A new polysaccharide-based ion-exchange resin for industrial wastewater treatment

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Abstract: A new ion-exchange resin was obtained by incorporating a tripropylamine group into a tamarind polysaccharide resin (TTA). The TTA resin was characterized by FTIR, elemental analysis, and other physicochemical properties. The influence of pH, treatment time and resin concentration on the adsorption of metal ions from industrial wastewater was investigated. It was found that the obtained TTA resin effectively removes heavy metal ions in the following order: Fe^{2+}> Cu^{2+}> Zn^{2+}> Cd^{2+}> Pb^{2+}.

Keywords: industrial wastewater, tamarind, tripropylamine, ion-exchange resin, adsorption, flocculants.

Nowa żywica jonowymienna na bazie polisacharydów do oczyszczania ścieków przemysłowych

Streszczenie: Nową żywicę jonowymienną otrzymano poprzez wbudowanie grupy tripropyloaminowej do żywicy polisacharydowej tamaryndowca (TTA). Żywicę TTA scharakteryzowano za pomocą FTIR, analizy elementarnej i innych właściwości fizykochemicznych. Zbadano wpływ pH, czasu obróbki i stężenia żywicy na adsorpcję jonów metali ze ścieków przemysłowych. Stwierdzono, że żywica TTA skutecznie usuwa jony metali ciężkich w następującej kolejności: Fe^{2+}>Cu^{2+}>Zn^{2+}>Cd^{2+}>Pb^{2+}.

Słowa kluczowe: ścieki przemysłowe, tamaryndowiec, tripropyloamina, żywica jonowymienna, adsorpcja, flokulanty.

In India ground water is used for domestic as well as agricultural purposes. Heavy metals ions such as iron, cobalt, cadmium, lead, mercury, chromium, selenium, arsenic, copper and zinc are invariably present in groundwater.These metal ions are considered to be toxins when they enter inside the body exceeding the prescribed limit, wherein they start causing illness [1–3]. Some of the heavy metal ions such as iron, cobalt, cadmium, lead, mercury, chromium, selenium and arsenic were found in industrial effluent of Okhla industrial area phase-II of Delhi NCR Region, India and because of this the groundwater of nearby areas is also contaminated [4]. Heavy metal ions isolated from the industrial effluents like Fe^{2+}, Cu^{2+}, Zn^{2+}, Cd^{2+}, Pb^{2+} have extensive applications in electronic appliances such as LED, Mobile phones, and other devices due to their chemical, electrical, optical and magnetic values [5]. Growing demand for reduction of metal ions in water has enhanced the strive for development of feasible ways to remove harmful metal ions from industrial contaminated water. Environmentally friendly adsorbents have been investigated for preferential adsorption of metal ions in multivariant [6].The main techniques for the recovery of these heavy metal ions from the industrial effluents involve co-precipitation [7], ion exchange [8], solvent extraction [9], membrane separation methods [10] and adsorption [11]. Out of these techniques, ion exchange technique has found wide application because of low operation cost, less generation of pollutants and high recovery rate [12, 13].

Nowadays, ion exchange resins, with the performance based on ion exchange technique find many applications in waste water treatment for the removal of heavy metal ions as they have greater selectivity and higher exchange properties [14]. For effective removal of trace heavy metal ions from the waste water of metal processing industry, a selective polymeric precipitant is widely used [15]. Polymeric ion exchange resin e.g. Tamarind based resin

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have dual property for they act as flocculants as well as selective binders [16]. Extensive work has been done for the development of new hydrophilic polysaccharide resins and exploring their application in water decontamination [17]. Nada et. al. [18] has reported the spectroscopic and cation exchange studies on phosphorylated cotton linters. In addition to Tamarind, other polysaccharide like wood flour [19], coconut coir dust [20] and various agricultural wastes [21] are also used for removal of heavy metal ions from waste water to control water pollution. Kong et. al. [22] have synthesized the hydrophilic poly(ionic liquid) that shows remarkable potential in water treatment for the effective separation of organic dyes or heavy metal ions like methyl blue and Cr^{6+}.

In this article, a cation exchange resin is used for wastewater treatment but its applications as drug carrier has also been reported by various co-workers [23-25]. The synthesis, characterization of new tamarind-tripropylamine resin (TTA) and its use as a toxic metal ions scavenger for industrial effluents is reported. In comparison with other conventional resins the metal selective and hydrophilic TTA resin has the advantage of being low cost, non toxic, biocompatible, regenerative and highly efficient in the effluent water treatment and environment friendly [26–28]. The polysaccharide modified ion exchange resin based on tamarind kernel powder (TKP) is hydrophilic and biodegradable in comparison to ion exchangers prepared from commonly available sources like petrochemical products [29] that are hydrophobic and non-biodegradable which constrain their usage. Rising prices of petroleum products and other limiting factors has been the lead factor for development of TTA resin as the product of choice. The low cost of TKP and its local availability in large quantities from agricultural resources makes the usage of the biopolymer economically sustainable. The TTA resin was characterized by FT-IR, elemental analysis, thermogravimetric analysis (TGA), distribution coefficient and degree of swelling. Several experiments on the effluent from Okhla Steel industry Pvt Limited have established the effectiveness of the proposed polymeric TTA ion exchange resin.

EXPERIMENTAL PART

Synthesis of TTA resin

Synthesis of TTA resin *via* epichlorohydrin was carried out using the method described earlier [30]. However, Porath's method [31] was used for functionalization of polysaccharide. The complete scheme for the synthesis of TTA resin is shown in Figure 1. The TTA resin synthesized by above mentioned procedure was obtained in good yield (80%) in the form of off-white crystalline solid as shown in Figure 2.

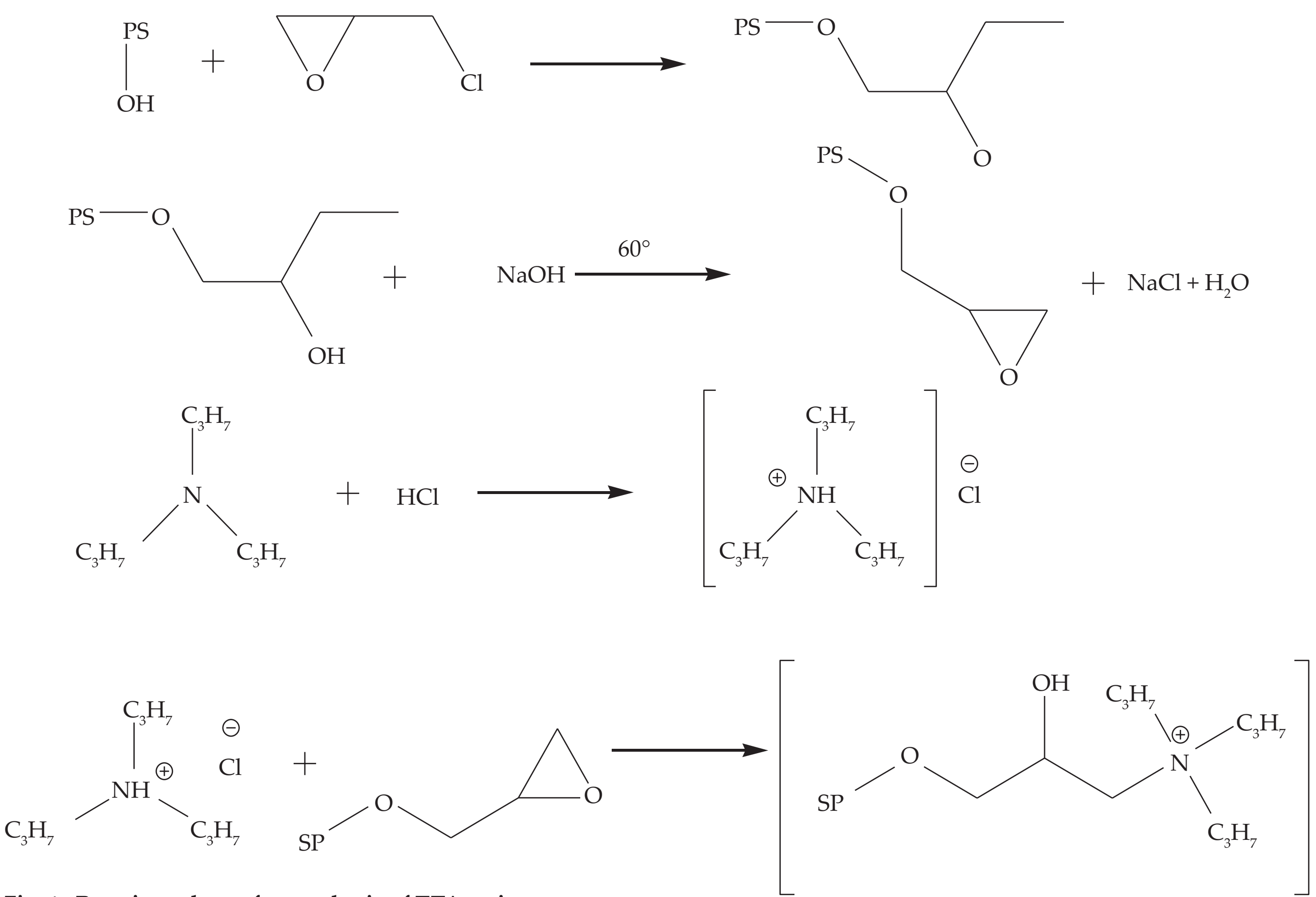

Fig. 1. Reaction scheme for synthesis of TTA resin

Fig. 2. TTA resin

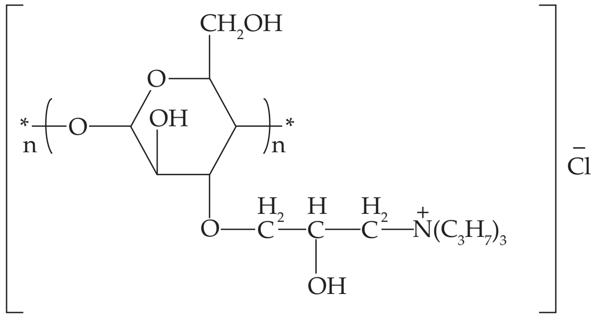

Fig. 3. Chemical structure of TTA resin

The efficiency of heavy metal ion removal from industrial wastewater with the TTA resin was tested with a glass column filled with 8.5 g (9 cm) of resin (internal diameter 1.6 cm and height 20 cm). 50 ml of a reference metal ion solution was passed through the column. The flow rate of the reference metal ion solution was controlled with a peristaltic pump (2 ml/min). To remove physically adsorbed metal ions (if present), the column was washed with deionized distilled water. Then different concentrations of hydrochloric acid and nitric acid were passed through which act as eluent. An atomic absorption spectrophotometer (AAS) was used to determine the concentration of metal ions present in the starting solution and the wastewater.

Determination of percentage removal of metal ions

100 ml of the different metal ions solution were taken in conical flask. 0.4 g of TTA resin was added to each flask. The stirring was carried out for 2 hours and then kept undisturbed for 1 hour. Then, the solution was filtered and the concentration of metal ion in the filtrate was determined by AAS. The percent removal of metal ions by TTA resin was calculated using this Equation (1):

$$\%\text{Removal} = \frac{\text{Initial Conc.} - \text{Final Conc.}}{\text{Initial Conc.}} \cdot 100\% \quad (1)$$

Determination of distribution coefficient (K_d)

The Batch method was used for the determination of K_d (Distribution Coefficient) of metal ions adsorbed on TTA resin. For this purpose, in a conical flask 20 ml of reference solution were taken in each of the cases. For the adjustment of pH, acidic and basic buffer solutions were used. To this solution TTA resin (20 mg) was added. It was stirred using magnetic stirrer for 2 hrs.(approximately) so that the contents were equilibrated and then this solution was filtered using Whatman filter paper No.42.The residue left over the filter paper was equilibrated with 4N HCl. The resulting solution was filtered using Whatman filter paper No.42. Atomic absorption spectrophotometer (AAS) was used to estimate the metal ions concentration present in residue and filtrate. After analysing a series of metal ions (using standard solutions) with the help of AAS, calibration curves for different metal ions were plotted. Various metal ions were estimated by different wavelengths of main resonance line and air acetylene flame was used. Determination of metal ions concentration in filtrate was carried out using calibration curves. Distribution coefficient of metal ions on TTA resin was calculated with the help of Equation (2) given below [32]:

$$K_d = \frac{I - F}{F} \cdot \frac{V}{M} \, ml \cdot g^{-1} \quad (2)$$

where:
I – initial amount of the metal ion in solution,
F – final amount of metal ion after equilibrium with resin,
V – volume of metal ion solution (ml),
M – molecular weight of the resin taken in grams.

Methods

For the synthesis of TTA resin a magnetic stirrer from Sisco, Delhi, India, was used. For C, H, N determination of synthesised TTA resin Carlo Erba element analyser model 1160 was used. IR of the synthesized TTA resin was determined by IRAffinity-1S Shimazu FT-IR instrument using KBr pellets and for thermal stability thermogravimetric analyser (TGA model 951) was used. Determination of pH of each stock solution of different metal ions was done using pH meter (Beckman Model 335). Atomic absorption spectrophotometer (AAS) model was used for the study of trace metal ions analysis.

RESULTS AND DISCUSSION

Elemental analysis

The results obtained from the elemental analysis of the synthesized TTA resin also supported the proposed structure of the resin as in Figures 1–3. The results of elemental analysis are given in Table 1.

Thermo gravimetric analysis (TGA)

For the measurement of TGA of TTA resin, the system was heated at a rate of 20°C/min under static air atmosphere. It was heated until the complete decomposition

T a b l e 1. Elemental analysis data of TTA resin

Element	Theoretical values, %	Experimental values, %
C	54.33	54.62
H	9.21	9.09
Cl	8.91	8.68
N	3.52	3.26
O	24.12	24.42

took place. This TTA resin is stable up to 280°C and then it starts degrading. Initial rate of degradation was slow but after 410°C it decomposed rapidly as displayed in Figure 4.

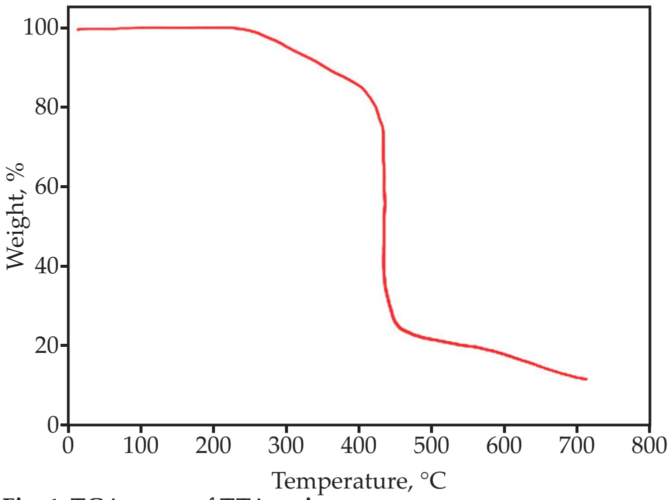

Fig. 4. TGA curve of TTA resin

T a b l e 2. Assignment of various signals in IR spectrum of TTA resin

Wavenumber, cm^{-1}	Assignment
3400	-O-H Stretching Vibration
3050	-C-H Stretching Vibration
1610	-C-C- stretching vibration
1500	-C-H bending vibration
1340	-C-N Asymmetric Stretching
1250	-C-N Symmetric Stretching
1150	-C-O-C- Stretching Vibration

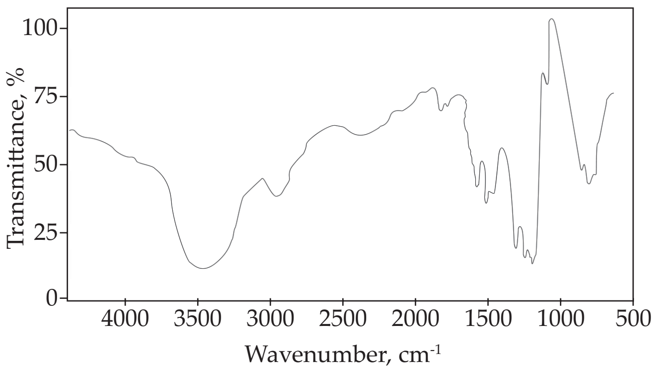

Fig. 5. FTIR spectrum of TTA resin

IR characterization

The complete assignments of various signals due to vibrations of different functional groups as shown in Figure 5 and due to skeletal vibrations are given in Table 2 that confirms the synthesis of TTA resin.

Swelling studies

The cross linked TTA resin was taken in a burette and packed gently. The resin was then equilibrated with solvent for 1 hour. The degree of the resin swelling can be calculated by measuring the relative increase in resin column height which is highly dependent on degree of the resin cross linking. The degree of different samples swelling of cross linked TTA resin is given in Table 3. The swollen resin was collected by filtration; adhering traces of water were removed by filter pressing and the sample was weighed. The swollen resin was dried in a vacuum for 24 h and weighed again. The following Equation (3) was used to determine the equilibrium water content (*EWC*):

$$EWC = \frac{\text{Weight of wet resin} - \text{Weight of dry resin}}{\text{Weight of wet resin}} \quad (3)$$

The equilibrium water content (*EWC*) of resin was 9.1%.

Effect of flow rate on removal of metal ions

The flow rate of the sample solution in the column affects the recovery of metal ions and also controls the

T a b l e 3. Swelling of different samples of cross linked TTA resin

Amount of reactants used for preparation of resin for 25 g of Tamarind			Swelling %		
NaOH g	Epichlorohydrin ml		Cold water	Hot water	10% NaOH sol.
	Before heating	After heating			
1.0	1.5	0.5	2.0	2.0	5.3
1.0	1.2	0.7	4.1	4.5	5.4
1.2	1.5	0.5	2.6	2.6	4.6
1.2	1.2	0.7	3.2	3.2	3.5

T a b l e 4. Removal of metal ions from industrial wastewater by TTA resin at various pH

pH	Pb^{2+}, %	Cd^{2+}, %	Zn^{2+}, %	Cu^{2+}, %	Fe^{2+}, %
2	46.09	61.24	64.44	67.12	71.18
3	52.46	67.57	72.98	75.84	76.69
4	62.55	72.81	77.15	80.08	83.67
5	72.31	81.65	83.71	87.49	88.54
6	86.56	91.46	92.11	94.77	96.39
7	53.38	71.35	74.88	77.84	83.42
8	41.32	47.21	53.08	57.74	62.07

T a b l e 5. Removal of heavy metal ions from different types of effluents using TTA resin

Metal ions	Type of effluents and concentration of various metal ions, ppm					
	Reference solution		Pond water		Underground industrial water	
	Initial	After treatment	Initial	After treatment	Initial	After treatment
Cu^{2+}	11.70	0.08	0.68	0.05	0.48	0.00
Cd^{2+}	20.47	0.07	0.43	0.04	0.32	0.02
Pb^{2+}	41.23	0.05	0.79	0.04	9.48	0.05
Zn^{2+}	12.50	0.05	0.78	0.03	20.82	0.04
Fe^{2+}	10.16	0.08	0.33	0.04	61.30	0.05
Hg^{2+}	9.68	0.04	–	–	–	–
Ag^{+}	5.82	0.04	–	–	–	–
Ca^{2+}	219.50	218.00	182.00	180.00	175.00	171.00
Mg^{2+}	117.00	117.00	62.00	62.00	81.00	80.00

time of analysis. Therefore it is an important parameter. The effect of flow rate on removal of metal ions was studied using optimum conditions of pH, eluent, etc. For this purpose, 100 ml of the sample solution were passed through the microcolumn. The flow rates were controlled by a peristaltic pump and adjusted in the range of 1.0–5.0 ml/min.

Effect of pH on adsorption of metal ions from industrial wastewater by TTA resin

The percent removal of metal ions from Okhla Steel Industry Pvt Limited (Delhi, India) wastewater was reported at different pH as given in Table 4. The pH is an important parameter for adsorption of metal ions from aqueous solution because it affects the solubility of the metal ions, the counter ions concentrations on the functional groups of the adsorbent and the ionization degree of the adsorbent during reaction. To examine the dependence of metal ions adsorption percentage on pH, the pH was varied from 2.0 to 9.0. It was observed that the optimum results were obtained at pH 6.0. The adsorption of different metal ions on TTA resin follows the order: $Fe^{2+} > Cu^{2+} > Zn^{2+} > Cd^{2+} > Pb^{2+}$. The variation of metal ions adsorption on polymeric TTA resin with *pH* of solution is given in Figure 6. This figure shows that adsorption of metal ions on TTA resin increases as the pH of solution changes from 2.0 to 6.0. This is partly due to the fact that hydrogen ions are strong competing adsorbents for the same binding sites and partly that the solution pH influences the chemical specifications of metal ions. Meanwhile the observed decrease in sorption at higher pH (greater than 6) is due to the formation of insoluble metal ions hydroxyl complexes. At pH greater than 9.0, precipitation of insoluble metal ions occurs that makes it difficult to study metal ion adsorption. Therefore, the solution is adjusted to such a pH, which is optimum for the adsorbent but low enough to prevent hydroxide from forming and high enough to ensure the capture of metal ions.

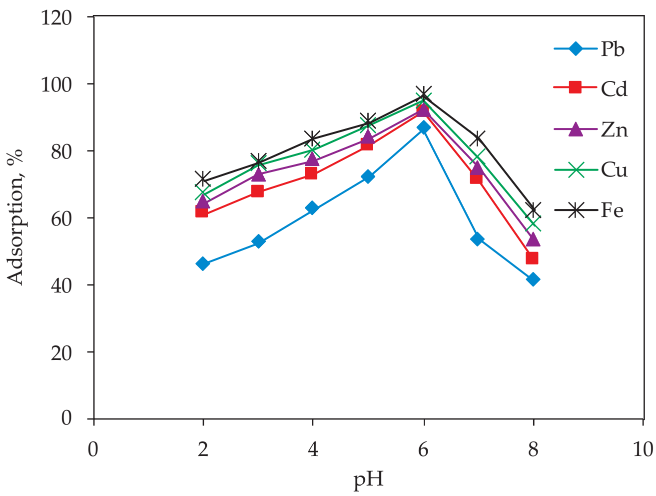

Fig. 6. Effect of pH on adsorption of metal ions on TTA resin

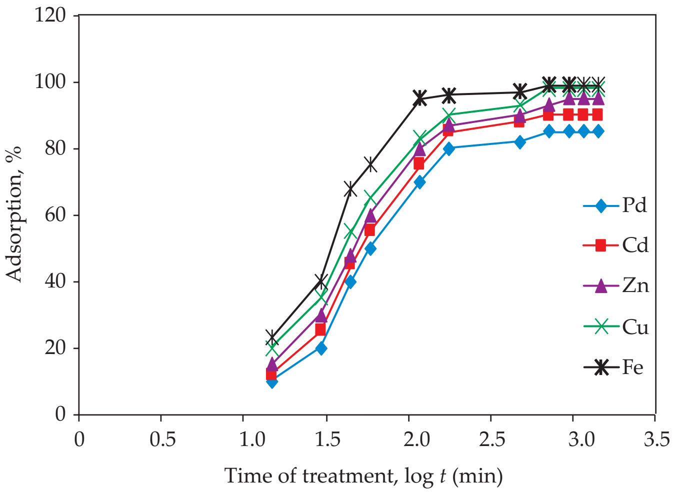

Fig. 7. Influence of treatment time on the adsorption of metal ions on TTA resin

Removal of metal ions from industrial wastewater

In order to compare results with natural effluent, a reference solution was prepared by mixing 100 ml solution of various metal ions having initial concentration as shown in Table 5 at pH 6.0 and then the solution was made up to one liter by adding water. The TTA resin was found to be very effective in removing heavy metal ions without the interface of Ca^{2+} and Mg^{2+} ion concentration (hardness) of treated water. TTA resin contains amine functional group, which possesses not only protons that can exchange with cations but also nitrogen atom that can co-ordinate directly with metal ions. Therefore, the adsorption ability of TTA resin for metal ions may be very strong. The bonds formed in this kind of metal adsorption have both covalent and ionic characteristics. This resin binds selectively with heavy metal ions and not with alkali and alkaline earth metals. This is due to their small size and high polarizing power in comparison to alkali and alkaline earth metal.

Influence of treatment time of TTA resin with effluent

Figure 7 shows the influence of treatment time of TTA resin on adsorption of metal ion. The influence of treatment time of TTA resin on metal ions adsorption was observed from 0 to 3.20 minutes keeping other parameters at optimum value. It was observed that on increasing the contact time of resin with effluent solution, a remarkable improvement occurs in binding capacity of resin because of the more sites availability for adsorption on the surface of TTA resin.

Influence of resin concentration on adsorption of metal ions

The influence of TTA resin concentration on the adsorption of metal ions is shown in Figure 8. The adsorption of metal ions increases initially with increase in resin concentration. This increase may be due to the availability of more sites for adsorption. The optimum adsorption by polymeric TTA resin was obtained at 100 mg of resin. After passing through the maxima, adsorption decreases on further increasing of the resin concentration. This may be due to the overlapping of adsorption sites on resin.

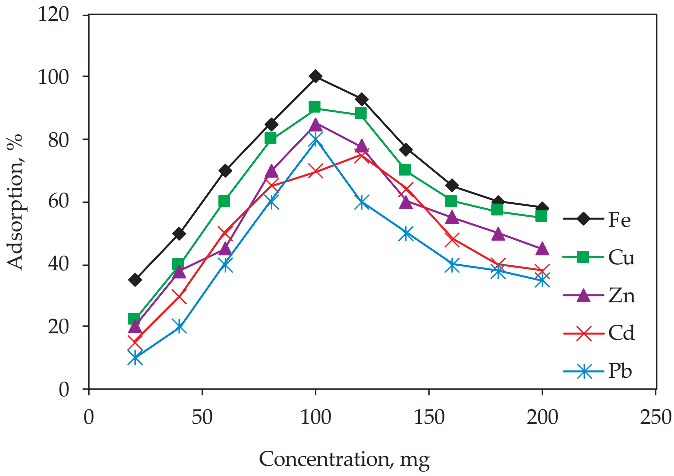

Fig. 8. Influence of resin concentration on adsorption of metal ions on TTA resin

Resin durability

It was observed that the adsorption of different metal ions on the TTA resin after 10 cycles (adsorption and desorption), and ion exchange capacities of resin, were almost constant. The adsorbed metal ions were easily desorbed by treatment with different strengths of acids, at room temperature. Table 6 shows the adsorption of different metal ions on the TTA resin after repetitive 10 cycles (adsorption and desorption).

T a b l e 6. The adsorption of different metal ions on TTA resin (adsorption and desorption

Metal ions	Adsorption for metal ions onto TTA resin after number of cycles , %				
	1 cycles	2 cycles	4 cycles	8 cycles	10 cycles
Cu^{2+}	94.05	94.76	92.10	92.10	92.01
Fe^{2+}	93.88	93.41	91.44	91.44	91.34
Zn^{2+}	93.50	92.20	91.23	91.23	91.24
Cd^{2+}	92.08	92.88	91.62	91.62	91.67
Pb^{2+}	91.10	89.15	88.31	88.31	88.38

T a b l e 7. Quantitative separation of metal ions on TTA resin column

Metal ion	Metal ion loaded mg	Metal ion found mg	Recover %	Eluent used	Eluent ml
Zn^{2+}	8.76	8.39	95.86	0.05 N HCl	50
Fe^{2+}	1.08	1.06	98.81	0.5 N HCl	55
Cu^{2+}	1.94	1.92	99.38	1.5 N HCl	50
Cd^{2+}	0.16	0.15	97.28	1 N HNO	45
Pb^{2+}	0.88	0.88	100.00	0.5 N HNO_3	40

Recovery of metal ions

The metal ions were eluted quantitatively with different strengths of acids. Zn^{2+} was eluted with 0.05 N HCl; Fe^{2+} with 0.5 N HCl; Cu^{2+} with 1.5 N HCl; Pb^{2+} with 0.5 N HNO_3 and Cd^{2+} was eluted with 1 N HNO_3. The resin column was then washed thoroughly with demineralized water. The reported results are given in Table 7.

CONCLUSIONS

The TTA resin was found to act as a promising adsorbent for the removal of various heavy metal ions from industry effluents, due to its low cost, less time requirement and eco-friendly procedure. The various factors like *pH* of the solution, time of contact, resin concentrations etc. were found to have significant effect on the adsorption of metal ions by TTA resin. Tripropylamine derivative of tamarind acts as flocculants *cum* metal ions exchanger that can be used as scavenger for toxic and hazardous metal ions from the effluent of industry. Generally, common ions exchangers are nonspecific and bind all metal ions (transition, alkaline and alkali) non selectively but the newly developed polymeric TTA resin was found to bind transition metal ions selectively and do not bind with alkali and alkaline earth metals ions. The adsorption of different metal ions on TTA resin follows the order: Fe^{2+}> Cu^{2+}> Zn^{2+}> Cd^{2+}>Pb^{2+}. The results of the study prove that the TTA resin can successfully be used for the selective separation of different metal ions. The resin has potential for removal of metal ions from industrial wastewater. The process is very efficient, especially in the case of low concentrations of pollutants in aqueous solutions, where other common methods are either economically unfavorable or technically complicated and not feasible.

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