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Research Article

Synthesis of a New Resin - MBHPE-TKP: Its Characterization and Application

Manisha Sharma[1]*, Bina Rani[2]

[1]*Assistant Professor, Department of Chemistry, Mahila PG Mahavidyalaya, Jodhpur (Rajasthan)*

[2]*Professor, Department of Engineering Chemistry & Environmental Engineering, PCE, Sitapura, Jaipur, Rajasthan*

**Corresponding Author: manisha.orgvyas@gmail.com*

ABSTRACT

3-(3-methyl) butoxy-2-hydroxy propyl ether of TKP (MBHPE-TKP) resin has 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.(MBHPE – TKP)resin act as flocculent cum metal ion exchanger 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, Iso-amyl alcohol, FTIR spectra, Ion exchange chromatography

1. INTRODUCTION

Water, the most vital resource for all kinds of life on this planet is also the resource, adversely affected both quantitatively and qualitatively by human activities. The defilement of water as a result of human activities, the increasing industrialization, urbanization and developmental activities and consequent pollution of water has brought a veritable water crisis. The industry continues to be one of the most significant cause of pollution of aquatic ecosystems due to a diverse kind of wastes especially toxic heavy metal ions produced by them. 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. Metal plating industries release substantial quantities of heavy metals and cyanides in their wastes. Jodhpur is centrally situated in desert region of western Rajasthan, a large number of industries have recently being setup in Jodhpur, which are mainly of four types : textile, guar gum, dying & printing and metal industries. 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. The effluents from smelters, mines and other non-ferrous industries have heavy toxic metal ions viz., Pb^{2+}, Cu^{2+}, Fe^{3+}, Cd^{2+} and Zn^{2+}. Concentration of these toxic metal ions in effluent is above the permissible limit. Pollution of water sources has further added to the problem of available water human consumption. Therefore, parallel development has been searched for new subsoil water and the recycling methods, which are cost effective for treatment of heavy metal ions contaminated industrial effluents.

The removal of toxic and heavy 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 heavy 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 and cheap method. Major advantage with the polysaccharide based resin is their hydrophobicity that makes the functional group readily accessible. 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 heavy metal ions from metallurgical industrial effluent. This research paper focuses on synthesis of new cation exchange resin (MBHPE – TKP) and developing a cost effective method for treatment of highly contaminated industrial effluents.

2. MATERIAL AND METHODS

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

2.1 *Synthesis of 3-(3-Methyl)Butoxy-2-Hydroxy Propyl Ether of TKP (MBHPE-TKP) Resin*

2.1.1 *Preparation of 1,2-Epoxy propyl-3-methyl butyl ether*

7.8 ml (0.1 Mole) of epichlorohydrin was taken in a 200 ml round bottom flask and 10.9 ml (0.1 mole) of iso-amyl alcohol was added slowly to it with stirring on a magnetic stirrer. The stirring was continued for two hours or till the epichlorohydrin layer disappeared. The product so formed is an open chain viscous compound, miscible with water, to this reaction mixture, 50% aqueous solution of NaOH containing 4.0 g (0.1 mole) sodium hydroxide was added drop wise, using phenolphthalein indicator. Stirring was continued for three hours and then extracted with acetone in order to separate it from solid sodium chloride formed during the reaction. The preparation of 1,2-Epoxy propyl-3-methyl butyl ether is shown in Fig. (1)

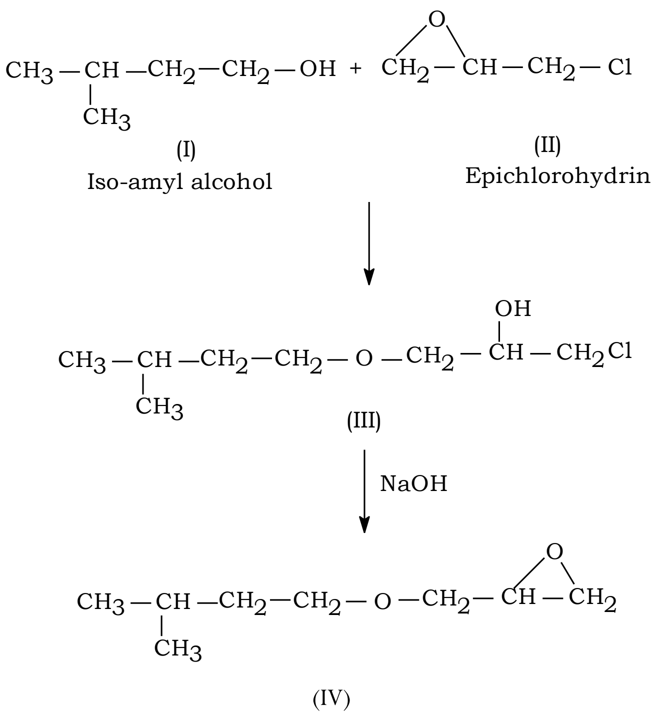

Fig 1: Preparation of 1,2-Epoxy propyl-3-methyl butyl ether

2.1.2 Preparation of 3-(3-Methyl) Butoxy-2-Hydroxy Propyl Ether of TKP (MBHPE-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, 1,2-epoxy propyl-3-methyl butyl ether (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 preparation is shown in Fig. (2)

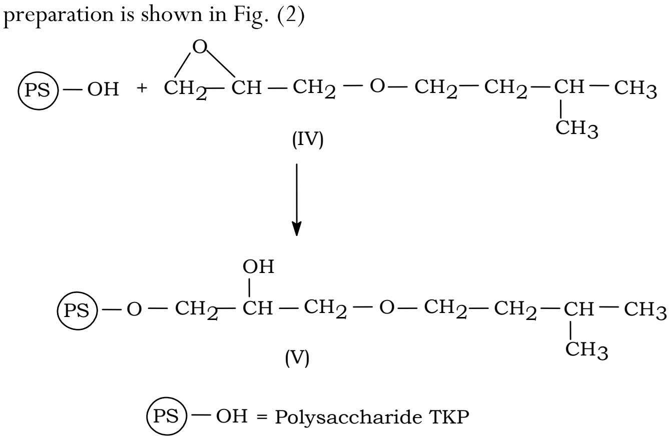

Fig 2 : Synthesis of 3-(3-Methyl) Butoxy-2-Hydroxy Propyl Ether of TKP (MBHPE-TKP) Resin

2.2 Characteristics of MBHPE-TKP resin

2.2.1 Moisture content

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

2.2.2 Nitrogen content in MBHPE-TKP Resin

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.

2.2.3 FTIR and IR spectra of chelating resins of Tamarind

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

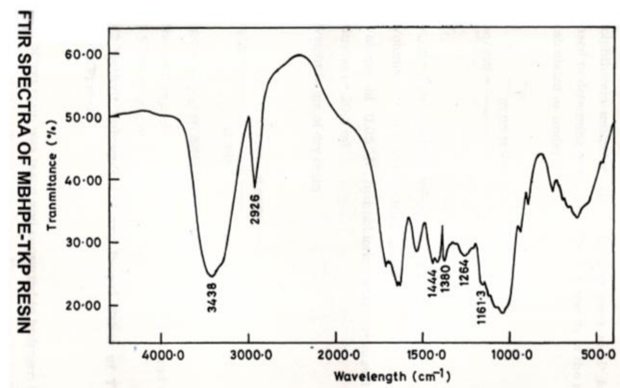

Fig 3: FTIR spectra of the MBHPE – TKP resin

2.2.4 Total Ion Exchange Capacity Determination

Back titration procedure was followed in which 1g of resin was converted to hydrogen ion form; about 1 g 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 indicator. The remaining resin in H^+ forms was used to determine moisture content. Capacity of the resin was calculated as :

$$Q\ (meq/gm) = \frac{(0.05\ N \times V_1) - 8\ (0.05\ N \times V_2)}{W}$$

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 = 20.8ml
W = Weight in gm of dry resin

2.3 *Treatment of Effluents With MBHPE-TKP Resin*

Effluent samples containing heavy metal ions were collected from metallurgical industries around Jodhpur region. The major part of effluents is generated by mineral and metal processing industries. The characteristics of samples of two metallurgical industries are reported in Table (1).

Table 1: Characteristics of Two Metallurgical Industries

Characteristics	Sources	
	Effluent of metallurgical industry C*	Effluent of industry D*
Colour	Dark Brown	Greenish Yellow
pH	8.9	7.3
Total hardness (ppm)	895	1121
Metal ions (ppm) :		
Iron	105.0	0.520
Copper	0.82	0.490
Zinc	5.90	0.58
Lead	0.55	0.94
Cadmium	0.24	0.042
Magnesium	83.12	62.01
Calcium	178.0	281.0
Anions (ppm) :		
Cyanide	0.042	0.079
Fluoride	0.31	0.310
Sulphate	733.0	845.0

** Names of industries are not mentioned due to legal reasons.*

3. RESULTS AND DISCUSSION

Moisture content– calculated as follows:

The weight of dry resin	= 0.9687 g
Weight of moisture	= 1-0.9687 = 0.0313 g
% moisture content	= 0.0313 x 100 = 3.13%

Nitrogen content – calculated as follows:

Amount of resin taken	= 0.2 g
Volume of 0.05 N HCl consumed	= 6.4 ml
mg of nitrogen in resin sample	= 0.7003 x 6.4 = 4.48mg/0.2g of resin
% of Nitrogen content	= 2.24%

FTIR Spectra - Assignment of peaks are mentioned in Table (2)

Table 2: Assignments of Peaks (FITR Spectra)

K-salt of ligand Bands cm^{-1}	Assignment
1161.3 (s)	ν (C-O) stretching of eth
1264 (m)	Secondary alcohol (C-O) stretching
1380 (s)	Alkane (C-H) bending of –$CH(CH_3)_2$
1444 (m)	ν (C-H) bending in alkane, -CH_2
2926 (m-s)	ν (C-H) stretching in –CH_2, CH_3
3438 (s)	Alcoholic ν (O-H) stretching

Total Ion Exchange Capacity- Calculated as follows:

$$Q\ (meq/g) = \frac{(0.05 \times 200) - 8\ (0.05 \times 20.8)}{0.9638}$$

$$= 1.7431\ meq/g \text{ of } H^+ \text{ form of dry resin}$$

The total capacity of MBHPE – TKP resin was found out to be 1.9589 meg/g of H^+ form of dry resin.

This research paper is aimed at finding method for removal of heavy 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 heavy 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.

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

Results are mentioned in table (3) shows that toxic metal ions can be removed from effluent up to safer limit by using newly synthesized resin.

Table 3: Treatment of effluent with new MBHPE-TKP resin

Source	Concentration of various metal ions (ppm)			
	Metal ions	Untreated effluents	After treatment with lime at pH 8.0	After treatment with MBHPE-TKP resin at pH 8.0
Effluent of metallurgical industry C pH = 8.9	Iron	105	Nil	Nil
	Copper	0.82	0.18	Nil
	Zinc	5.90	0.30	0.27
	Lead	0.55	0.08	Nil
	Cadmium	0.24	Nil	Nil
	Magnesium	83.12	83.12	83.12
	Calcium	178.0	178.0	178.0
Effluent of industry D pH = 7.3	Iron	0.520	Nil	Nil
	Copper	0.490	0.09	0.02
	Zinc	0.58	0.29	Nil
	Lead	0.94	0.06	Nil
	Cadmium	0.042	Nil	Nil
	Magnesium	62.0	62.0	62.0
	Calcium	281.0	281.0	281.0

Magnesium and calcium metal ions have not been removed from effluents by this 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 resins i.e. MBHPE–TKP 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 MBHPE-TKP resins can be effectively used for removal of toxic heavy metal ions from industrial effluents.

4. ACKNOWLEDGMENT

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