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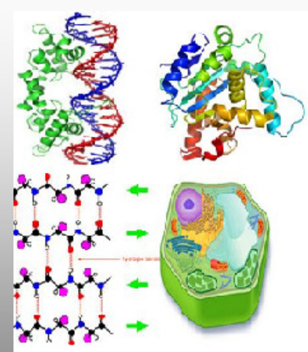
By
Manisha Sharma



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RESEARCH PAPER

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Synthesis, Characterization and Analytical Application of New Resin – CMAHPE-TKP

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ABSTRACT

3-(1-CARBOXY METHYL) AMINO–2-HYDROXY PROPYL ETHER OF TKP (CMAHPE – TKP) resin have been synthesized by using natural polymer tamarind kernel powder(TKP) a hydrophilic polysaccharide, obtained from seeds of tamarind fruit. This new resin was characterized by moisture content, nitrogen content, FTIR spectra and total ion exchange capacity of synthesized resin. CMAHPE – TKP resin act as flocculent cum metal ion ex changer and can be used as scavenger for toxic & hazardous metal ions present in effluent of mineral and metallurgical industries.

Keywords - Tamarind kernel powder (TKP), Epichlorohydrin, Glycine, FTIR spectra, Ion exchange chromatography.

INTRODUCTION

The defilement of water as a result of human activities, increasing industrialization urbanization and development activities and consequent pollution of water (Fig.**1**) has brought a water crisis. The industrial wastes have the greatest potential for polluting the recipient water. The nature and composition of industrial waste depends upon the raw materials, process and operational factors (Halim *et al*., 2003). Many toxic metal ions have been discharged into the environment as industrial waste, causing serious water pollution. Pb^{2+}, Zn^{2+}, Cd^{2+}, Cu^{2+}, Fe^{3+} and Cr^{6+} are especially common metal ions that tend to accumulate in organism, causing numerous diseases and disorders. (Jumbe and Nandini, 2010)

Fig. 1

The removal of toxic metal ions from contaminated waste water is one of the most important environmental and economic issues today. The ever increasing demand for water of high quality has caused considerable attention towards recovery and reuse of wastewater. Numerous processes exist for removing dissolved toxic metal ions including ion exchange, precipitation, ultra filtration, reverse osmosis and electrolysis. Among various physico-chemical treatment processes, adsorption is found to be highly effective, cheap and easy method (Dabrowskie et al., 2004).

High capacity cation and anion exchange resins are used for the removal of many toxic metal ions from industrial effluent (Kim and Keane, 2002).The greatest advantage of these exchange methods is their regenerability. The metal ions are easily separated from the resin by slightly altering the pH i.e. washing the used resin with slightly more acidic or alkaline water with pH greater than the pH of their maximum adsorption. (Rengaraj and Moon, 2002).

Major advantage with the polysaccharide based resin is their hydrophilicity that makes the functional group readily accessible (Kumar et al., 2006).Tamarind Kernel Powder (TKP) being hydrophilic in nature is preferred as a base polymer for preparation of cation exchangers and it is considered that cross-linking of TKP polymers matrix shall yield insoluble resins suitable for use in a column to remove toxic metal ions from metallurgical industrial effluent. This research focuses on synthesis of new cation exchange resin (CMAHPE – TKP) and developing a cost effective method for treatment of highly contaminated industrial effluents.

MATERIAL AND METHODS

All chemicals were of Analar grade and were used as supplied without further purification.

(A) SYNTHESIS OF 3-(1-CARBOXY METHYL) AMINO–2-HYDROXY PROPYL ETHER OF TKP (CMAHPE – TKP) RESIN

(I) Preparation of Secondary Amine Epoxide –

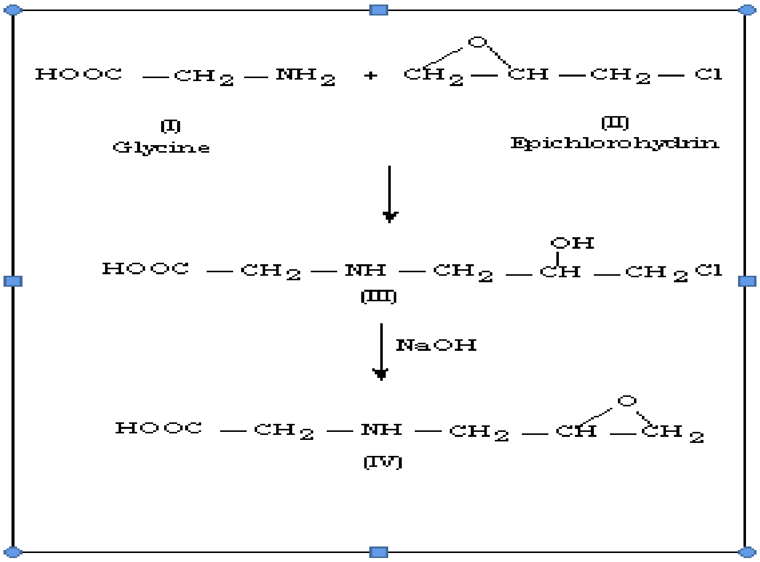

Fig. (2) Preparation of secondary amine epoxide

7.8 ml (0.1 mole) of epichlorohydrin was taken in 200 ml round bottom flask fitted with reflux condenser. 7.59 g (0.1 mole) glycine (40% solution in water) was added to it drop by drop and the temperature maintained between 40-45 °C, about 20 ml of 50% aqueous solution of NaOH containing 4.0 g (0.1 mole) sodium hydroxide was added drop wise to the reaction mixture, using a drop of phenolphthalein indicator. Stirring was continued for three hours and then extracted with acetone 3 to 4 times in order to separate it from solid sodium chloride formed during the reaction. The preparation of secondary amine epoxide was shown in Fig. 2.

(II) Preparation (CMAHPE-TKP) resin-

83 g (0.5 mole) TKP was taken in dioxane and 5 ml of 50% aqueous sodium hydroxide was added in order to make it alkaline, secondary amine epoxide (IV) solution in dioxane was added to the alkaline TKP (Tamarind Kernel Powder), with stirring and the reaction was continued for four hours. The product (V) was filtered and washed with 70% methanol containing some acetic acid and dried in air. A light yellow, free flowing powder obtained. The yield of CMAHPE-TKP resin was 95 g. The preparation was shown in Fig. 3.

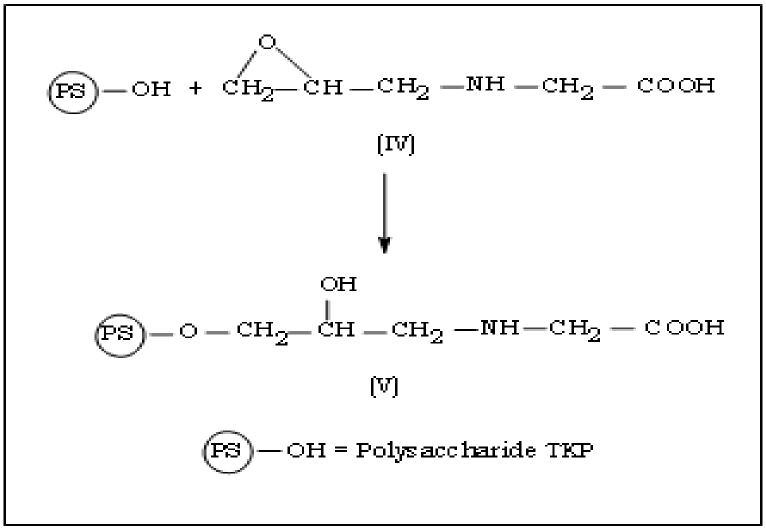

Fig. (3) Synthesis of 3-(1-Carboxy methyl) amino–2-hydroxy propyl ether of TKP (CMAHPE-TKP) resin

(B) CHARACTERISTICS OF CMAHPE-TKP RESIN

Moisture content

1 g of the resin in hydrogen form was taken and dried to a constant weight in a vacuum dessicator at 70 °C for 24 hours, and then resin was weighed.

Nitrogen content

The Kjeldahl's method is the standard method for determination of nitrogen content, 0.2 g of vacuum dried resin was taken in a dried Kjeldahl's flask and 10 ml of concentrated sulphuric acid (18 N) and 0.60 g of the catalyst were added in it. Heating was continued for 2 hrs. The solution was then chilled and quantitatively transferred with 30 ml of water to a distillation apparatus for ammonia determination. 12 ml of 10 N sodium hydroxide was then added and total volume in the flask was made to 75 ml. Liberated ammonia was steam distilled for 5 minutes into the receiver containing 5 ml of 4% boric acid and 5-6 drops of indicator. The distilled ammonia was titrated with 0.05 N hydrochloric acid.

1 ml of 0.05 N HCl = 0.7003 mg of nitrogen.

FTIR spectra

FTIR spectra of newly synthesized chelating resin was recorded using Shimadzu, Japan- 8101 'A' (4000-400 cm^{-1}) spectrophotometer, Fig. 4.

Total Ion Exchange Capacity Determination

Back titration procedure was followed in which 1g of resin was converted to hydrogen ion form; about 1g of wet resin was taken in an Erlenmayer flask. 200 ml of 0.05 N (standardized) sodium hydroxide containing 5 ml of 5% sodium chloride solution was added to it and contents were allowed to stand overnight. 25 ml of aliquot from supernatant liquid was taken for titration with standard 0.05 N HCl using phenolphthalein indicators. The remaining resin in H^+ forms was used to determine moisture content. Capacity of the resin was calculated as under:

$(0.05 \text{ N} \times V_1) - 8\,(0.05 \text{ N} \times V_2)$

Q (meq/gm) = ---W

Where,

Q (meq/g) = Total scientific weight capacity (meq/g)

V_1 = Volume of 0.05 N sodium hydroxide solution (200 ml) V_2 = Volume of 0.05 N hydrochloric acid consumed in titration = 20.3 ml

W = Weight in gm of dry resin

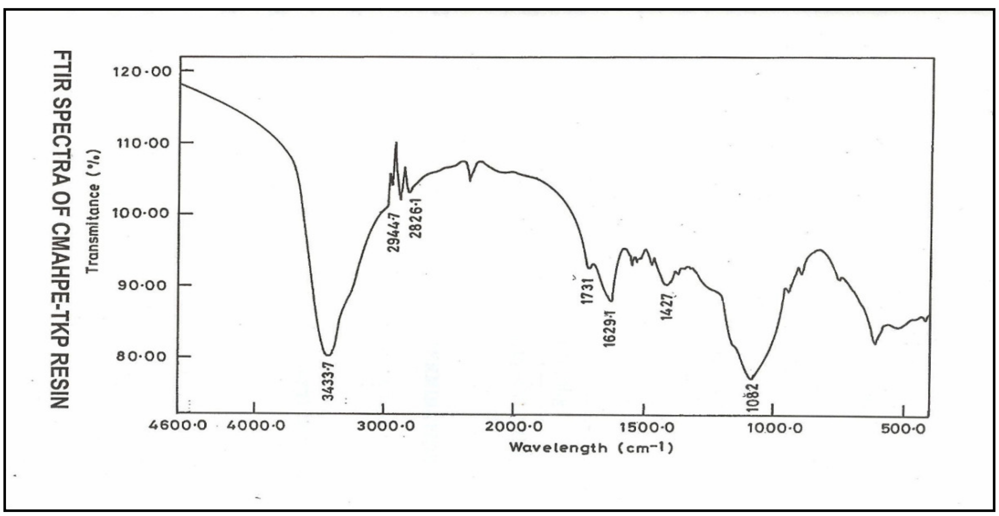

Fig. 4. FTIR-Spectra of CMAHPE-TKP Resin

(C) TREATMENT OF EFFLUENTS WITH CMAHPE-TKP RESIN

Effluent samples contaminated with toxic metal ions were collected from metallurgical industries around Jodhpur region. There is a possibility of recycling of industrial effluent after suitable treatments. The characteristics of samples of two metallurgical industries are reported in Table (1), and then industrial effluents were treated by new CMAHPE-TKP resin.

Table.1 Characteristics of effluents contaminated with toxic metal ions obtained from two units of metallurgical industries

Characteristics	Sources	
	Effluent of industry **A***	Effluent of industry **B***
Colour	Blackish	Brown
pH	8.1	7.9
Total hardness (ppm)	735	969
Metal ions (ppm) :		
Iron	119	105.0
Copper	0.82	0.63
Zinc	6.56	0.32
Lead	0.81	0.97
Cadmium	0.31	0.08
Magnesium	85.05	61.01
Calcium	159.0	264.8
Anions (ppm) :		
Cyanide	0.08	0.092
Fluoride	0.311	0.435
Sulphate	695.1	889.01

RESULTS AND DISCUSSION

Moisture content – Calculated as follows:

The weight of dry resin = 0.9558 g

Weight of moisture = 1-0.9558 = 0.0442 g

% moisture content = 0.0442 x 100 = 4.42%

Nitrogen content – Calculated as follows:

Amount of resin taken = 0.2 g

Volume of 0.05 N HCl consumed = 7.0 ml

Mg of nitrogen in resin sample = 0.7003 x 7.0

= 4.9021 mg/0.2g of resin

% of Nitrogen content = 2.45%

FTIR Spectra - Assignment of peaks is mentioned in table. 2.

Total Ion Exchange Capacity - Calculated as follows:

$$Q\ (meq/g) = \frac{(0.05 \times 200) - 8\ (0.05 \times 20.3)}{0.9597}$$

$= 1.9589$ meq/g of H^+ form of dry resin

The total capacity of CMAHPE – TKP resin was found out to be 1.9589 meq/g of H^+ form of dry resin. This research is aimed at finding method for removal of toxic metal ions from industrial Effluents. Water treatment does not require elaborate pretreatment, so the efforts are made to prepare specific chelating resins which can remove these toxic metal ions at pH of natural water. However in case of treatment of industrial waste water, where the initial pH of waste water may be different, certain pretreatment is necessary.

Toxic metal ions can be removed from effluent up to safer limit by using newly synthesized CMAHPE-TKP resin and results are mentioned in table (3).

Table. 2. FTIR spectra- Assignment of peaks

K-salt of ligand Bands cm^{-1}	Assignment
1082 (s)	ν (C – O) stretching of ether
1374 (m)	Secondary alcohol (C-O) stretching
1427 (m)	(C–H) bending in alkane. $-CH_2$
1629.1 (s)	(N-H) bending
1731 (m)	Carboxylic ν (C=0) stretching
2826 – 2944 (m)	ν (C-H) stretching of alkane ($-CH_2$, $-CH_3$)
3433.7 (s)	Band for alcoholic (O-H) stretching and (N-H) stretching

Table. (3) Treatment of industrial effluents with CMHPE-TKP resin

Source	Concentration of various metal ions (ppm)			
	Metal ions	Untreated effluents	After treatment with lime at pH 8.0	After treatment with **CMHPE – TKP** resin at pH 8.0
Effluent of metallurgical industry A pH = 8.1	Iron	119	Nil	Nil
	Copper	0.82	0.32	Nil
	Zinc	6.56	0.45	0.08
	Lead	0.81	0.08	Nil
	Cadmium	0.31	0.19	Nil
	Magnesium	85.05	85.05	85.05
	Calcium	159.0	159.0	159.0
Effluent of metallurgical industry B pH = 7.9	Iron	105.0	Nil	Nil
	Copper	0.63	0.23	Nil
	Zinc	0.32	0.14	Nil
	Lead	0.97	0.12	Nil
	Cadmium	0.08	0.019	Nil
	Magnesium	61.01	61.01	61.01
	Calcium	264.8	264.8	264.8

Removal of toxic metal ions from the effluents by treatment with new synthesized resin:

Magnesium and calcium metal ions have not been removed from effluents by resin treatment because there is incomplete dissociation of salts of bivalent metals Mg and Ca in these effluents. The absorption of ions on resin depends upon the degree of cross-linking and nature of functional groups in the resins.

Tamarind Kernel Powder being cheap and abundantly available is preferred on economic ground. The new synthesized CMAHPE–TKP resin is hydrophilic and biodegradable, so after effluent treatment used resins can be disposed off without facing any environmental problem. Thus, present research reveals that newly synthesized CMAHPE-TKP resins can be effectively used for removal of toxic metal ions from metallurgical industrial effluents.

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