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Synthesis, characterization, and performance of chitosan/nylon 6/polyurethane blend for the removal of chromium (VI) and lead (II) ions from aqueous solutions for enhanced kinetic adsorption studies

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ABSTRACT

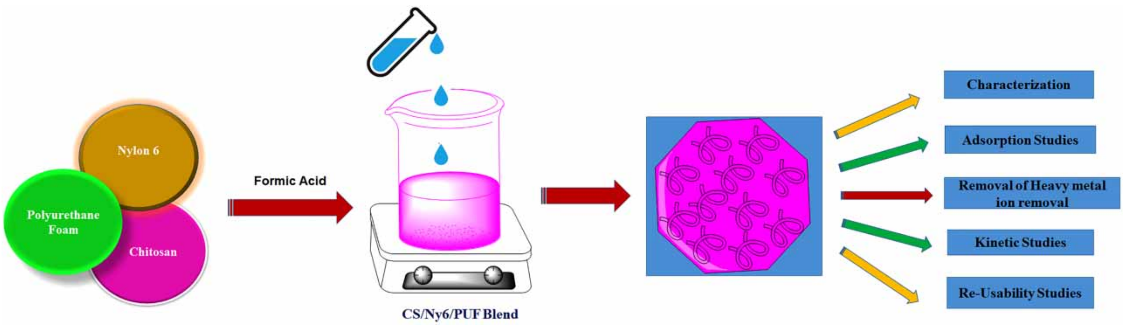
Adsorption is vital for the elimination of Cr^{6+} and Pb^{2+} ions in the contaminated solution medium. A ternary blend made up of chitosan, nylon 6 and polyurethane foam (CS/Ny 6/PUF) blend in the ratio of 2:1:1 has been investigated. These blends are used as an adsorbent due to the insoluble nature in acidic and basic medium. The adsorption efficacy was analyzed by modifying pH, contact time, and adsorbent dosage. The maximum uptake of metal ions has been exhibited in the pH range 5. An equilibrium adsorption statistic indicated that adsorption isotherm follows the Freundlich model. The adsorption kinetic parameters specified that the adsorption of chromium has shown pseudo-second-order and lead pseudo-first-order reaction.

Key words: chitosan, Freundlich, Langmuir, nylon 6, polyurethane foam

HIGHLIGHTS

- CS/NY 6/PUF blends adsorption efficacy was analyzed by modifying pH.
- Contact time, and adsorbent dosage.
- Langmuir and Freundlich adsorption isotherm was utilized.
- An adsorption harmony of metal particles was adsorbed by the adsorbent.
- The adsorption of chromium and lead ions exhibits pseudo-second-order and pseudo-first-order kinetic reaction.

GRAPHICAL ABSTRACT



INTRODUCTION

Water has a dominant role in all types of activities on Earth. However, human activity leads to causes of pollution. Freshwater is now in a dangerous condition and it is difficult to research this completely. Research is conducted worldwide to establish the management of various pollutants in companies' wastewater. A hazardous metal is also a type of poisonous pollutant. Some of the recognized noxious metallic elements are arsenic (As), iron (Fe), lead (Pb), chromium (Cr), cadmium (Cd), copper (Cu), nickel (Ni), and mercury (Hg). They are also toxic in nature and non-biodegradable with accumulation in living organisms (Ghorai *et al.* 2014). Stream pollution has received serious attention due to its augmented impact on different lifeforms. Recently, the removal of substantial amounts of metal particles from water bodies has received increased attention. The contaminated water can cause cancer and other harmful impacts for humans and the environment (Sekar *et al.* 2004; Al-Omair & El-Sharkawy 2007; Ren *et al.* 2021). Chromium and lead contamination in water bodies is mainly due to industries such as tanneries, textiles, metal processing, pigment, paints, batteries, and electroplating (Ong *et al.* 2007; Minisy *et al.* 2018). The non-bio decomposable nature of these heavy metal particles will cause a few destructive, intense and deadly impacts. Various techniques such as ion exchange, invert assimilation, adsorption, complexation, and precipitation have been utilized to dispose of harmful metals from streams. However, these techniques are not economic; subsequently some cost-effective adsorbents have been created for the removal of poisonous metal particles (Bayramoglu *et al.* 2005; Parthiban & Sudarsan 2021a, 2021b).

A blend is a mixture of monomers in the form of immiscible compounds and it possesses a hydrophilic nature and insolubility. Highly reactive sites are available which are utilized to remove heavy metal ions from the industrial effluent wastewater. This is mainly based on the nature of interaction of metal ions and blend (adsorbent). Chitosan has been prepared by the deacetylation of chitin. The polymer of glucosamine from oceanic biomass has been discovered to be a competent adsorbent for different heavy metal particles in the wastewater (Wan Ngah *et al.* 2002; Kandile *et al.* 2009; Rathore *et al.* 2020). Hydroxyl and amino functionalities of chitosan may be involved in chelation to trap the harmful metal particles (Guibal *et al.* 2002; Parthiban *et al.* 2022), proteins, and humic acid, among others. In order to enhance the adsorption capabilities of chitosan it can be physically or chemically modified. These modifications will improve pore size, mechanical properties, chemical inertness, hydrophilicity, and bioadaptability (Maruca *et al.* 1982; Wu *et al.* 2002; Sirshendu & Rekha Panda 2015).

Nylon 6 is the most crucial polymer with respect to the fiber business. The reagent e-caprolactam is an essential material for the process of polymerization. It is produced from exceedingly low-cost materials including cyclohexane, benzene, and phenol. The excessive cost of nylon 6 may be decreased by way of the introduction of blends with decreased cost for polymers, including polyolefins. Blend introduction is broadly taken into consideration as an economically feasible and flexible approach for enhancing residences or cost–benefit relationships in polymers without the need for synthesizing new polymers (Camila Alves de Rezende *et al.* 2006).

Polyurethane (PUF) has a wide range of physical and chemical properties and versatile applications, due to its augmented flexibility, great elasticity, hardness, and ability to withstand extreme pH and temperature conditions (Ma *et al.* 2012). Blends of polyamide have gained importance due to their limited pore size conveyance, enhanced mechanical properties and chemical inertness (Ibrahim 2010; Parthiban *et al.* 2019). However, it has low ligand compactness and its framework displays unreliable adsorption. To conquer these issues, it is blended with common macromolecules, such as chitosan and cellulose, which increment responsive destinations in the grid, and has been utilized successfully for the removal of contaminations from wastewater frameworks (Darko *et al.* 2012).

The present study was used to assess adsorption competence of Cr^{6+} and Pb^{2+} ionic particles on 2:1:1 blends of chitosan, nylon 6 and polyurethane foam (CS/Ny 6/PUF), by considering the influencing parameters such as contact period, dosage of adsorbent and pH. An adsorption isotherm (Langmuir and Freundlich) was utilized to contemplate the adsorption harmony of metal particles on the adsorbent. The kinetics of adsorption were analyzed and resolved quantitatively by pseudo first- and second-order equations.

MATERIALS AND METHODS

Deacetylated chitosan (92%) was purchased from Sea Foods in India. Nylon 6 (DuPont) and polyurethane was bought from Star Foams, India. AR grade SD Fine Chemicals were used for the other purposes. The whole reaction was done using double distilled water.

Preparation of polymer blend

1 g each of chitosan, nylon 6 and polyurethane foam was liquefied in HCOOH (formic acid) independently. The polymeric liquids were homogenized in the ratio of 2:1:1 mass proportion and with constant stirring for 1 h. After the mixing was completed, the obtained solution was dispensed on to Petri dishes. The samples were vacuum dried to eliminate any traces of solvent as portrayed in our past work (Jayakumar & Sudha 2013). The samples were labeled for further uses.

Adsorption studies

The sorption capacity of CS/Ny 6/PUF (2:1:1) blends was determined by adding adsorbent dose of 1 g to 100 mL solution of Cr^{6+} and Pb^{2+} ions with differing strengths ranging from 10 to 200 ppm prepared from 200 mg/L stock solution of potassium dichromate and lead nitrate respectively. Introductory adsorption tests indicated that this time frame was sufficient to guarantee harmony among adsorbed and unadsorbed metal particles. After equilibrium, the concentration of metal was found using an atomic adsorption spectrophotometer.

RESULTS AND DISCUSSION

Effect of pH

The adsorption process's impact on pH has a remarkable influence on the acceptance of substantial quantities of metal particles; meanwhile it resolves charges on the adsorbent surface and specification of adsorbate (Zhang & Bai 2003). The level of Cr and Pb ions eliminated by adsorption increases with pH and it attains optimum adsorption at pH 5.0 and then decreases through additional increments in pH up to 8.0. (Figure 1).

The acid–base properties of blends give clear information on changes in the adsorption performance with the solution. Equations (1) and (2) show some significant reactions that may occur during the adsorption process.

$$H^{+} + R - NH_2 \rightarrow H(R - NH_2)^{+} \quad (1)$$

$$M^{2+} + R - NH_2 \rightarrow M(R - NH_2)^{2+} \quad (2)$$

When the pH is lower, the amine functionalities of the blend are effectively protonated (Equation (1)) which contend with Cr^{6+} and Pb^{2+} for sorption sites resulting in minimal uptake of metal ions because of repulsion of ions. Conversely, as the pH builds, negative charge is developed and sites become accessible for the uptake of metal ions (Equation (2)). At greater pH, the adsorption diminishes because of decreased dissolvability and precipitation of both ions (Cr^{6+} and Pb^{2+}) occurs (Wan *et al.* 2010; Parthiban & Sudarsan 2021b). This kind of interaction has been clearly explained: initially as the pH increases the removal tendency of ions is also enhanced followed by decreases. This process mainly occurs because of increases in electrostatic interaction of ions and adsorbent.

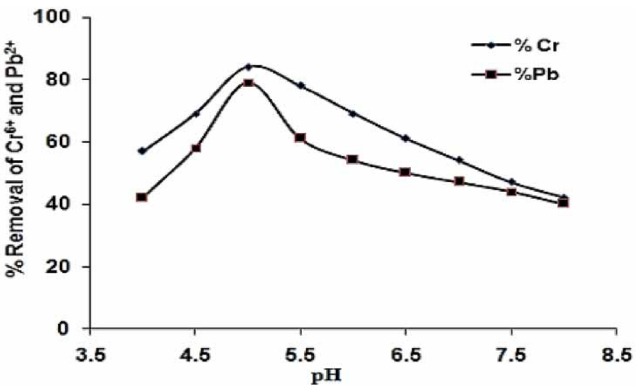


Figure 1 | Impact of pH on the adsorption of Cr(VI) and Pb(II) ions onto a 2:1:1 mixture of CS/Ny 6/PUF blend.

Effect of adsorbent dose

The outcome of the adsorbent also depends on how the materials' properties interact within an ionic solution. The reliance of uptake of Cr^{6+} and Pb^{2+} ions by 2:1:1 ternary blend was observed at optimum pH 5.0 with adsorbent dosage ranging from 1 to 6 g. It is evident that the percentage removal of Cr^{6+} and Pb^{2+} increases with expanding dosage of adsorbent (Figure 2) due to the accessibility of more adsorption sites. The blend possesses more cavities and holes. Hence, the graph clearly indicates that as the adsorbent dosage is increased removal percentage is also enhanced and this is due to the nature of ionic interaction that takes place gradually. The obtained results show that the percentage removals of Cr and Pb are 89 and 81, respectively (Habiba *et al.* 2017; Rosli *et al.* 2022).

Effect of contact time

The influence of contact time has been observed from 0 to 400 minutes. Adsorption proficiency of 2:1:1 ternary blend with time was observed for Cr^{6+} and Pb^{2+} ions at a constant adsorbent amount (1 g) and pH 5. Figure 3 indicates that the uptake of Cr^{6+} and Pb^{2+} ions increases with increased contact period. Then the equilibrium was accomplished after 300 min. The underlying quick period of adsorption is because of the accessibility of huge quantities of vacant adsorption sites on the adsorbent (Parthiban *et al.* 2020). After all the sorption sites were occupied by Cr^{6+} and Pb^{2+} ions adsorption attains saturation which causes a reduction in the rate after 300 min. The outcome of the investigation has shown that the ideal contact period for the greatest percentage removal of Cr (91.9%) was 300 min and for Pb (82.1%) it was 360 min. The adsorption of Cr was more fruitful than Pb ions at increased contact times due to the smaller ionic radii of Cr^{6+} ions (0.58 Å) than

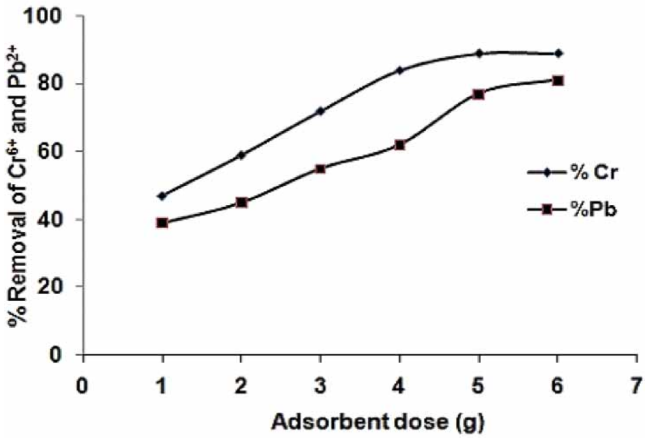


Figure 2 | Impact of adsorbent dose on the adsorption of Cr(VI) and Pb(II) ions onto a 2:1:1 mixture of CS/Ny 6/PUF blend.

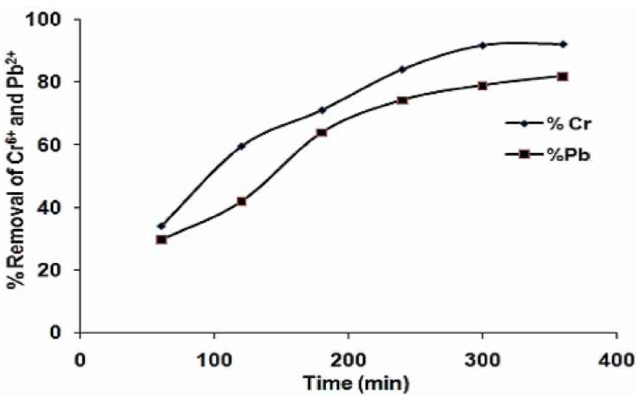


Figure 3 | Impact of contact time on the adsorption of Cr(VI) and Pb(II) ions onto a 2:1:1 mixture of CS/Ny 6/PUF blend.

Pb^{2+} ions (1.33 Å). Nevertheless, when the reaction contact time was increased, the present active sites or holes were saturated and resulted in regular uptake (Habiba *et al.* 2017; Koushkbaghi *et al.* 2017).

Adsorption isotherms

To evaluate the interaction of the adsorption sites, assumptions about the removal mechanism and the nature of the surface of the adsorbents have been explained by the Langmuir and Freundlich adsorption isotherm model and their constraints. Inter- and intra- molecular interaction performance of adsorbent with adsorbate has been studied by adsorption isotherms, which provide one of the most valuable pieces of information to comprehend the adsorption mechanism. Different adsorption isotherms have been employed to comprehend the interaction between adsorbate and adsorbent, among which the Langmuir and Freundlich isotherms were utilized for metal ion sorption studies (Swayampakula *et al.* 2009; Parthiban & Sudarsan 2021b). The Langmuir model is formed as a linear equation:

$$\frac{C_e}{q_e} = \frac{1}{K_L q_m} + \frac{C_e}{q_m} \tag{3}$$

where q_e is adsorption capacity, C_e is adsorbate concentration at equilibrium, and K_L is a Langmuir constant. The q_m and K_L values were acquired by plotting C_e/q_e vs. C_e. The sorption has been noted as the following Freundlich empirical equation,

$$\log q_e = \log K_F + \left(\frac{1}{n}\right) \log C_e \tag{4}$$

where K_F (sorption capacity) and n (sorption intensity) are resolved by log q_e against log C_e. The above adsorption isotherm has been confirmed for the synthesized polymeric blends suitable for elimination of different toxic metal ions from the wastewater and well fitted for the kinetic parameters.

The linearized Langmuir and Freundlich isotherms of Cr and Pb are shown in Figures 4 and 5, respectively. The estimated isotherm parameters with relationship coefficient (R^2) are shown in Table 1, indicating the pertinence of the isotherm model best fit to the Freundlich model for both lead and chromium.

Both the Langmuir adsorption isotherm and Freundlich adsorption isotherms fit the model impeccably. This is due to the performance of the heterogeneous blend adsorbents. Every adsorbed metal ion has followed various adsorption isotherms. This overall effect cannot be elucidated by a single Langmuir or Freundlich model (Weber *et al.* 1992; Olu-Owolabi *et al.* 2014). The comparison of room temperature adsorption capacity for the CS/Ny 6/PUF blend with different adsorbents reported in the literature is shown in Table 2.

Kinetic study of adsorption

The kinetic reaction of adsorption of Cr^{6+} and Pb^{2+} ions of the prepared ternary blend was studied using Lagergren first-order (Harijan & Chandra 2016; Parthiban & Sudarsan 2021b) and second-order equations. The linearized kinetic

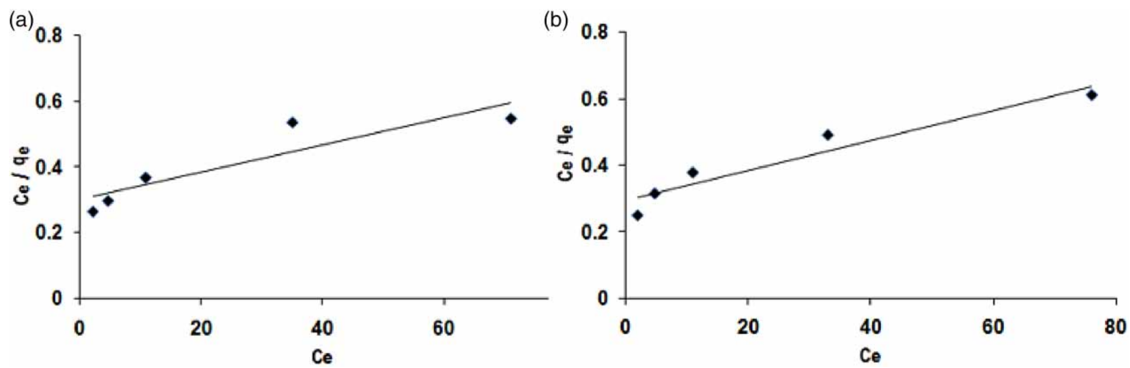


Figure 4 | Langmuir plot for (a) Cr(VI) ions and (b) Pb(II) ions.

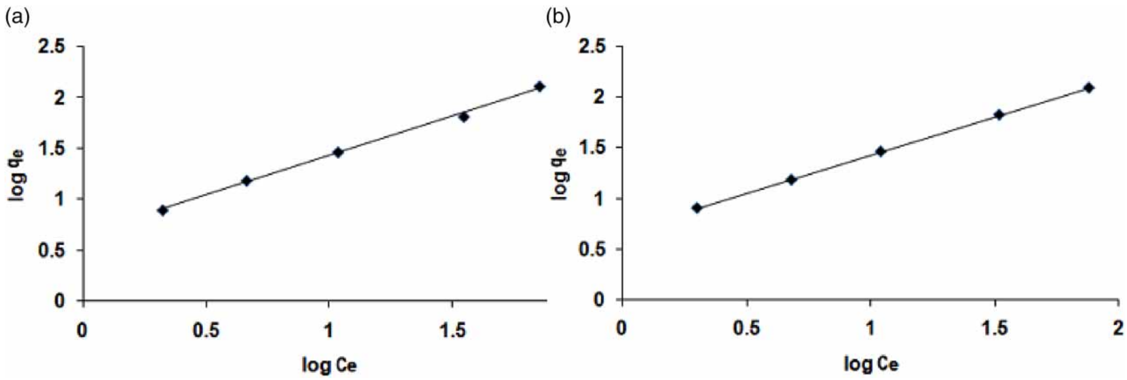


Figure 5 | Freundlich plot for (a) Cr(VI) ions and (b) Pb(II) ions.

Table 1 | Cr(VI) and Pb(II) isotherm constants for a 2:1:1 CS/Ny 6/PUF blend of ions

	Freundlich			Langmuir		
Metal ions	K_F	$1/n$	R^2	K_L	Q_O (mg/g)	R^2
Chromium (Cr^{6+})	4.5604	0.771	0.997	0.0132	250	0.809
Lead (Pb^{2+})	4.6989	0.756	0.999	0.6742	223	0.914

Table 2 | Comparison of room temperature adsorption capacity for the CS/Ny 6/PUF blend with different adsorbents reported in the literature

Adsorbents	Target heavy metal ions	Adsorption capacity (mg/g)	Reference
CS/PVA	Pb^{2+}	166.34	Rosli *et al.* (2022)
CS/PVA/Zeolite	Cr^{6+}	117	Habiba *et al.* (2017)
Fe_3O_4@PANI/IA MNCs	Cr^{6+}	218	Parthiban & Sudarsan (2021a)
CS/SA/PVA particles	Pb^{2+}, Cu^{2+}	39.28 26.03	Dong & Xiao (2017)
CS/Ny 6/PUF blend	Cr^{6+}, Pb^{2+}	250 223	Present study

PVA, polyvinyl alcohol; Fe_3O_4@PANI/IA MNCs, Fe_3O_4@Polyaniline/Itaconic acid magnetic nanocomposite; SA, sodium alginate.

equation for first-order is given by:

$$\log(q_e - q_t) = \log q_e - k_1 t/2.303 \tag{5}$$

where $k_{1,}$ and qe are the rate constant and uptake competency at equilibrium and q_t is the quantity of metal uptake at a certain time t. The kinetic parameters are acquired by plotting log $(q_e - q_t)$ against t. In addition, second-order kinetic parameters are determined by using the equation based on the adsorption equilibrium,

$$\frac{t}{q_t} = \left(\frac{1}{K_2 q_e^2}\right) + \frac{t}{q_e} \tag{6}$$

where K_2 is a second-order rate constant. By plotting t/q_t vs. time the kinetic parameters of adsorption can be obtained. Pseudo-first-order and pseudo-second-order kinetic plots are depicted in Figures 6(a) and 6(b) and 7(a) and 7(b), respectively. The acquired outcomes are summed up in Table 3. The outcomes demonstrated that the coefficient 'R' of Cr^{6+} ion fits better

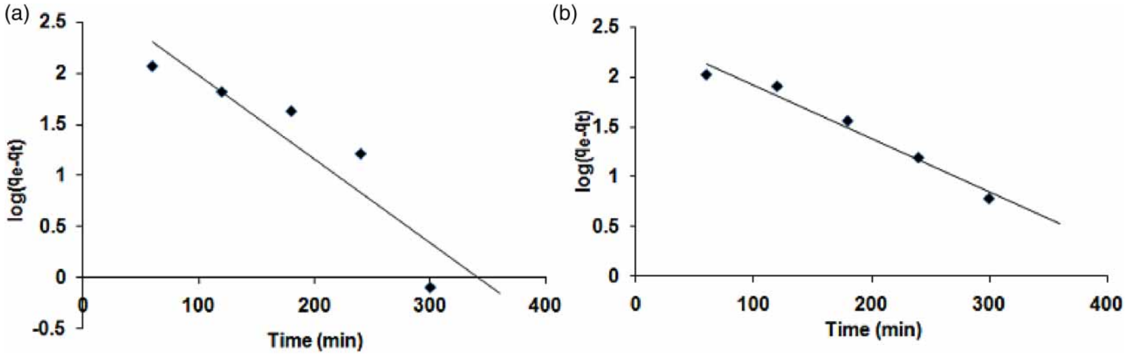


Figure 6 | Pseudo-first-order plot for the adsorption of (a) Cr^{6+} onto 2:1:1 CS/ Ny 6/PUF blend and (b) Pb^{2+} ions onto 2:1:1 CS/ Ny 6/PUF blend.

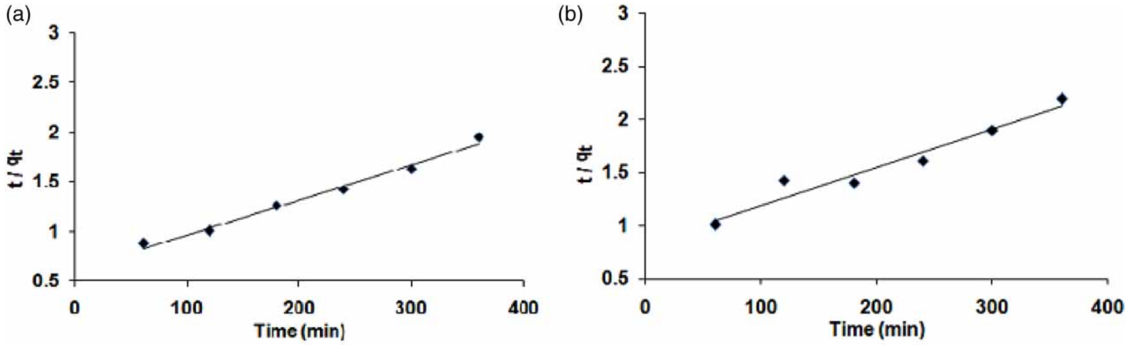


Figure 7 | Pseudo-second-order plot for the adsorption of (a) Cr^{6+} onto 2:1:1 CS/ Ny 6/PUF blend and (b) Pb^{2+} ions onto 2:1:1 CS/ Ny 6/PUF blend.

Table 3 | Comparison of kinetic models for the adsorption of Cr (VI) and Pb (II) ions onto a 2:1:1 mixture of CS, Ny 6, and PUF

	Pseudo-first-order			Experimental	Pseudo-second-order		
Metal ions	q_e (mg/g)	K_1 (l/min)	R^2	q_e (mg/g)	q_e (mg/g)	K_2 (g/mg min)	R^2
Chromium (Cr^{6+})	633.87	0.0184	0.834	184.3	333.33	1.45×10^{-5}	0.980
Lead (Pb^{2+})	280.5	0.0115	0.970	164.2	333.33	1.07×10^{-5}	0.945

into pseudo-second-order (0.986) than pseudo-first-order (834) indicating a chemisorption process (Li *et al.* 2013; Dong & Xiao 2017), whereas for Pb^{2+} pseudo-first-order (0.970) is better than pseudo-second-order model (0.947).

Based on the calculated correlation coefficients, the adsorption of Cr^{6+} and Pb^{2+} ions follows second-order kinetics rather than first order. The correlation equation has been established for the adsorption behavior and it is because of the interaction of adsorbent (blend) and metal ions in the wastewater.

Adsorption experiment of Cr (VI) and Pb (II) metal ion samples from industrial wastewater

Industrial wastewater was tested by prepared CS/Ny 6/PUF blend used as an adsorbent for 3 hours through the batch adsorption experimental technique. Figure 8 indicates that the removal of Cr (VI) and Pb (II) metal ions was 91.9% and 82.1% respectively. The Cr (VI) and Pb (II) metal ion-containing wastewater was treated by CS/Ny 6/PUF blend (adsorbent) and before and after parameters are shown in Table 4 (Dong & Xiao 2017).

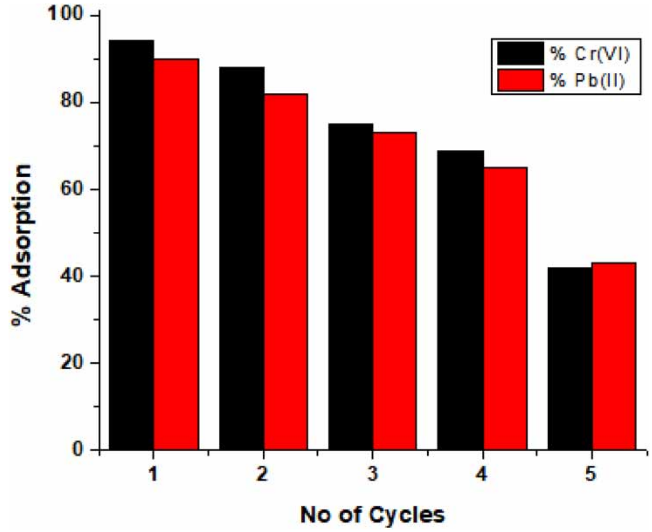


Figure 8 | Reusability of CS/Ny 6/PUF blend.

Table 4 | Mixture used to reduce the levels of Cr (VI) and Pb (II) metal ions in wastewater samples before and after treatment

S. No	Constituents in wastewater	Before treatment	After treatment	
			Cr (VI)	Pb (II)
1.	pH	8–10	6.5–7.0	6.2–6.8
2.	Total dissolved solids (ppm)	3,600	900	1,000
3.	Total suspended solids (mg/L)	450	<72	<78
4.	Biological oxygen demand (mg/L)	950	<45	<50
5.	Chemical oxygen demand (mg/L)	2,000	<150	<165
6.	Heavy metal (ppm)	100	<1	<2

Removal mechanism of Cr (VI) and Pb (II) metal ion samples from industrial wastewater

The Cr (VI) and Pb (II) hazardous metal ions were eliminated from the industrial wastewater and the adsorption mechanism was also analyzed using different pH solutions. As the pH range increases from 8 to 10, the percentage of Cr (VI) and Pb (II) metal ion adsorption also progressively increases. The highest adsorption was displayed at pH 10. At lower pH, weak Van der Waals force of attraction leads to an increase in the porosity of the structure that causes the precipitation of metal ions, whereas, strong affinity of metal ions has been observed at higher pH range, which is due to the strong electrostatic attraction in a medium of acidic pH. The effect of the CS/Ny 6/PUF blend has been observed as a definite removal of Cr (VI) and Pb (II) metal ions from the aqueous solution (Habiba *et al.* 2017).

Reusability studies

The recovery of adsorbent is more important for reusability studies. It can be reused more than three times without loss of efficiency. Figure 8 shows the absorption, the above problems, the CS/Ny 6/PUF adsorbent blend was recovered and reused for further recovery studies. Different concentrations of desorption chemicals were used for desorption or reusability studies such as acidic, basic and neutral medium (Rosli *et al.* 2022). The CS/Ny 6/PUF blend (1 g) was occupied by chromium and lead particles in 50 mL samples at room temperature, an interaction period of 6 hours, pH 2, and agitation speed 300 rpm. Based on the above studies 92 and 90% of adsorbent was recovered under acidic media desorption process and reusability can also be adjusted at pH 2.0–4.0.

CONCLUSION

The present adsorption characteristics of Cr^{6+} and Pb^{2+} ions with the ratio of 2:1:1 of CS/Ny 6/PUF blends were studied at room temperature by batch process. The obtained outcome has been demonstrated in an ideal adsorption process at pH 5. The quantity of Cr^{6+} and Pb^{2+} ions adsorption has also been observed with different dosages of adsorbent and contact period. The removal percentage of Cr^{6+} is more prominent than that of Pb^{2+} ions because of the smaller ionic radii of Cr (0.58 Å) than Pb (1.33 Å) which rapidly diffuses through the adsorbent pores. The adsorption isotherms of both metal ions have been shown to best fit the Freundlich model, indicating the heterogeneity of sorption sites. The kinetic investigations have revealed that chromium ion follows the pseudo-second-order and lead follows the pseudo-first-order reaction. This present work confirmed an adsorbent blend to treat different types of industrial wastewater containing multiple toxic and heavy metals.

DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

CONFLICT OF INTEREST

The authors declare there is no conflict.

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