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Preparation, Characterization of Tamarind Isopropyl amine Resin and It's Application in Industrial Wastewater Treatment

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ABSTRACT

The isopropyl amine group has been incorporated onto tamarind by a modified Porath's method of functionalization of polysaccharides. The tamarind isopropyl amine (TIPA) resin can selectively remove of lead and cadmium ions, which are contained in industrial wastewater. The TIPA resin was characterized by FTIR. The effects of various adsorption conditions, such as pH, treatment time, temperature, and adsorbent dose were also investigated. The orders of distribution coefficient values were determined.

Keywords: Tamarind Isopropyl amine Resins, Industrial Wastewater, Distribution Coefficient.

INTRODUCTION

Toxic metal ions are harmful metal ions, since they are not biodegradable and can be accumulated in nature. They also cause various diseases and disorder the ecosystem of our nature. Toxic metals such as, ferrous, copper, zinc, lead, etc are detected in waste streams from industries. It has a harmful effect on human physiology and other biological systems when they exceed the tolerance levels. Therefore, the need of development of technology that can not only treatment of wastewater but also recover metal ions from industrial wastewater. In recent years, the adsorption process has also achieved much attention and has become one of the most popular method for the removal of toxic metal ions from wastewater. The various adsorbents have been reported for removal of toxic metal ions, such as chitosan [1], polysaccharide [2-5], guaran [6], and cyclodextrin [7], which are not only eco-friendly and cost effective but are also effective intermediation of common effluents present in wastewater. Other polysaccharide-based materials and alumino silicates are used as adsorbents in wastewater treatment [8-10]. Adsorption using commercial activated carbon (CAC) can remove metal ions from wastewater, such as cadmium, nickel, chromium and copper [11-13]. Tamarind kernel powder is obtained from the seeds of the tamarind tree, Tamarindus Indica, a common forest and cultivated tree found primarily in India, Burma, Bangladesh and Sri Lanka. Tamarind powder is a polymer with an average molecular weight of 52,350 and a monomer containing three sugars, glucose, galactose and xylose in a molar ratio of 3:1:2, was reported by Khanna *et al*. [14]. Wastewater treatment is technologically simple and it is both economically advantageous and efficient. It may have many disadvantages, for e.g. if the metals are complexed, it requires an oxidation step, handling and disposal problems etc [15]. The main objective of this research work is to prepare tamarind based chelating resins for removal of heavy metal ions from industrial wastewater.

MATERIALS AND METHODS

Chemicals: Tamarind powder (Aces Chemical Works, Jodhpur, India), calcium carbonate (Aces Chemicals, India), Epichlorohydrin (Sisco Chem Industries, Mumbai, India), isopropyl amine (Sisco–Chem Industries, Mumbai, India), sodium hydroxide (Sarabhai Hydroxide Chemicals, Baroda, India), dioxane (E. Merk, Mumbai, India).

Sample: The effluent of Neelkanth Steel Industry, Jodhpur, Rajasthan (India) has the following characteristics features as summarized in table 1. Perkin-Elmer model 2380 Atomic Absorption Spectrophotometer used for quantitative determination of trace metals. For different metal ions standard wavelengths of main resonance line and air acetylene flame were used.

Sample of Effluents: Effluents containing heavy metal ions are collected from the Neelkanth Steel Industry. This sample contains heavy metal ison like Cu^{2+}, Pb^{2+}, Ni^{+2}, Cd^{+2}, Fe^{+2}, Co^{+2} along with the turbidity and pulp. The characteristics of Neelkanth Steel Industry sample are reported in table 1.

Table 1. The Characteristics of Neelkanth Steel Industry, Jodhpur

pH	4.5									
Appearance	Light Green									
Total Hardness	968									
Metal ion	Cu^{2+}	Zn^{2+}	Pb^{2+}	Cd^{2+}	Ni^{2+}	Mg^{2+}	Cr^{2+}	Ca^{2+}	Fe^{2+}	Co^{2+}
Concentration (ppm)	2.32	9.45	0.86	0.24	0.62	24.6	0.84	90.8	118	1.60
Others anions(ppm)	Fluoride=0.36; Sulphate=860; Cyanide=0.04									

Synthesis of Tamarind Isopropyl amine (TIPA) Resin: 32 g tamarind powder was taken in round bottom flask and slurred in dioxane. 50 % of aqueous solution of sodium hydroxide was added in the flask to make it alkaline, till pH reached to 9.5. The solution was stirred for 1h. 9.25 g (0.1 mol) epichlorohydrin was added drop wise and stirring was continued for 4 h at 60°C. The product epoxypropyl ether of tamarind was formed. Epoxy propyl ether of tamarind was allowed to react with 5.91 g (0.1 mol) of isopropyl amine in the alkaline medium and the stirring was continued for another 4 h at 60°C. The product was filtered under vacuum and washed with 90% methanol, containing few drops of hydrochloric acid to remove inorganic impurities. Finally, it was washed with pure methanol. The product tamarind isopropyl amine resin was free flowing light white powder. The yield was 62.5 g.

IR characterization: Perkin Elmer FTIR (model 5000, USA) Instrument was employed for FTIR spectra analysis of tamarind powder and functionalized tamarind derivative (TIPA resin). The FTIR spectra of tamarind powder shows broad band in the region 3600-3200 cm^{-1} characteristic -OH stretching frequency. The peak at 2929 cm^{-1} is attributed to C-H stretching vibrations. Another strong and sharp peak at 1650 cm^{-1} may be due to - OH bending. Another variable peak at 1480-1350 cm^{-1} is attributed to C-H bending. A strong peak at 1300-1000 cm^{-1} denotes C-O stretching vibration. A strong peak in the region 3000-2500 cm^{-1} denotes O-H stretching in group. A strong peak in the region 1250-1070 cm^{-1} denotes C-N stretching vibrations.

Thermogravimetric analysis: For this purpose, Thermogravimetric analyzer **(**Dupont 951, USA**)** was employed. The polymer sample was powdered to the same average mesh size and dried carefully in vacuum desiccator. The boat was packed uniformly for analysis. For the dynamic measurement, the system was heated at a constant heating rate of 20°C min^{-1} under static air atmosphere till the complete decomposition. The TIPA resin is found to stable up to 398°C and then the degradation was found to be rapid.

Removal of metal ions from effluent of Neelkanth Steel industry, Jodhpur, India: The results of percentage removal of metal ions from effluent of Neelkanth Steel Industry by TIPA resin are given in table 2. It is clear from the reported Table that the percentage removal of metal ions first increases and then deceases with increasing pH, the optimum results obtained at pH 6.0

Distribution coefficient (K_d) of metal ions: The pH has a strong effect on the distribution coefficient (K_d) of metal ions. The results of distribution coefficient

Table 2. Percentage removal of metal ions from the industrial effluent by TIPA Resin

pH	Pb (II)	Cd (II)	Zn (II)	Cu (II)	Fe (II)
2.0	42.16	58.32	62.48	65.56	74.82
3.0	54.68	69.48	73.38	72.49	81.12
4.0	59.98	75.34	79.93	83.92	85.64
5.0	71.40	82.87	84.72	91.37	94.96
6.0	88.35	92.64	95.13	95.96	98.12
7.0	56.22	71.57	72.92	80.62	83.14
8.0	44.29	52.76	54.26	59.75	67.28

(K_d) of metal ions from effluent of Neelkanth Steel Industry, Jodhpur are given in table 3. Metal sorption starts when the pH rises to the range where most acidic ion exchange sites start to exchange hydronium ion for metal and the capacity reaches the maximum value in the pH range where all the ion exchange sites take part in the reaction and the functional group is able to form complex with the metal cations.

Determination of distribution coefficient (K_d) of metal ions: The distribution coefficient (K_d) of metal ions was determined by batch method. The pH of the solution was adjusted to the desired value using acetate buffer and the resin was equilibrated for 4 h. A sample solution (100 mL) containing a known concentration of the studied metal ions were transferred to a Erlenmeyer flask and after adjusting its pH values, 0.1 g of the modified TIPA resin was added to the solution and the mixture was shaken continuously in a temperature controlled shaker at 25 ±2°C. The amounts of metal ions in the solution before and after equilibration were determined by using AAS. The distribution coefficient (K_d) of metal ions was calculated by the following equation.

$$K_d = \frac{\text{Amount of Metal ion in resin phase}}{\text{Amount of Metal ion in solution phase}} \ \text{X} \ \frac{\text{Volume of Solutions (mL)}}{\text{weight of dry resin}} \ \text{mL g}^{-1}$$

$$\text{i.e. } K_d = [(I - F / F) \text{ x } V/ M] \quad \text{mL g}^{-1}$$

Where *I* is the initial amount of the metal ion in solution, *F* is the final amount of metal ion after equilibrium with resin, *V* is the volume of metal ion solution (mL) and M is the weight of the resin taken (g).

Column operation: The column operation used for analyzed the recovery of metal ion. In the column experiment, a glass tube with 1.6 cm internal diameter and 20 cm height, packed with 9 cm of resin (8.5 g) was used. Separation of metal ions by selective elution on column was carried out for binary mixture. The flow rate was controlled by a peristaltic pump. The column followed by treating with distilled water to remove the last traces of unadsorbed ions. The solution mixture was passed through the column at a flow rate of 1mL min^{-1}. Elution was carried out at different concentration of hydrochloric acid solutions.

RESULTS AND DISCUSSION

Ion exchange capacity (IEC) of TIPA resin: It was found to be 2.47 meq g^{-1} of the dry TIPA resin.

Distribution coefficient (K_d) of metal ions: The pH has a strong effect on the distribution coefficient (K_d) of metal ions. The perusal of the results shown that the distribution coefficient value first increases and decreases with increasing pH, the optimum results were obtained.

Table 3. Distribution coefficient (K_d) of metal ions

pH	Pb (II)	Cd (II)	Zn (II)	Cu (II)	Fe (II)
2.0	09.14	26.32	20.28	24.83	25.38
3.0	12.46	28.74	24.84	35.38	46.52
4.0	16.87	31.18	37.25	36.44	62.65
5.0	28.46	68.28	55.89	98.18	186.34
6.0	82.12	112.12	149.94	246.28	750.75
7.0	20.48	28.67	32.38	41.24	50.79
8.0	10.56	12.17	14.72	18.94	20.84

Effect of pH: The pH is an important parameter for adsorption of metal ions from aqueous solution because it affects the solubility of the metal ions, concentration of the counter ions on the functional groups of the adsorbent and the degree of ionization of the adsorbent during reaction. To examine the adsorption % of metal ions with pH, the pH was varied from 2.0 to 8.0. The uptake of free metal ions depends on pH, where optimum adsorption of metal ions occurs at pH 6 and then declining at higher pH. Adsorption of metal ions on TIPA resin increased over pH range from 2.0 to 6.0.

Effect of TIPA dose on adsorption of metal ions: The adsorption of metal ions is significantly influenced by the amount of the TIPA resin added. The amount of TIPA resin added into the solution determined the number of binding sites available for the adsorption. The effect of the adsorbent dose on the amount of metal ions removed was studied by the application of varying TIPA doses. The maximum adsorption by TIPA resin was achieved with an adsorbent dose of 0.1 g and then constant unto 1.0 g. The initial increase adsorption percentage of metal ions was due to the availability of more adsorption sites. On increasing the TIPA resin concentration, further the binding of metal ions becomes constant. This effect might be attributed to overlapping or aggregation of adsorption sites of resin resulting in constant total surface area of the adsorbent.

APPLICATION

The prepared TIPA resin is applicable in removing heavy metal ions like Pb (II), Cd (II), Zn(II), Cu (II), Fe (II) from Industrial effluent.

CONCLUSION

The TIPA resin is applicable for the removal and recovery of heavy metal ions from industrial effluent.

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