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4 **Synthesis and Characterization of a New Ion Exchanger Tamarind 5-Amino Salicylic Acid**  
5 **Hydroximate [TASAH] Resin and its Application in Removal and Recovery of Toxic Metal**  
6 **Ions from Effluent of Karishma Textile, Jodhpur, India**  
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19  
20 **Abstract**

21  
22 A new composite ion exchanger tamarind 5-amino salicylic acid hydroximate [TASAH] resin  
23 was synthesized for removal of toxic metal ions, which have good ion-exchange capacity, higher  
24 stability, reproducibility and selectivity. The characterization of the resin was carried out by  
25 determining the ion exchange capacity, elemental analysis, pH titration, FT-IR spectra and  
26 thermal analysis (TGA). The distribution coefficients ( $K_d$ ) of toxic metal ions were determined in  
27 aqueous solution and effluent of Karishma Textile at different pH. On the other hand, physical  
28 property of TASAH resin was also studied. The effects of pH, agitation speed, treatment time,  
29 flow rate, contact time, temperature and adsorbent dose, on the removal of metal ions from  
30 industrial wastewater were investigated. The adsorbed metal ions were effectively eluted by  
31 different strength of acid solution. The resin is amenable for continuous process and can be  
32 regenerated several times. The adsorption of different metal ions on TASAH resin follows the  
33 order;  $Zn^{2+} > Fe^{2+} > Co^{2+} > Cd^{2+} > Cu^{2+}$ .  
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46 **Subject Headings:** Environmental protection agency; Environmental issues; Waste  
47 management; Waste treatment; Wastewater management; Water  
48 management; Water pollution.  
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50  
51 **Key Words:** - Distribution coefficients; Ion exchange capacity; TASAH resin; Wastewater;  
52 Toxic metal ions  
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## Introduction

It is well known that, toxic metal ions are remaining a serious environmental problem facing the world for water pollution as a result of their numerous industrial applications. In many of them are known to be toxic or carcinogenic even at low concentration, not biodegradable and tend to accumulated in living organism causing serious diseases and disorders. Water pollution caused by heavy metal ions from various industrial effluents has gained a great worldwide attention (Nriagu et al. 1988). A number of regulation agencies have taken care to restrict their untreated discharge into public sewers, rivers, the sea and on land. The removal of heavy metal ions is very important due to their heavy nature in water, posing serious hazards to human health as well as to other living organisms. Therefore, it is essential to extend methods for the removal of metal ions to decrease the pollution load on the environment.

Different methods such as precipitation (Harper and Kingham, 1992), solvent extraction (Kiezyk et al. 2009; Rios et al. 2010) chemical and electrochemical technique (Diaz et al., 2009), advanced oxidation process (Kepa et al. 2008; Diwani et al. 2009), ion-exchange method (Saha et al. 2000; Wang et al. 2002; Singh and Saraf 2009; Das et al. 1999; Lata et al. 2003), ultrafiltration and reverse osmosis (Rengaraj et al. 2001; Yurlova et al. 2002; Benito et al. 2002) have been developed for the removal of heavy metal ions from industrial effluents and wastewater. A major drawback with precipitation is sludge production. Adsorption using commercial activated carbon (CAC) can remove heavy metal ions from wastewater, such as Cd (Ramos et al. 1997), Ni (Shim et al. 2001), Cr (Ouki et al. 1999) and Cu (Monser et al. 2002). However, CAC remain an expensive material for heavy metal removal. The resins prepared with DVB-styrene backbone are hydrophobic (Sarin et al. 1980). Also these resins are prepared from petrochemicals, which are very costly. The ion exchange capacity of these resins depends mainly

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4 on quantity of functional groups and pH of solution. The most widespread chelating functional  
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6 groups are: thiol (Kantipuly et al. 2001), thiourea (Hubicki et al. 2009), dithizone (Grote et al.  
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8 2001; Chwastowska et al. 2001; Shah et al. 1998; Chwastowska et al. 2004) and triisobutyl  
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10 phosphine sulfide (Hidalgo et al. 2001; Sanchez et al. 2001) which are used for removal of heavy  
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12 metal ions from effluent and slow adsorption kinetics is their undesired characteristics.  
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16 Ion-exchangers of polyvalent metals often exhibit much better ion-exchange behavior and  
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18 are always of interest because of their higher selectivity, resistance to higher temperature,  
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20 radiation field and to some extent chemical stability than those of commercial ion-exchange  
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22 resins (Vesely et al. 2001; Inoue 2003). Inorganic ion-exchange materials also have been  
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24 established to have applications in various disciplines, i.e. environmental studies, medical  
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26 science (kidney dialysis), ion-selective electrode preparation (Hassan et al. 2000), heterogeneous  
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28 solid-state membrane formation, preconcentration and ion-exchange fibers preparation.  
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30 Inorganic-organic materials are known to have a large selectivity for mono and multivalent  
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32 cations (Maya 2003; Clearfield 2001; Khan et al. 2003; Khan et al. 2004; Amicangelo and  
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34 Leenstra 2005; Patel et al. 2006).  
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41 Tamarind kernel powder (TKP) is obtained from the seeds of the tamarind tree,  
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43 *Tamarindus indica*, a common forest and cultivated tree found primarily in India, Burma,  
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45 Bangladesh and Shri Lanka. TKP is recommended as a stabilizer in ice-cream, mayonnaise,  
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47 cheese and as an ingredient or agent in a number of pharmaceutical products. Other industrial  
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49 uses included employment in colour printing of textiles, paper sizing, leather treating, the  
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51 manufacture of a structural plastic, a glue for wood, stabilizer in bricks, and a thickener in some  
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53 explosives. Medicinal uses of the TKP are uncountable. TKP is a polymer with an average  
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55 molecular weight of 52,350 and a monomer contains three sugars glucose, galactose, and xylose  
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4 in a molar ratio of 3:1:2 were reported by Khanna et al (Khanna et al. 1987). The polymer  
5 consists of cellulose type spine, which carries xylose and galactoxylose substituents. About 80 %  
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7 consists of cellulose type spine, which carries xylose and galactoxylose substituents. About 80 %  
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9 of the glucose residues are substituted by a 1+6 linked xylose units, which themselves are  
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11 partially substituted by 1-2 galactose residues (Lang et al. 1993) .The structural of Tamarind  
12  
13 kernel powder is given in **Fig 1**.

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16 The ion exchangers based on tamarind kernel powder is hydrophilic and biodegradable,  
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18 where as other ion exchangers has prepared from petrochemical product which is not hydrophilic  
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20 and biodegradable. Due to rising prices of petroleum products the tamarind kernel powder has  
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22 been selected for development of tamarind 5-amino salicylic acid hydroximate (TASAH) resin,  
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24 which cost is very low, because it is locally available in large quantities from agriculture  
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26 resources and these biopolymers are environment friendly.  
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31 In this work, we describe the synthesis and characterization of novel tamarind 5-amino  
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33 salicylic acid hydroximate resin and its applications for removal and recovery of heavy metal ions  
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35 from reference solution and effluent of Karishma Textile, Jodhpur, India. For this purpose,  
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37 various factors affecting the adsorption, pH, distribution coefficient ( $K_d$ ), removal percentage of  
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39 metal ions were studied.  
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## 42 43 **Experimental**

### 44 45 **Chemicals**

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47 Analytical grade chemicals supplied by (Loba Chemic, Mumbai) were used in all  
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49 experiments. Tamarind kernel powder was supplied by (Ases Chemical Works, Jodhpur, India).  
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51 All other compounds used in the synthesis were of commercial high purity grade, and used  
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53 without further purification.  
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## Sample

Reference solution have metal ions concentration (ppm) - 44 ( $\text{Fe}^{2+}$ ); 22 ( $\text{Cu}^{2+}$ ); 55 ( $\text{Zn}^{2+}$ ); 18 ( $\text{Co}^{2+}$ ); 48 ( $\text{Cd}^{2+}$ ) and the effluent of Karishma Textile, Jodhpur (Rajasthan) have following characteristic features as given in **Table 1**.

## Synthesis of Tamarind 5-Amino Salicylic acid Hydroximate [TASAH] Resin

### (a) Preparation of Methyl ester of 5-amino salicylic acid

15.3 g (0.1 M) of 5-amino salicylic acid was taken in a 250 ml round bottom flask. To the acid excess methanol was added and then dry HCl gas was passed till the saturation was completed. The reaction mixture was refluxed for 3 hrs on mantle. The methyl ester of 5-amino salicylic acid was formed.

### (b) Synthesis of hydroximate of 5-amino salicylic acid

8.35 g (0.05 M) of methyl ester of 5-amino salicylic acid and methanolic solution of 3.49 g (0.05 M) hydroxyl amine hydrochloride were stirred on magnetic stirrer for 5 hrs in round bottom flask at 30°C. The pH of the reaction mixture was adjusted by sodium methylate to pH 9.2. The hydroxamic acid of 5-amino salicylic acid was formed which was filtered on Buchner funnel and washed with 80 % methanol solution.

### (c) Incorporation of hydroximate of 5-amino salicylic acid in tamarind

3.36 g (0.02 M) of hydroxamic acid of 5-amino salicylic acid was taken in 10 ml methanol and stirred at 45°C for 5 hrs. During the process of stirring 4.36 g (0.01 M) of epoxy ether of tamarind was added.

The product formed was filtered on Buchner funnel and the resin was successively treated with 0.1 N HCl, 0.1 N NaOH and 0.1 N HCl and was finally suspended in 200 ml portion of 0.1 N HCl. Supernatant liquid was decanted and resin was washed 5-6 times with 80 %

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4 methanol to remove the suspended impurities which do not settle down. Washing was continued  
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6 till supernatant liquid was clear and free from acid. Lastly, the product was washed with absolute  
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8 alcohol and dried in vacuum. The product was free flowing powder and the yield was 7.98 g.  
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11 The reaction scheme is shown in **Fig 2**.

### 12 13 14 **Physical property of TASAH resin**

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16 The physico –chemical properties like percentage moisture content, percentage solid, true  
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18 density, apparent density, void volume fraction, swelling studies and volume capacity were  
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20 studied according to standard methods (Vogel, 1989; Helfferich, 1962). These results are shown  
21  
22 in **Table 2**.

### 23 24 25 **Swelling studies of TASAH Resin**

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27 The H<sup>+</sup> from of dry resin (0.5 g) was equilibrated with 50 ml of distilled water for 4 days.  
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29 The swollen resin was collected by filtration, adhering trace of water were removed by pressing  
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31 with filter and sample was weighed. The swollen resin was dried in a vacuum for 24 hrs and  
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33 weighed again form the swollen and dry weight of the sample the equilibrium water calculated  
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35 (EWC) using the following equation:  
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$$\text{EWC} = \frac{\text{Weight of wet resin} - \text{Weight of dry resin}}{\text{Weight of wet resin}}$$

### 66 67 68 **Column experiment**

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70 In the column experiment, a glass tube with 1.6 cm internal diameter and 20 cm height,  
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72 packed with 9 cm of resin (8.5 g) was used. 50 ml of the reference metal ion solution were  
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74 passed through the column at a flow rate of 2 ml per minute. The flow rate was controlled by a  
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peristaltic pump. The column was washed with 20 ml of deionized water and the washing was rejected. The metal ions were eluted quantitatively with different strength of acids.

### Determination of Distribution coefficient

The distribution coefficient ( $K_d$ ) of metal ions on resins were determined by batch method. In all cases for the determination of  $K_d$  50 ml reference solution was taken in a conical flask and the pH was adjusted by ammonium hydroxide and ammonium chloride. Twenty milligrams of TASAHA resin were added to the solution and stirred on a magnetic stirrer for 2 hours and the contents were equilibrated. The solution was filtered through Whatman filter paper No. 40. The residue on the filter paper was equilibrated with 4 N HCl, and the solution was filtered through Whatman filter paper No. 42.

The toxic metal ion concentrations in the filtrate as well as in the residue were estimated using atomic absorption spectrophotometer. The calibration curves for different metal ions were plotted, by analyzing a series of standard solutions of metal ions using AAS. The different wavelength of main resonance line and air acetylene flame used for the estimation of various metals. The concentration of metal ion in filtrate was determined by the calibration curves and the percentage removal of the metal ions & their distribution coefficient ( $K_d$ ) on TASAHA resin were calculated using the following formula (Khan et al. 2009)–

$$K_d = \frac{\text{Amount of metal ion in resin phase}}{\text{Amount of metal ion in solution phase}} \times \frac{\text{Volume of solution (ml)}}{\text{Weight of dry resin (g)}}$$

i.e.  $K_d = [(I - F / F) \times V / M]$  (ml/g)

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4 Where I is the initial amount of the metal ion in solution, F is the final amount of metal  
5 ion in solution after equilibrium with resin, V is the volume of metal ion solution (ml) and M is  
6 the weight of the resin taken (g).  
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11 **Determination of percentage removal of metal ions concentration**  
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14 The initial metal ion concentration in solution and filtrate after equilibrium with resin  
15 were estimated using atomic adsorption spectrophotometer. The percentage removal of metal  
16 ions was calculated using this formula.  
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21 Percentage Removal of Metal Ions =  $[(I - F / I) \times 100]$   
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23 I = Initial concentration of metal ion in solution.  
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25 F = Final concentration of metal ion in solution after equilibrium with resin.  
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29 **Determination of Ion exchange capacity (IEC)**  
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31 Resin capacity is usually expressed in terms of equivalents per liter (eq/L) of resin or  
32 milliequivalents per dry gram of resin. 10 g of the TASAH resin were completely converted into  
33 chloride form by treatment with liberal excess of 0.1 N HCl. Then the resin was washed with  
34 distilled water. Washing were also collected in the same volumetric flask and the contents were  
35 neutralized with dil. HNO<sub>3</sub> and volume made up to 250 ml. 25 ml aliquot of the effluent were  
36 titrated against 0.11 N silver nitrate solution using potassium chromate as indicator (10 %  
37 solution). The Scientific weight capacity (Q weight) was calculated using the formula:  
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50 Effluent volume X milli equivalents of titrant used  
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53 Q weight =  $\frac{\text{Effluent volume X milli equivalents of titrant used}}{\text{Volume of aliquot X weight of dried resin}}$  meq/g  
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4 **Results and Discussion**  
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6 The Tamarind 5-Amino Salicylic Acid Hydroximate ion exchanger can be used as  
7 flocculant cum metal ions scavenger from effluent of the steel industries. Primary and  
8 secondary treatments of effluent from steel industries considerably reduce the concentration of  
9 undesired metal ions in the form of insoluble hydrous oxides followed by their adsorption and  
10 flocculation. However, such treatments still leave behind the residual metal ions concentration at  
11 a level considered unsafe for discharge into natural streams (Martin 1974). Therefore, it becomes  
12 necessary to develop a more sophisticated tertiary treatment method to remove heavy metal ions.  
13 Ion exchanger can effectively reduce the heavy metal ion concentration to a desired safe limit  
14 (Calmon et al.1973). Ion selective exchangers are also used to remove heavy and expensive  
15 metal ions from effluent containing large concentration of alkali and alkali earth metal ions  
16 (Warshawsky et al. 1980).  
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33 Ion exchange resin based on hydroxyethyl cellulose has been synthesized to remove the  
34  $Fe^{2+}$ ,  $Cu^{2+}$ ,  $Co^{2+}$  and  $Zn^{2+}$  from their aqueous solutions (Beker et al. 1999). Chitosan beads were  
35 crosslinked with a dialdehyde or tetracarboxylic acid are used for removal of  $Ni^{2+}$ ,  $Zn^{2+}$ ,  $Cd^{2+}$ ,  
36  $Mg^{2+}$  and  $Ca^{2+}$  ions (Becker et al. 2000). The use of cellulose containing agricultural by-  
37 products modified with the cross-linking reagent dimethyloldihydroxyethylene urea (DMDHEU)  
38 and the quaternary amine, choline chloride, as a anion exchange resin used to removal of arsenic,  
39 chromium, and selenium in a simulated wastewater at pH 7.  
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## Characterization of tamarind 5-amino salicylic acid hydroximate [TASAH] resin

### Spectral characterization of tamarind 5-amino salicylic acid hydroximate [TASAH] resin

FT IR spectra analysis of tamarind 5-amino salicylic acid hydroximate (TASAH) resin was employed by Perkin Elmer FT IR Instrument using KBr pellets. FT IR spectra analysis of the resin was done in H<sup>+</sup> form. The characteristic bonds observed in the spectrum of TASAH resin results from-

The peak at 2950 cm<sup>-1</sup> is attributed is to C-H stretching vibration. A strong peak in the region 1250 – 1000 cm<sup>-1</sup> denotes C-O stretching vibrations. The C-N stretching band observe at 1200-1020 cm<sup>-1</sup>, peak in the region 3500-3200 cm<sup>-1</sup> denotes >NH stretching vibration. The peak at 1700-1680 cm<sup>-1</sup> is attributed to C=O stretching vibrations of aryl amide. The peaks at 1620, 1602, 1499 cm<sup>-1</sup> are attributed to C=C stretching in aromatic nuclei. The TASAH resin shows another variable peak at 1600-1500 cm<sup>-1</sup> is attributed to C-H bending. The spectra of polysaccharide are generally observed in the region 3600-3200 cm<sup>-1</sup> due to O-H stretching frequency (Rao 1963). The IR spectrum of TASAH resin is given in **Fig 3**.

### Thermogravimetric analysis

For this purpose 951-Thermogravimetric analyzer 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<sup>0</sup>C per minute under static air atmosphere till the complete decomposition. The TASAH resin is found to stable up to 401<sup>0</sup>C and then the degradation was found to be rapid. The obtained TGA curve of TASAH resin is shown in **Fig 4**.

## Elemental analysis of TASAHA resin

The 1165 Carlo Erba Elemental analyzer was carried out for elemental analysis. The results of the elemental analysis are in good agreement with calculated value. The elemental analysis results are in support of the proposed structure of the resin presented in **Fig 2**. The results of elemental analysis are given in **Table 3**.

## Moisture contents

A physical property of the ion exchange resins with changes in cross linkage is the moisture content of the resin. The amount of cross linking depends on the proportions of different monomers used in the polymerization step. So it is important to determine the moisture contents of the synthesized resins. One gram of the resin in hydrogen form was taken and dried to a constant weight in vacuum desiccator at 75°C overnight and the resin was weighed. **Moisture percentage** = 5.9 %. The synthesized resin shows lower moisture content compared to other reported resins (Shah et al., 2003; 2004), indicating the high degree of cross-linking in this resin. The moisture content of ion-exchange resins was known to vary depending on the production batch (Peryshkina et al. 1968; Ionity: Katalog, 1989).

## Swelling studies

The Equilibrium water content (EWC) of resin is 7.9 %.

## Ion exchange capacity (IEC) of TASAHA resin

It was found to be 2.26 meq/g of the dry TASAHA resin.

## Resin durability of TASAHA resin

It was observed that the adsorbency of different metal ions on the TASAHA resin and ion exchange capacities of reported resin after 10 cycles (adsorption and desorption) was almost constant. The adsorbed metal ions were easily desorbed by treatment of different strength of

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4 acids, at room temperature. **Table 4** shows the absorbency of different metal ions on the TASA  
5 resin after 10 cycles (adsorption and desorption).  
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### 8 **Distribution coefficient ( $K_d$ ) of metal ions in effluent of Karishma Textile, Jodhpur**

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10 The pH has a strong effect on the distribution coefficient ( $K_d$ ) of metal ions. The perusal  
11 of the results shows that the distribution coefficient value first increases and then decreases with  
12 increasing pH, the optimum results obtained at pH 7.0 which is shown in **Fig 5**. Metal sorption  
13 starts when the pH rises to the range where most acidic ion exchange sites start to exchange  
14 hydronium ion for metal and the capacity reaches the maximum value in the pH range where all  
15 the ion exchange sites take part in the reaction and the functional group is able to form complex  
16 with the metal cations (Nenov et al. 2010).  
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### 28 **Statistical analysis**

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30 The relative standard deviation values (RSD) of optimum removal percentage of metal  
31 ions are shown in **Table 5**. All data represent the mean of three independent experiments. The  
32 results revealed that the relative standard deviation (RSD) of the method was lower than 4.0 %,  
33 which indicated that the method had good precision for the analysis of trace metal ions in  
34 solution samples.  
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### 43 **Quantitative separation of metal ions**

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45 The superior selectivity towards multivalent cations exhibited by chelating resin has been  
46 demonstrated in column experiments by using  $K_d$  values. The values of  $K_d$  are shown in **Table 6**.  
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48 The considerable difference in distribution coefficient of the divalent cations implies that  
49 separation of metal ion from their mixtures would be possible. An ideal situation would be such  
50 that one  $K_d$  values is greater than the  $K_d$  value for other metal ions. The first eluting fractions of  
51 hydrochloric acid carry one metal ion which has smaller  $K_d$  values. The second metal ion was  
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4 eluted by changing the hydrochloric acid concentrations. In the case of the separation of Lead  
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6 (II) from other metal ions, chelate lead (II) and other metals were separated by selective  
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8 adsorption. Pb 0 was eluted with 0.05 N HCl solution of pH 7.0 because at the same condition  
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10 other metal ions showed highest  $k_d$  values. Therefore, first few fraction contained only lead (II)  
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12 and other metal ions were eluted quantitatively with different strength of HCl solution. The  
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14 results are summarized in **Table 6**.  
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### 18 **Effect of pH**

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21 The pH is an important parameter for adsorption of metal ions from aqueous solution  
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23 because it affects the solubility of the metal ions, concentration of the counter ions on the  
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25 functional groups of the adsorbent and the degree of ionization of the adsorbent during reaction.  
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27 To examine the adsorption percentage of metal ions with pH, the pH was varied from 2.0 to 8.0  
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29 as shown in **Table 7**. The uptake of free metal ions depends on pH, where optimum adsorption  
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31 of metal ions occurs at pH 7 and then declining at higher pH. Adsorption of metal ions on  
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33 TASAHA resin increased over pH range from 2.0 to 7.0. These results are reported in **Table 7**.  
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35 After pH 9.0, the insoluble metal ions starts precipitating from the solution, making true sorption  
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37 studies impossible.  
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### 43 **Effect of agitation speed**

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45 The effect of agitation speed on adsorption of metal ions was studied by varying the  
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47 speed of agitation from 0 (without shaking) to 180 rpm, while keeping the optimum temperature  
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49 (25°C) and optimum pH as constant. It is clear from **Fig 6**, the adsorption of metal ions on ion  
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51 exchange resin generally increased with increasing agitation speed. The adsorption of metal ions  
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53 on TASAHA resin increased when agitation speed increased from 0 to 120 rpm. These results can  
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55 be associated to the fact that the increase of the agitation speed, improves the diffusion of metal  
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4 ions towards the surface of the adsorbents. This also indicates that a shaking rate in the range  
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6 120-180 rpm is sufficient to assure that all the surface binding sites are made readily available  
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8 for metal ions uptake. Then, the effect of external film diffusion on adsorption rate can be  
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10 assumed not significant. For convenience, agitation speed of 130 rpm was selected as the  
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12 optimum speed for TASAHA resin for removal of metal ions from effluent of KarishmaTextile.  
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14 These results are in close agreement with the reports by Jeon and Park (Jeon a et al. 2005).  
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### 19 **Effect of treatment time**

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21 The results of treatment time indicate that adsorption percentage of metal ions increased  
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23 with an increase in contact time before equilibrium is reached. It is clear from **Fig 7** that  
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25 adsorption of metal ions on TASAHA resin increased when contact time was increased from 30 to  
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27 240 minutes, optimum contact time for TASAHA adsorbent was found to be 240 min. Other  
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29 parameters such as pH of solution and agitation speed were kept optimum, while temperature  
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31 was kept at 25<sup>0</sup>C. Greater availability of hydroximate and hydroxyl functional groups on the  
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33 surface of TKP which are required for interaction with metal ions, significantly improved the  
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35 binding capacity and the process proceeded rapidly. This result is important, as equilibrium time  
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37 is one of the important parameters for an economical wastewater treatment system.  
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### 43 **Effect of treatment temperature**

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45 **Fig 8** shows the effect of treatment temperature on the adsorption percentage of the metal  
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47 ions on TASAHA resin. The adsorption percentage of metal ions decreases by increasing the  
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49 treatment temperature from 25<sup>0</sup>C to 50<sup>0</sup>C and than 75<sup>0</sup>C at optimum treatment time of 4 hrs. This  
50  
51 is due to the exothermic ion exchange reactions of divalent cations. The equilibrium constants  
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53 for such reaction decrease with temperature and the reaction products were not favored at high  
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4 temperatures. This observation is in full agreement with the published results by Khalil et al  
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6 (Khalil et al. 1998).  
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### 8 9 **Effect of TASAHA dose on adsorption of metal ions**

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11 The adsorption of metal ions is significantly influenced by the amount of the TASAHA  
12 resin added. The amount of TASAHA resin added into the solution determined the number of  
13 binding sites available for the adsorption. The effect of the adsorbent dose on the amount of  
14 metal ions removed was studied by the application of varying TASAHA doses. The maximum  
15 adsorption by TASAHA resin was achieved with an adsorbent dose of 0.1 g and continued  
16 decreasing up to 0.2 g. The initial increase adsorption percentage of metal ions was due to the  
17 availability of more adsorption sites. On increasing the TASAHA resin concentration further, the  
18 binding of metal ions steadily decreased. This effect might be attributed to overlapping or  
19 aggregation of adsorption sites of resin resulting in a decrease in the total surface area of the  
20 adsorbent. The results are shown in **Fig 9**.  
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### 36 **Effect of flow rate**

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38 In the column experiment, the flow rate of the sample solution was an important  
39 parameter not only affecting the recovery of metal ions, but also controlling the time of analysis.  
40 Therefore, the effect of flow rate on adsorption of metal ions were investigated under the  
41 optimum conditions (pH, eluent, etc.) by passing 100 ml of sample solution through the micro  
42 column. The flow rates were adjusted in range of 1.0 – 5.0 ml min<sup>-1</sup> controlled by a peristaltic  
43 pump. It was found that the optimum flow rate for these metal ions was 3 - 4 ml min<sup>-1</sup> for  
44 maximum loading and thereafter for stripping off from the chelating resin. Flow rates slower  
45 than 2 ml min<sup>-1</sup> were not studied to avoid long analysis times. However, at a flow rate greater  
46 than 4 ml min<sup>-1</sup>, there was a decrease in the percentage of metal ion recover, as the metal ions  
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4 probably could not equilibrate properly with the resin bed. Thus, a flow rate of  $4.0 \text{ ml min}^{-1}$  was  
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7 selected throughout the column experiment.  
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### 9 **Conclusion**

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11 The experimental results reported here in validate that TASAHA resin is a promising  
12 adsorbent for removal of heavy metal ions from industrial effluents. The  $K_d$  determinations  
13 revealed that there is a considerable difference between the distribution coefficients of metal ions  
14 at optimum condition, which can be used in the successful separation of heavy metal ions. It was  
15 found that, the TASAHA resin is effective adsorbent (almost 95 %) for the removal of different  
16 toxic metal ions from effluent of Karishma Textile, Jodhpur, India follows the order:  $\text{Zn}^{2+} >$   
17  $\text{Fe}^{2+} > \text{Co}^{2+} > \text{Cd}^{2+} > \text{Cu}^{2+}$ . The adsorption–desorption studies showed that the resin were  
18 reusable without a significant decrease in the ion adsorption capacities. Therefore the TASAHA  
19 resin is applicable for the removal and recovery of toxic metal ions from effluent of Industries. It  
20 would be interesting to use the TASAHA resin for the economic treatment of effluent containing  
21 the aforementioned metal ions.  
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### 38 **Acknowledgment**

39  
40 The Authors are thankful to Head, Department of Chemistry, J.N.V. University, Jodhpur, for  
41 providing all necessary facilities.  
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**Table caption:**

**Table 1. The characteristics features of Effluent of Karishma Textile, Jodhpur**

**Table 2. Physical property of TASAHA resin**

**Table 3. Elemental analysis of TASAHA resin**

**Table 4. The adsorption percentage of different metal ions on TASAHA resin (adsorption and desorption)**

**Table 5. Optimum results for the removal of metal ions from the effluent of Karishma Textile, Jodhpur on TASAHA resin at pH 7.0**

**Table 6. Quantitative separation of metal ions on TASAHA column**

**Table 7. Percentage removal of metal ions from effluent of Karishma Textile, Jodhpur on TASAHA resin**

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**Table -1**

**The characteristics features of Effluent of Karishma Textile, Jodhpur**

Appearance : Turbid								
Colour : Dirty brown / Green								
pH : 4.5								
Total hardness : 835 ppm								
Metal ion	Fe <sup>2+</sup>	Cu <sup>2+</sup>	Zn <sup>2+</sup>	Pb <sup>2+</sup>	Co <sup>2+</sup>	Cd <sup>2+</sup>	Mg <sup>2+</sup>	Ca <sup>2+</sup>
concentration (in ppm)	0.67	0.82	7.16	0.68	1.07	0.13	102.4	184.5
Other anions (ppm) : fluoride = 0.92 ; sulphate = 874.2 ; cyanide = 0.03								



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**Table -2**

**Physical property of TASAH resin**

<b>Property</b>	<b>Value (SD)</b>
% moisture	5.9 ± 0.6
% Soild	88.2± 0.7
True density g/cm <sup>3</sup>	2.18± 0.04
Apparent density g/ cm <sup>3</sup>	1.6216 ± 0.0063
Void volume fraction	0.5442 ± 0.1224
Volume capacity, mmol/cm <sup>3</sup>	2.97 ±0.07

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**Table -3**  
**Elemental analysis**

<b>Element</b> <b>Values</b>	<b>C</b>	<b>H</b>	<b>N</b>	<b>O</b>
<b>Calculated %</b>	45.83	5.90	2.43	45.83
<b>Found %</b>	44.91	5.78	2.38	44.91

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Table -4

The adsorption percentage of different metal ions on TASAH resin (adsorption and desorption)

Metal Ions	Adsorption percentage of different metal ions onto TASAH resin after cycles				
	1 cycle	2 cycles	4 cycles	8 cycles	10 cycles
Zn <sup>2+</sup>	95.89	93.98	93.09	92.55	92.51
Cd <sup>2+</sup>	92.86	91.85	91.15	91.10	91.05
Fe <sup>2+</sup>	93.99	92.05	91.50	91.37	91.34
Co <sup>2+</sup>	93.46	92.87	92.19	92.15	92.13
Cu <sup>2+</sup>	91.50	90.99	90.67	90.56	90.54

Table -5

Optimum results for the removal of metal ions from the effluent of Karishma Textile, Jodhpur on TASAHA resin at pH 7.0

Metal ions	Amount of metal ions in effluent	Amount loaded on TASAHA resin	% Removal	RSD %
<b>Zn<sup>2+</sup></b>	7.16	6.84	95.67	2.25
<b>Cd<sup>2+</sup></b>	0.13	0.10	84.61	3.76
<b>Co<sup>2+</sup></b>	1.07	0.98	92.52	1.46
<b>Cu<sup>2+</sup></b>	0.82	0.60	73.58	2.69
<b>Fe<sup>2+</sup></b>	0.67	0.61	92.53	3.92

Table -6

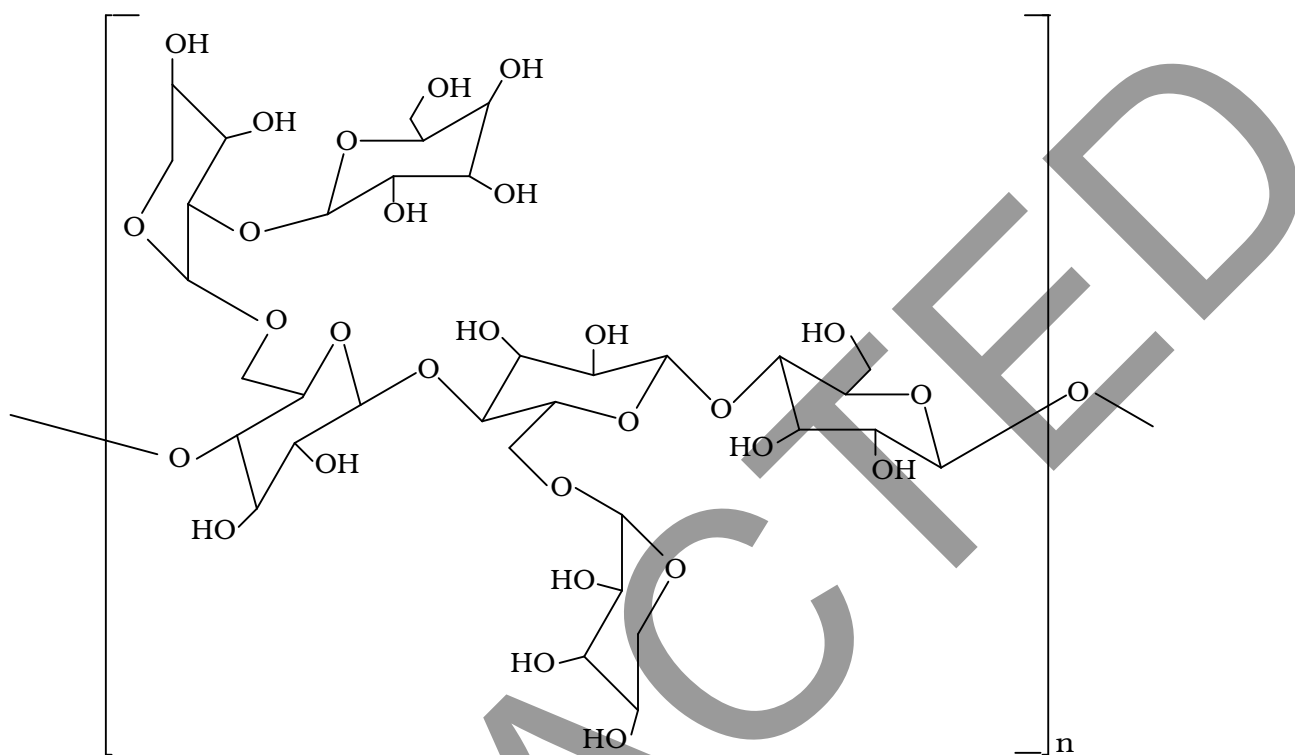
Quantitative separation of metal ions on TASAH column

Metal ion	Amount loaded	Amount found	% Recovery	Eluent use	Eluent volume
<b>Zn<sup>2+</sup></b>	6.84	6.79	99.27	0.5 N HCl	50
<b>Cd<sup>2+</sup></b>	0.10	0.09	90.90	0.5 N HCl	55
<b>Co<sup>2+</sup></b>	0.98	0.94	95.95	0.05N HCl	50
<b>Cu<sup>2+</sup></b>	0.60	0.58	97.17	1 N HNO <sub>3</sub>	45
<b>Fe<sup>2+</sup></b>	0.61	0.61	100.00	0.5 N HNO <sub>3</sub>	40

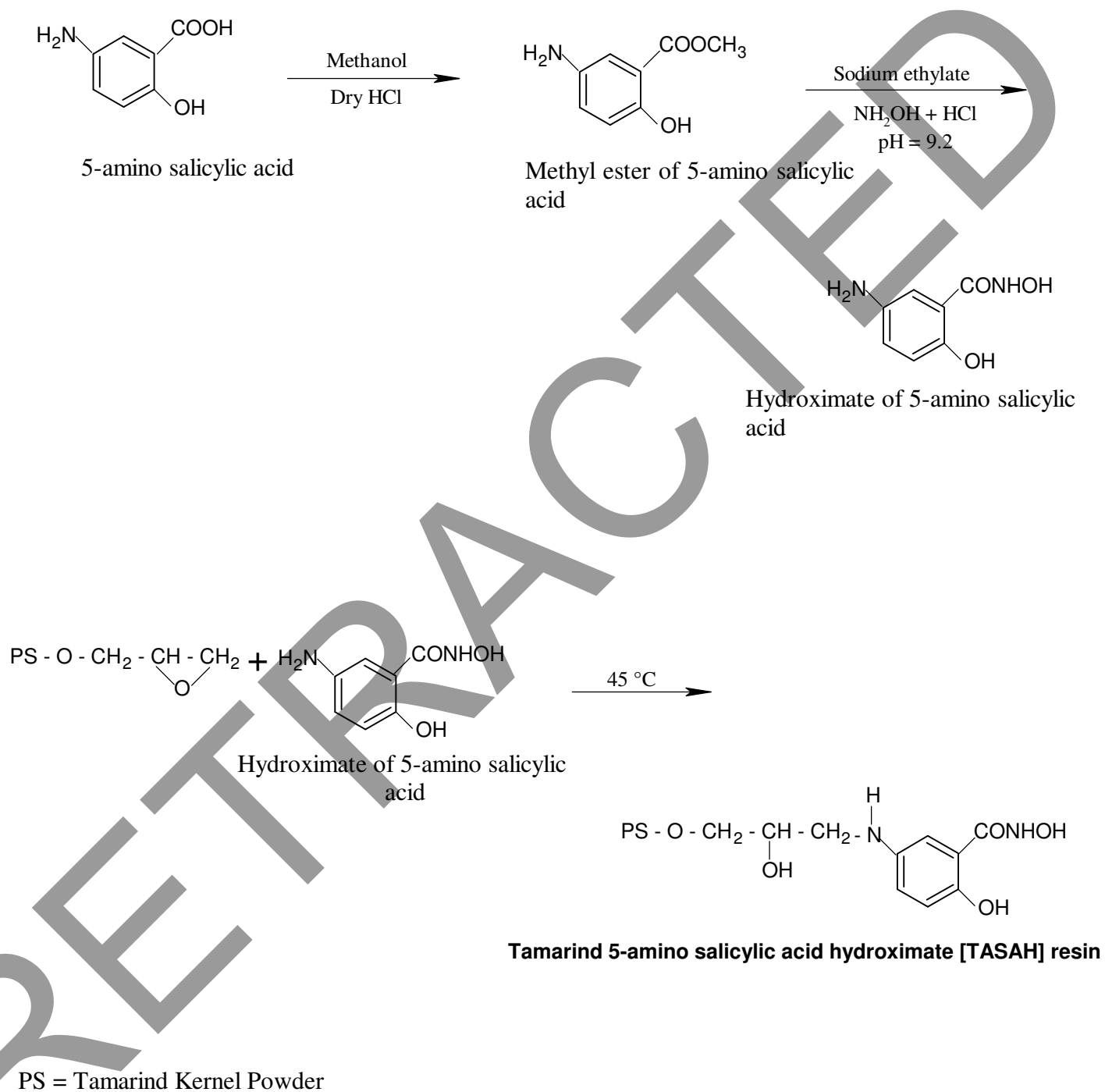
Table -7

Percentage removal of metal ions from effluent of Karishma Textile, Jodhpur on TASAH resin

pH	Zn <sup>2+</sup>	Cd <sup>2+</sup>	Fe <sup>2+</sup>	Co <sup>2+</sup>	Cu <sup>2+</sup>
2	75.43	79.20	34.98	75.28	69.50
3	80.55	79.82	42.78	76.92	71.48
4	86.28	80.55	66.02	76.98	78.66
5	87.11	80.65	75.98	77.40	79.50
6	89.74	84.75	81.04	84.75	85.48
7	95.89	92.86	93.99	93.46	91.50
8	85.47	78.87	62.48	79.05	73.68

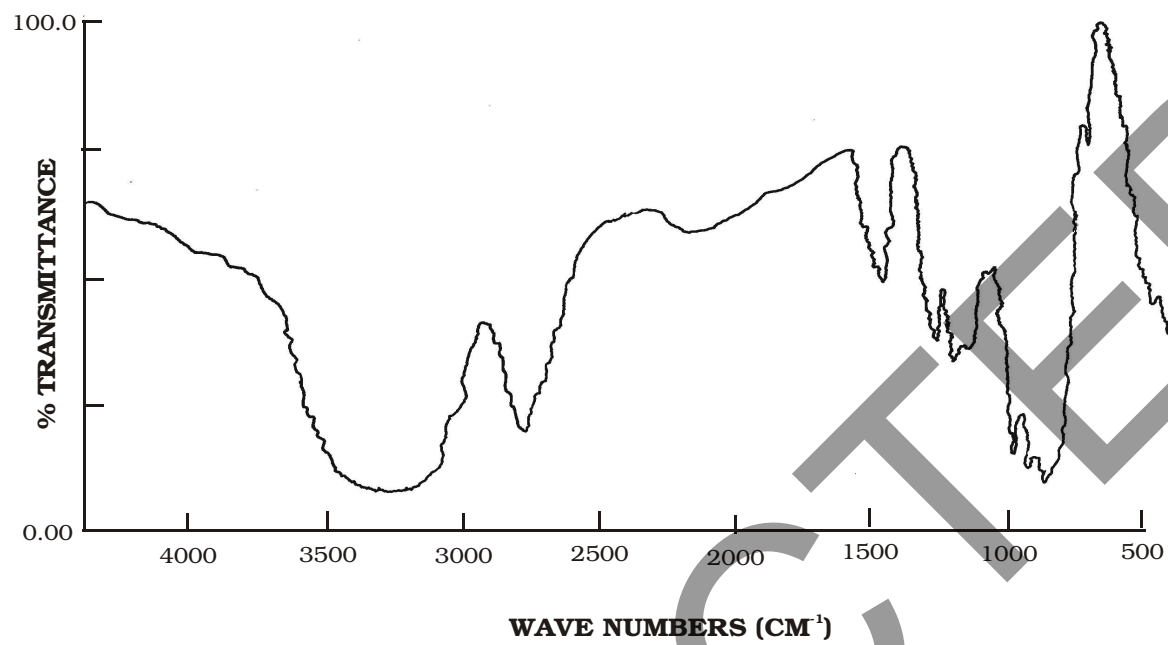


**Fig 1. Average primary structure of Tamarind seed polysaccharide. The cellulose type backbone is substituted by xylose (a 1-6) and galacto (8 1-2) xylose (cu 1-6) residues**

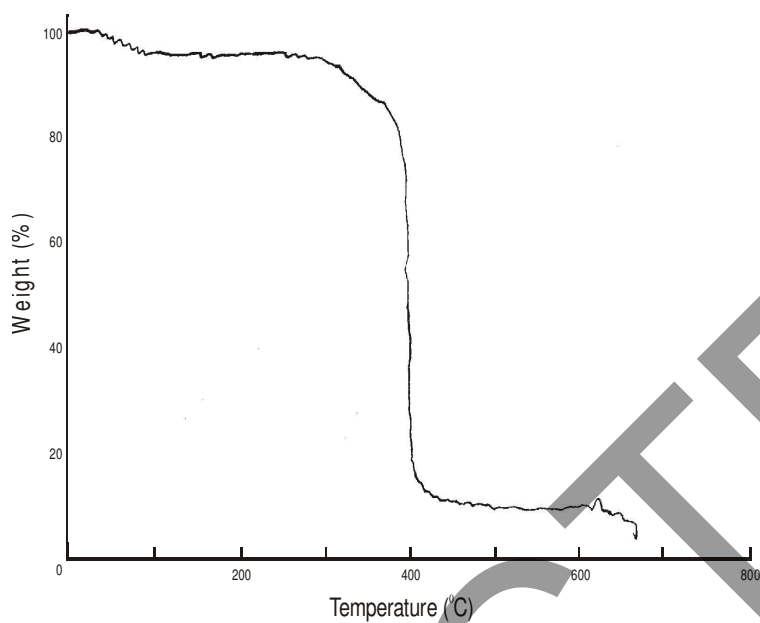


**Fig 2. Synthesis of Tamarind 5-amino salicylic acid hydroximate [TASAHA] resin**





**Fig 3. FT IR of Tamarind 5-amino salicylic acid hydroximate [TASAH] resin**



**Fig 4. TGA Curve of TASA resin**

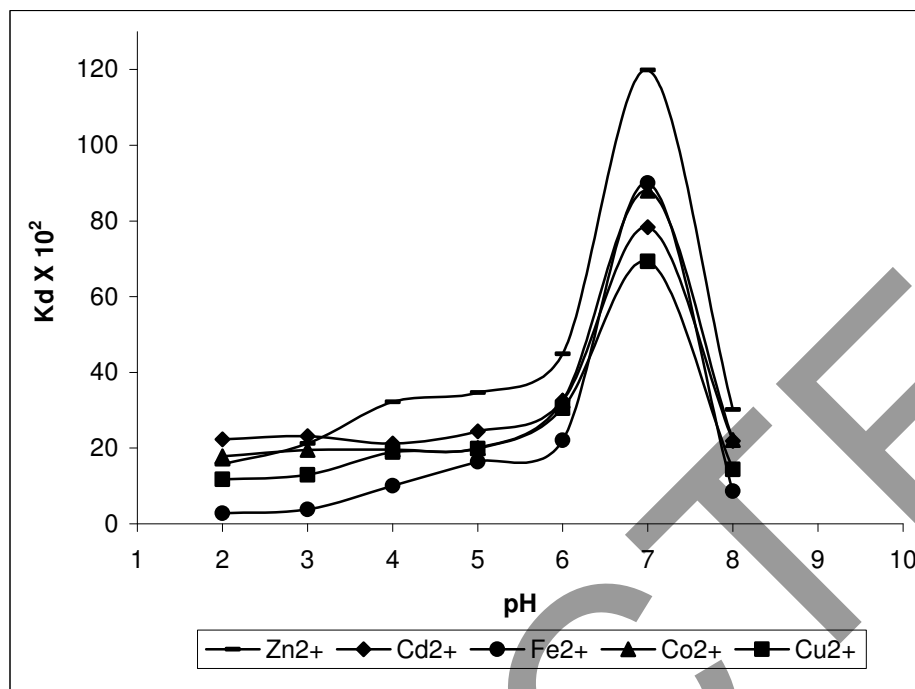
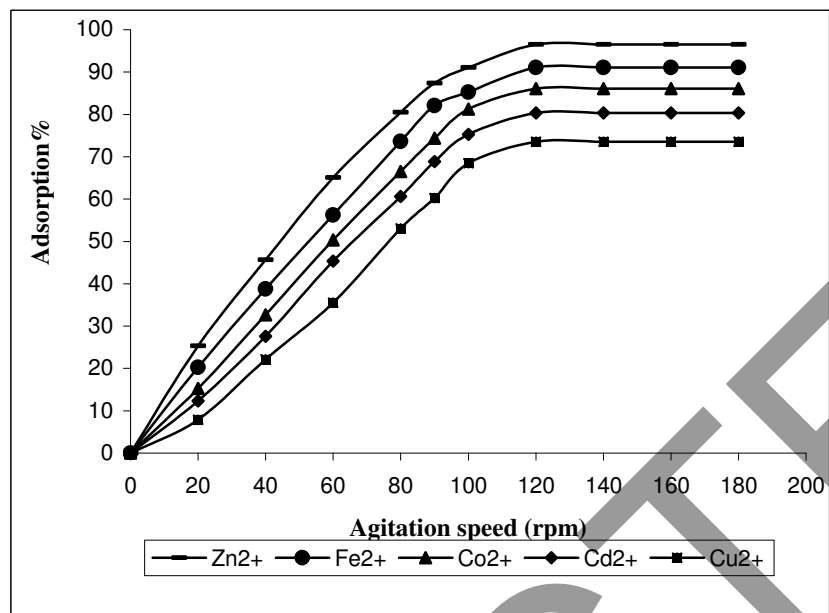
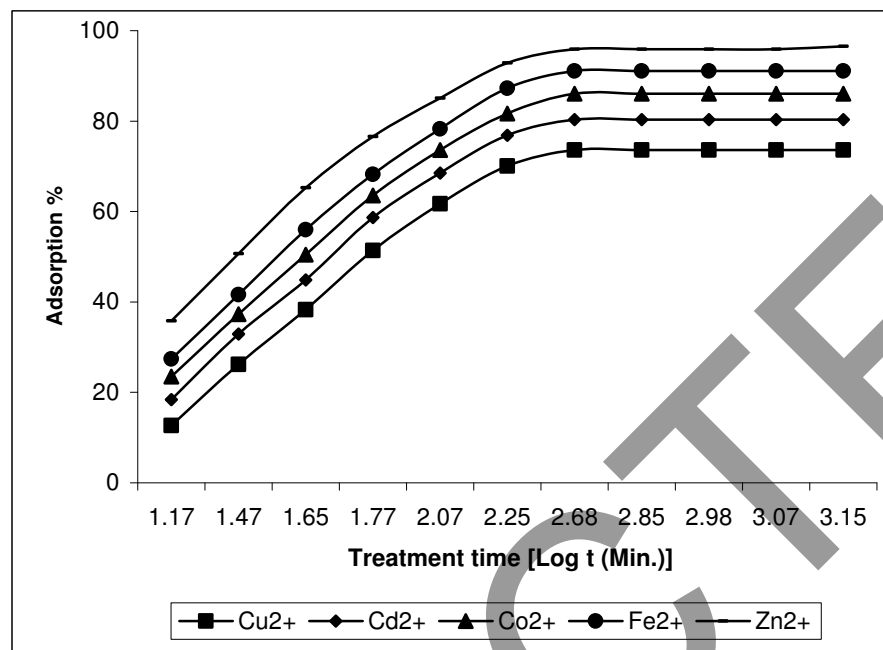


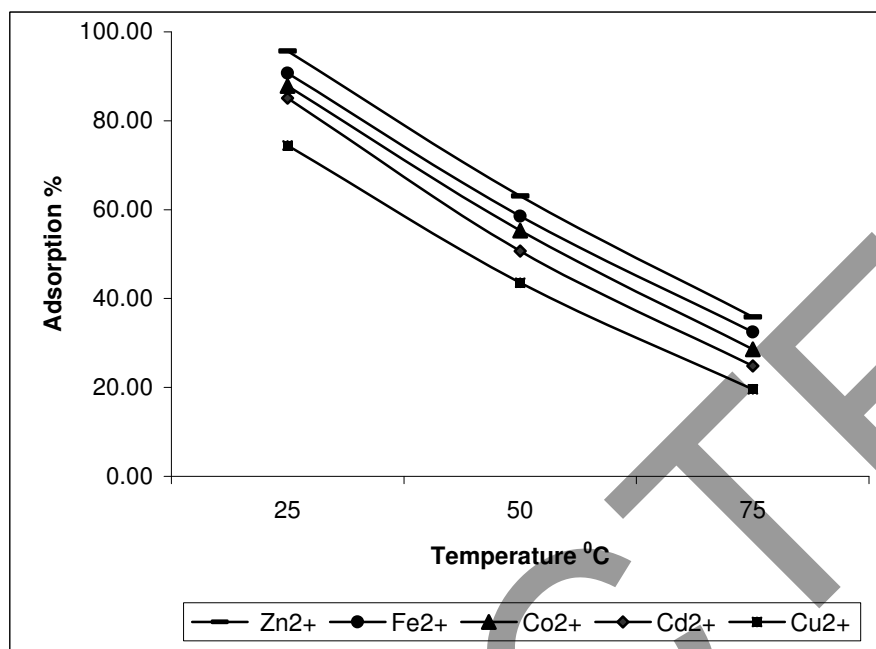
Fig 5.  $K_d$  value (Distribution Coefficient) of metal ions on TASA resin



**Fig 6. Effect of agitation speed on adsorption of metal ions on TASA resin**



**Fig 7. Effect of changing treatment time on the adsorption percentage of different metal ions on TASA resin**



**Fig 8. Effect of changing treatment temperature on the adsorption percentage of different metal ions on TASA resin**

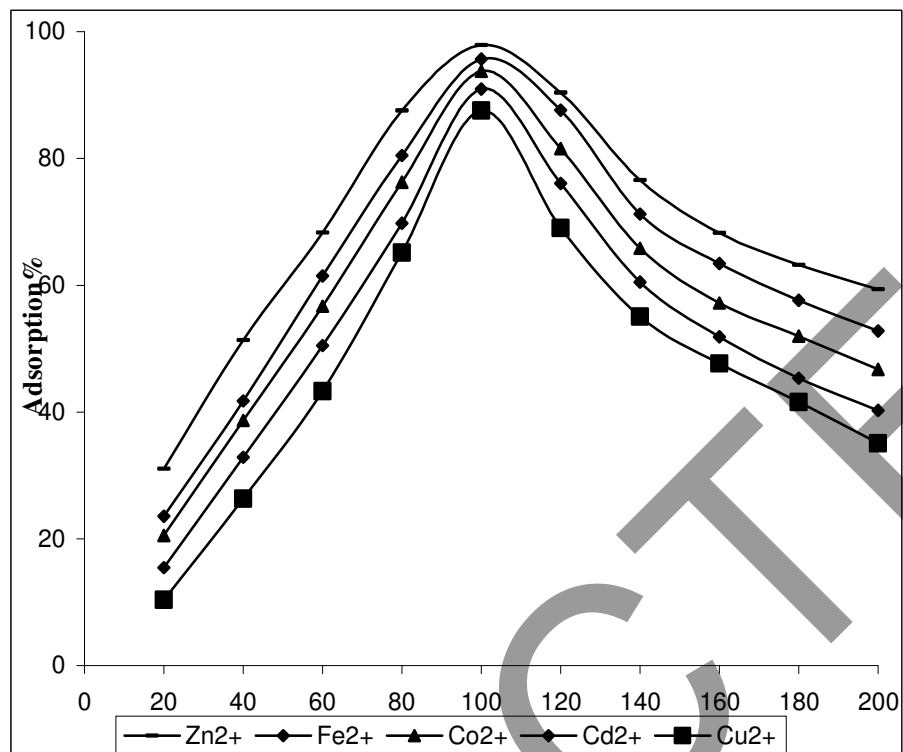


Fig 9. Effect of adsorbent dose on adsorption of metal ions on TASA resin

**Fig caption:**

- Fig 1** Average primary structure of Tamarind seed polysaccharide. The cellulose type backbone is substituted by xylose (a 1-6) and galacto (8 l-c2) xylose (cu 1-6) residues
- Fig 2** Synthesis of Tamarind 5-amino salicylic acid hydroximate [TASAH] resin
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- Fig 4** TGA Curve of TASAH resin
- Fig 5**  $K_d$  value (Distribution Coefficient) of metal ions on TASAH resin
- Fig 6** Effect of agitation speed on adsorption of metal ions on resin
- Fig 7** Effect of changing treatment time on the adsorption percentage of different metal ions on TASAH resin
- Fig 8** Effect of changing treatment temperature on the adsorption percentage of different metal ions
- Fig 9** Effect of adsorbent dose on adsorption of metal ions on TASAH resin