

Synthesis and Application of New 2-Amino Ethyl Ether of Tamarind Kernel Powder as Ion-exchanger

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The effects of wastewater from Ankur Mineral Pvt. Ltd and Kothari Metal Industries were studied by sampling and analyzing for some physico-chemical properties between Jan '03 to June '03. The effluent from the industries is discharged to the local river and agriculture areas. Samples were collected before and after discharge point. It reveals that the farms and fields were polluted and the level of Cu^{2+} , Zn^{2+} , Pb^{2+} , Cd^{2+} , Mg^{2+} , Ca^{2+} , Fe^{2+} , CN^- , SO_4^{2-} were fairly high.

Tamarind kernel powder (TKP) powder obtained from seeds of tamarind plant as a hydrophilic polysaccharide matrix has been used for preparation of a new chelating resin. Ethanolamine derivative of TKP acts as a flocculent-cum-metal ion exchanger and can be used as scavenger for toxic and hazardous metal ions from the effluents of mineral and metallurgical industries. The ethanolamine group was anchored on TKP backbone in dioxane medium.

Key Words: Metal producing industries, Effluent, TKP (tamarind kernel powder), Polysaccharide, Chelating resin, ethanolamine.

INTRODUCTION

The presence of heavy metal ions in sewage from metal industries, dyeing industries and paint industries has received greater attention in recent years, as these are toxic even in trace quantities. Several methods are employed such as precipitation, adsorption, electrochemical reduction etc. to remove heavy metal cations from effluent.

The use of cheap agricultural waste in this field is receiving attention on account of economic considerations coupled with reasonable good efficiency.

Ion exchange technique has been found to be useful in this regard. The use of complex ion formation in ion exchange chromatography allows the separation of toxic metal ions from effluent. A number of chelating ion exchange resins have been produced by resin manufacturers to overcome this problem and to encourage the application of ion exchange and to a broader range of process solutions. Using high capacity cation and anion exchange resin with the metal complexing effluent the separation of many transition and post transition metal was accomplished.

Ion selective exchanger and inorganic as well as organic selective precipitation have been used to remove toxic metal ions from effluent containing large concentration of alkali and alkaline earth metal ions.

In this paper we describe the synthesis and application of amino ethanol derivative of tamarind kernel powder (TKP) and it has been employed for removal of toxic metal ions such as lead, copper, iron, zinc and cadmium from the effluents of various metal industries.

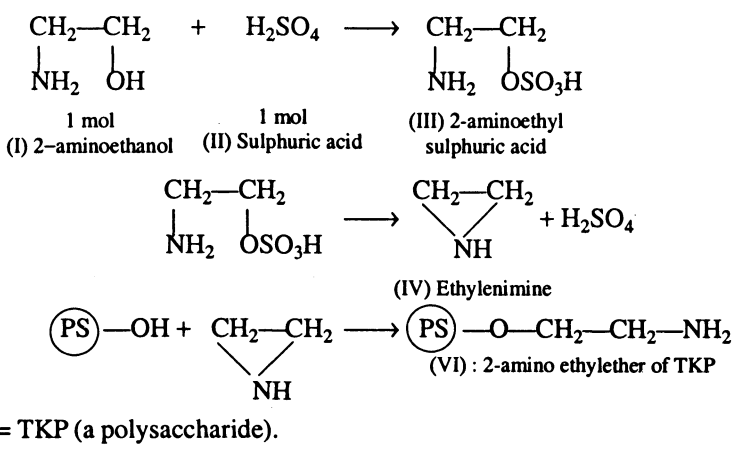
EXPERIMENTAL

Preparation of 2-amino ethyl ether of TKP

(a) **Preparation of ethylenimine:** 61 g (1 mol) of monoethanolamine was taken in a 500 mL round-bottom flask. 1 mol of 96% sulphuric acid was added dropwise in the flask. It was heated rapidly over a free flame up to 250°C temperature. Thereafter charging began. The product began to turn brown rapidly when the degree of dehydration was reduced. On cooling, the clear brown liquid solidified to a hard, white, crystalline cake of β -aminoethyl sulphuric acid. It was ground in a mortar with 60% ethanol, filtered under suction and washed with ethanol.

28.2 g (0.2 mol) of β -aminoethyl sulphuric acid was distilled with 88 g (2.2 mol) of 40% sodium hydroxide solution. The mixture was heated over a free flame till boiling started. At this point external heating was discontinued. The reaction that begins at the boiling point keeps the mixture boiling for several minutes and therefore, during this time, heating must be discontinued. One distillation required about 45 min. First KOH and then NaOH was added and finally the mixture was fractionated (b.p. = 56.5°C).

(b) **Preparation of 2-aminoethyl TKP:** 1 mol of TKP was taken in a 500 mL round-bottom flask. It was stirred with 325 mL of dioxane and then 10 mL of 50% (w/v) aqueous sodium hydroxide was added to it gradually with continuous stirring at 45°C. 14 g (0.33 mol) of ethylenimine was added to the reaction mixture and stirring was continued for a further 4 h at 60°C. The product thus formed was washed with 80% methanol, containing few drops of hydrochloric acid to neutralize excess sodium hydroxide and to convert the resin into chloride form. The product was finally washed with methanol to remove inorganic impurities and the yield was about 180 g (Scheme-1).



Scheme-1. Preparation of new 2-aminoethyl ether of TKP

Treatment of effluents

Effluent samples were collected from Ankur Mineral Pvt. Ltd. and Kotha Metal Industries. After discharge through filtration plant, these samples containe

heavy metal ions along with Cu^{2+} , Zn^{2+} , Pb^{2+} , Cd^{2+} , Mg^{2+} , Ca^{2+} , Fe^{2+} , CN^- , F^- , and SO_4^{2-} . The characteristics of Ankur Mineral Pvt. Ltd. and Kothari Metal Industries are reported in Table-1.

The procedure adopted to remove heavy toxic metal ion is as follows: 50 mL aliquots of effluents were taken in clean beakers. In each case the pH was adjusted from 6 to 10 with lime. 30 mg aminoethyl ether of TKP was added and the solution was stirred with a magnetic stirrer for about 10 min. The solution was transferred to a 100 mL measuring cylinder, pH was again checked and the mixture was allowed to stand for 4 h. The whole mass was filtered through Whatman filter paper No. 40. The filtrates after necessary treatment were used for estimation of metal ion with atomic absorption spectrophotometer model AAS 175. Air-acetylene mixture was used as fuel.

RESULTS AND DISCUSSION

The results of atomic absorption spectrophotometer are reported in Table-1. The major part of effluent is generated by mineral and metal processing industries. There is a possibility of recycling of wastewater after suitable treatment. These observations have indicated the necessity to derive more sophisticated methods to scavenge the heavy metal ions to a safer limit. The tolerance limits of heavy toxic metal ions for discharge of effluent into natural stream are different for different metals (*e.g.*, for India, Cu 3.0 ppm, Cd 2.0 ppm, Pb -0.1 ppm, Zn 5.0 ppm and Fe -3.0 ppm). 2-Aminoethyl ether of TKP reduces the heavy toxic metal ion concentration from effluent to levels much below the discharge limit as shown in Table-2. Hence the polymeric reagent can be considered a very effective reagent.

TABLE-1
CHARACTERISTICS OF EFFLUENTS CONTAMINATED WITH HEAVY TOXIC METAL IONS OBTAINED FROM VARIOUS UNITS OF MINERAL AND METAL PROCESSING INDUSTRIES

Characteristics	Sources	
	Effluent of Ankur Mineral Pvt. Ltd.	Effluent of Kothari Metal Industries
Colour	Brown	Reddish
pH	4.70	6.50
Total hardness (ppm)	890	1085
Iron	103	0.29
Copper	0.75	0.03
Zinc	6.50	0.25
Lead	0.50	0.89
Magnesium	90.23	60.23
Cadmium	0.13	0.01
Calcium	180.00	290.00
Cyanide	0.03	0.05
Fluoride	0.23	0.38
Sulphate	768.60	987.00

TABLE-2
REMOVAL OF TOXIC METAL IONS FROM THE EFFLUENT FROM VARIOUS
NON-FERROUS MINERALS AND METAL PROCESSING INDUSTRIES

Source	Metal ions	Concentration of various metal ions (ppm)		
		Untreated effluents	After treatment with lime at 8.0 pH	After treatment with 2-aminoethyl ether of TKP 8.0 pH
Effluent of Ankur Mineral Pvt. Ltd. (pH 4.7)	Iron	103.00	Nil	Nil
	Copper	0.75	0.21	Nil
	Zinc	6.50	0.41	0.02
	Lead	0.50	Nil	Nil
	Magnesium	90.23	90.23	90.23
	Cadmium	0.13	Nil	Nil
	Calcium	180.00	180.00	180.00
Effluent of Kothari Metal Industries (pH 6.5)	Iron	0.29	Nil	Nil
	Copper	0.03	Nil	Nil
	Zinc	0.25	0.17	Nil
	Lead	0.89	Nil	Nil
	Magnesium	60.23	60.23	60.23
	Cadmium	0.01	Nil	Nil
	Calcium	290.00	290.00	290.00

These reagents are dual functioning reagents, acting as flocculants-cum-selective ion binders. They can be used on a large scale on commercial basis. The polysaccharides used in the preparation of the reagent is basically a flocculant. It is cheap and also available in abundance. The reagent being cheap, the spent reagent can be discarded as floc and regeneration is not required. It is possible to incinerate the floc and recover the metals. Since the reagents are directly added to the effluent, there is no limitation to the quantity of water that can be treated in one batch. In case of conventional ion exchangers, such restrictions are imposed by the resin bed and flow rate.

ACKNOWLEDGEMENT

The authors are thankful to Head, Department of Chemistry, J.N.V. University, Jodhpur for providing laboratory facilities.

REFERENCES

1. R.E. Wing, W.M. Doane and C.R. Runell, *J. Appl. Polym. Sci.*, **19**, 847 (1975).
2. J.S. Fritz and J.N. King, *Anal. Chem.*, **48**, 570 (1976).
3. A.V. Singh, S. Gupta and S.C. Gupta, *Desalination*, **104**, 235 (1996).
4. S. Mishra, *Curr. Sci. (India)*, **75**, 1915 (1998).
5. J.P. McIntyre, *Industrial Water Reuse and Wastewater Minimization*, Betz Dearborn Inc. (1998).
6. A.K. Mitra De and A. Karechoudhari, *Indian J. Chem.*, **39B**, 311 (2000).
7. V. Shrivastav, R. Gupta and R.R. Gupta, *Indian J. Chem.*, **39B**, 223 (2000).
8. S.C. Suit, *Chem. Innovation*, **30**, 27 (2000).
9. Daneiel I.O. Ikhu-Omoregbe and O. Igwueokwu, *Res. J. Chem. Environ.*, **6** (2002).
10. M. Farooqi, S. Kotharkar and A. Zaheer, *Asian J. Chem.*, **14**, 95 (2002).
11. V. Gupta, A.V. Singh and K. Bana, *Res. J. Chem. Environ.*, **7**, 2 (2003).

(Received: 23 December 2003; Accepted: 22 April 2004)

AJC-3401