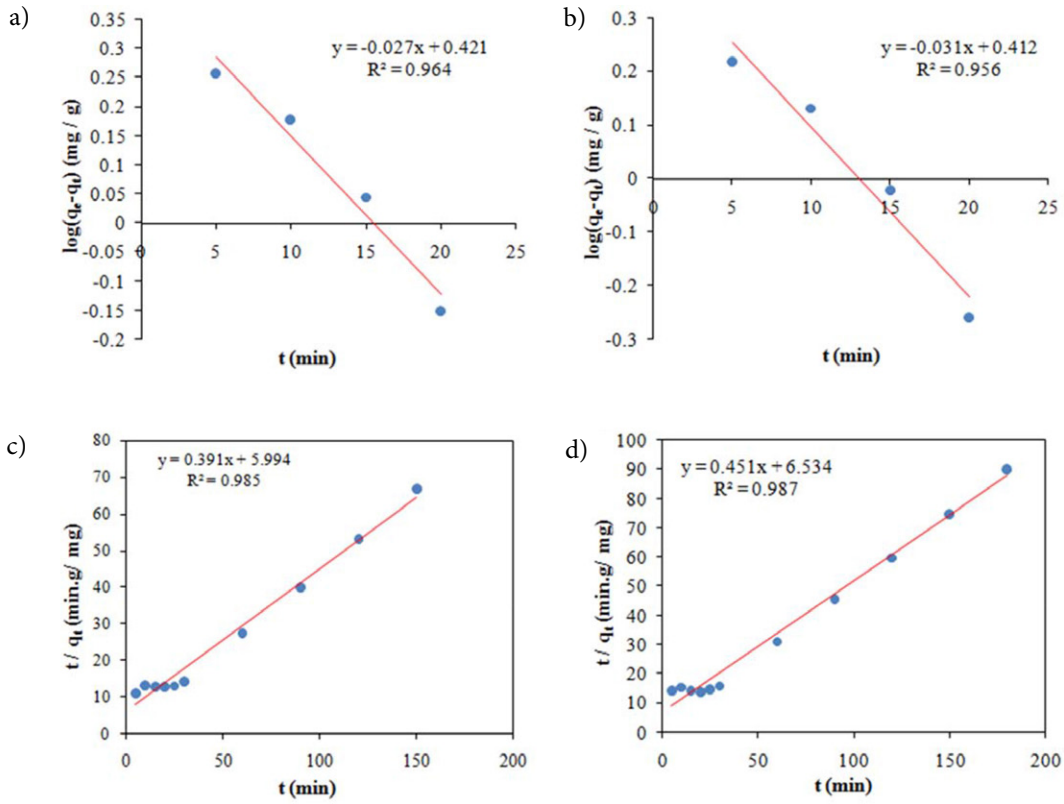

Figure 3. (a & b) Pseudo first order and (c & d) Pseudo second order kinetics plot for Pb (II) ion adsorption by SPU and CPU

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} t \quad (4)$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (5)$$

where q_e and q_t are the amount of metal ion adsorbed (mg/g) at equilibrium and at time t, k_1 is pseudo first order rate constant (min^{-1}) and k_2 is pseudo second order rate constant (min^{-1}).

The graphs were plotted between log (q_e–q_t) versus t and t/q_t versus t to estimate the rate constants for pseudo first order and pseudo second order kinetic model respectively. The kinetic plots for SPU and CPU on Pb (II) adsorption were shown below (Fig.3a–d). The estimated kinetic constants were given in Table.3 and observed that the pseudo second order model have good agreement with the experimental data since the R^2 values are closer to unity for both SPU and CPU. Similar results were reported for Pb (II) ions adsorption using different adsorbents.[26–27] The higher consistency of q_e experimental with q_e calculated from pseudo second order model indicates that the adsorption process was controlled by chemisorption.[28]

Table 3. Pseudo first order and pseudo second order kinetic constant for Pb (II) ion adsorption by SPU and CPU

Adsorbent	Pseudo first order constants			Pseudo Second order constants		
	$q_{e,cal}$ (mg . g^{-1})	K_1 (min^{-1})	R^2	$q_{e,cal}$ (mg . $g^{-1)}$	K_2 (min^{-1})	R^2
SPU	2.636	0.0621	0.964	2.558	0.0255	0.985
CPU	2.582	0.0713	0.956	2.217	0.2060	0.987

3. 4. Desorption and Reusability Studies

The recycling and regeneration of adsorbent was useful for making the process cost effective by reutilizing the adsorbent for several cycles.[29] Table.4 represented the maximum adsorption of Pb (II) ions using SPU and CPU as adsorbent for five adsorption – desorption cycles. From the adsorption- desorption cycles of Pb (II) adsorption by SPU and CPU revealed that the adsorption was reversible. However the removal efficiency of each adsorbent (SPU and CPU) was decreasing for each cycles. About 15–22% of reduction in adsorption efficiency was observed in SPU and 26 to 32 % reduction in adsorption efficiency was observed in CPU after 5 cycles. Similar results were obtained by Lingamdinne et al.[30] for Pb (II) ions using graphene oxide based nickel ferrite nano composite for 5 cycles.

4. Conclusions

The present study revealed that the efficiency of SPU and CPU for the removal of Pb (II) ions from the aqueous solution was highly depends on pH, contact time, adsorbent dosage and initial adsorbate concentration. 15 g /L of SPU effectively removed 95% of Pb (II) ion from 10 mg/L concentrated aqueous solutions at pH 4 for 90 minutes contact time. 20 g/L of CPU effectively removed 85 % of Pb (II) ion from 10 mg / L concentrated aqueous solutions at pH 4 for 120 min contact time.Adsorption data fitted with Langmuir and Freundlich isotherm model. Based on R^2 value, Freundlich isotherm model fitted well than Langmuir adsorption isotherm model. The Langmuir isotherm parameters such as adsorption capacity and adsorption intensity of SPU on Pb (II) ion adsorption was obtained as 5.435 mg/g and 0.235 L/ mg respectively. The adsorption capacity and adsorption intensity of CPU on Pb (II) ion adsorption was obtained as 5.495 mg/g and 0.056 L/mg respectively. Kinetic modeling studies revealed that Pb (II) ion adsorption process onto SPU and CPU followed pseudo second order kinetics. Chemically Modified PU can be utilized as a cheap adsorbent for Pb (II) ion removal for waste water.

Table 4. Maximum Pb (II) ion adsorption in adsorption-desorption cycles by SPU and CPU

Adsorbent	Adsorption- desorption cycles				
	1	2	3	4	5
SPU	93	89	86	82	78
CPU	84	81	78	73	68

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Povzetek

Zaradi vsesplošne industrializacije predstavlja onesnaženje s težkimi kovinami resno grožnjo vsem živim bitjem. V tej študiji smo preučevali adsorpcijo svinčevih (II) ionov na modificiran poliuretan. Poliuretan (PU) smo kemijsko modificirali s sulfonacijo in kloriranjem, s čimer smo pridobili sulfoniran PU (SPU) in kloriran PU (CPU). Pri obeh adsorbentih smo optimirali parametre adsorpcije kot so pH vrednost, kontaktni čas, količina adsorbenta in začetna koncentracija kovinskih ionov. Pod optimalnimi pogoji smo dosegli 90% in 85% adsorpcijo ionov na SPU oziroma CPU. Ravnotežni rezultati adsorpcije so pokazali, da gre za večplastno adsorpcijo, ki jo lahko opišemo s Freundlich-ovo izotermo. Hitrost adsorpcije na oba nosilca je sledila kinetiki pseudo-drugega reda. Rezultati študije so pokazali, da lahko kemijsko modificiran PU učinkovito odstranjuje Pb (II) ione iz odplak.