



Synthesis, Characterization and Application of Green Chelating BE-TH Resin for the Treatment of Industrial Effluents: A Novel Approach.

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KEYWORDS

BE-TH resin, green chelating resin, heavy metal removal, industrial effluents.

ABSTRACT:

Industrial effluents containing heavy metals pose a significant threat to the environment and human health. In this study, a green chelating resin BE-TH was synthesized using barley as a natural biopolymer. The resin was developed to provide an efficient, sustainable, and cost-effective solution for heavy metal removal from industrial wastewater. The synthesis involved chemical modification of barley to enhance its chelating properties.

The resin was characterized using FT-IR, TGA-DTA, and SEM. The FT-IR analysis confirmed the successful incorporation of chelating functional groups, TGA demonstrated its thermal stability up to 300°C, and SEM revealed a porous structure conducive to effective adsorption. The % removal efficiencies of the resin were observed under various conditions with effect of pH, contact time, and resin dosage as well as their practical application to the industrial waste water was conducted. The result showed that the BE-TH resin efficiently removed heavy metals such as Ni(II), Cu(II), and Zn(II), with over 70% removal efficiency with respect to resin dosage. The study demonstrated the potential of BE-TH resin as an innovative and eco-friendly material for industrial effluent treatment, promoting green chemistry principles and resource valorization.

List of abbreviations:

AAS - Atomic Absorption Spectrometer

BE-TH- Barley Epoxy Tyrosine Hydroxamate

FT- IR - Fourier transform infrared spectroscopy

SEM- Scanning electron microscope

TGA-DTA- Thermo gravimetric Analysis- Differential Thermal Analysis

Introduction:

Industrial effluents containing heavy metals and other toxic contaminants are a major environmental concern due to their harmful effects on aquatic ecosystems and human health. Effective treatment of industrial wastewater is critical to mitigate these impacts. Among various remediation methods, green chelating resins have gained attention for their ability to selectively bind and remove metal ions. However, the use of synthetic resins raises environmental concerns, driving the need for

sustainable alternatives [1-5]. Green chelating resins, derived from natural biopolymers, offer an eco-friendly solution to wastewater treatment. Barley, a readily available and renewable agricultural resource, presents a promising base material for the development of such resins due to its abundance of hydroxyl and carboxyl functional groups, which enhance its chelating capacity [6-10]. Chelating resins have attracted interest over time due to their high selectivity in metal removal compared to conventional cation exchangers [11-13].

This study aimed to synthesize a novel barley-based chelating resin, termed BE-TH (Barley Epoxy Tyrosine Hydroxamate), and evaluated its potential for industrial effluent treatment. The resin is synthesized using the amino acid Tyrosine, which is a bio-based, biodegradable, and non-toxic compound [14,15]. Amino acids are characterized by their amino (-NH₂) and carboxyl (-COOH) functional groups, along with a unique side chain (-



R) for each amino acid [16]. Functional macromolecular materials derived from biological chiral resources like amino acids have gained significant attention due to their biocompatibility and biodegradability, which help alleviate ecological concerns [17]. The research involved characterizing the resin's structural, thermal, and chemical properties and assessing its efficiency in removing heavy metals from wastewater. By utilizing barley, this work not only promotes the valorization of an agricultural by-product but also contributed to the development of sustainable, cost-effective solutions for industrial pollution control. The growing interest in using agricultural by-products for resin production is largely due to their high content of cellulose and hemicellulose. These

components contain substantial amounts of readily available hydroxyl groups, which facilitate various chemical reactions like esterification, etherification, and copolymerization [18-20]. This approach aligns with global efforts to implement green technologies aimed at environmental protection and resource conservation.

To the best of our knowledge synthesis, characterization and application of barley-based BE-TH resin for the treatment of industrial wastewater is a novel approach. So far no research has been reported on the above mentioned research title. The graphical abstract of the research is shown in the figure-1.

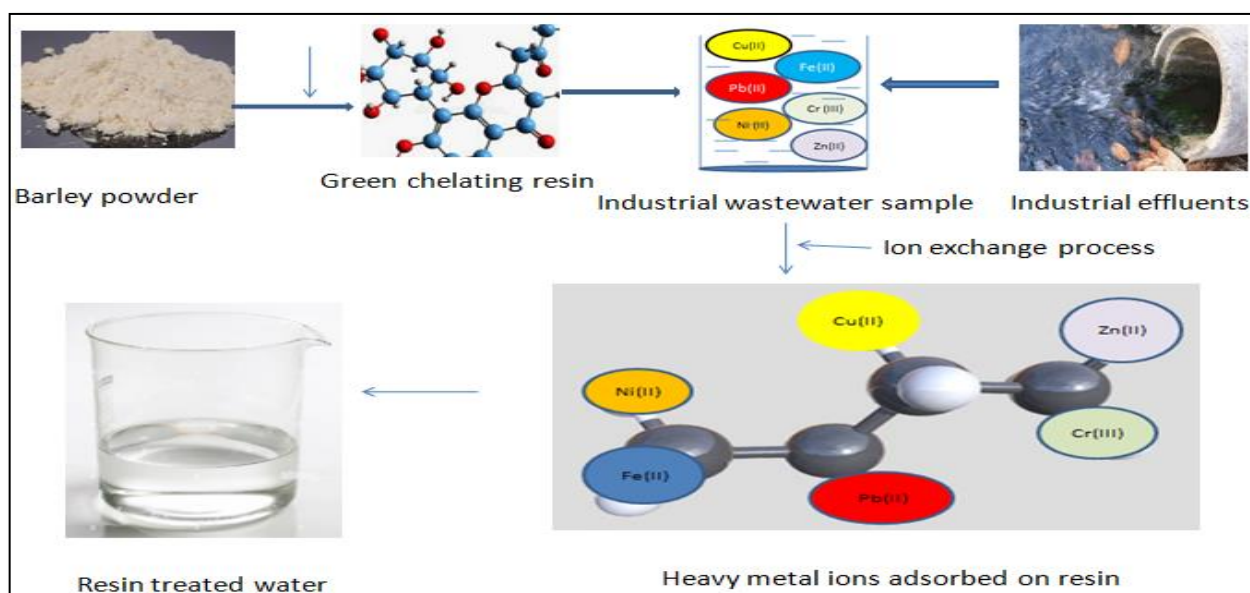


Figure 1: Graphical abstract for the present research work

Materials and Methods:

Barley was procured from WeikField Foods Pvt. Ltd., Pune, India, while epichlorohydrin was obtained from Loba Chemicals Pvt. Ltd., Mumbai, India. All other chemicals used in the experiments, including those of analytical reagent (A.R.) grade, were sourced from Sigma, E. Merck, Loba Chemicals, and Asses Chemicals.

Instrumentation:

Metal analysis was conducted using a Perkin-Elmer 2380 AAS. FT-IR spectra of the synthesized resin were recorded using a Shimadzu IR 400 spectrophotometer. The morphological characteristics and surface chemistry of the BE-TH resin were examined SEM using by using a scanning electron microscope model 30kV EVO-18 Carl Zeiss. Thermal stability was evaluated through TGA-DTA.



Sample collection: The sample solution to be studied the various reaction parameters such as effect of pH and contact time were prepared as follows:

Single metal chloride solutions were prepared by dissolving respective salts in distilled water. Stock solutions were standardized via atomic absorption spectrophotometry. Nickel, copper, chromium, zinc, iron and lead solutions contained 5 g/L metal ions. The pH

values were measured using a Digital pH meter Model – 335 of Systronics, India. The resin was rinsed thoroughly with water to remove residues. In order to understand the practical applications of the BE-TH resin, industrial effluent samples were collected from four different industries represented as A,B,C and D from industrial area, Jodhpur, Rajasthan. The % removal efficiency was calculated by using the given formula:

$$\text{Removal (\%)} = \frac{(C_i - C_f)}{C_i} * 100$$

Where

C_i = initial metal ion concentration.

C_f = the final metal ion concentration.

Synthesis of Barley Epoxy Tyrosine Hydroxamate resin (BE-TH)

- 1. Synthesis of Cross-linked Barley:** Barley powder was soaked in dioxane, followed by the addition of epichlorohydrin and NaOH. The mixture was shaken at 50–55°C for 5 hours, filtered, washed with dioxane, methanol, and nitric acid, then dried. The resulting cross-linked Barley was used for derivatization.
- 2. Synthesis of Barley Epoxy Tyrosine:** Epoxy Barley was stirred with NaOH and Tyrosine in dioxane at 50–55°C for 5 hours. The product was filtered

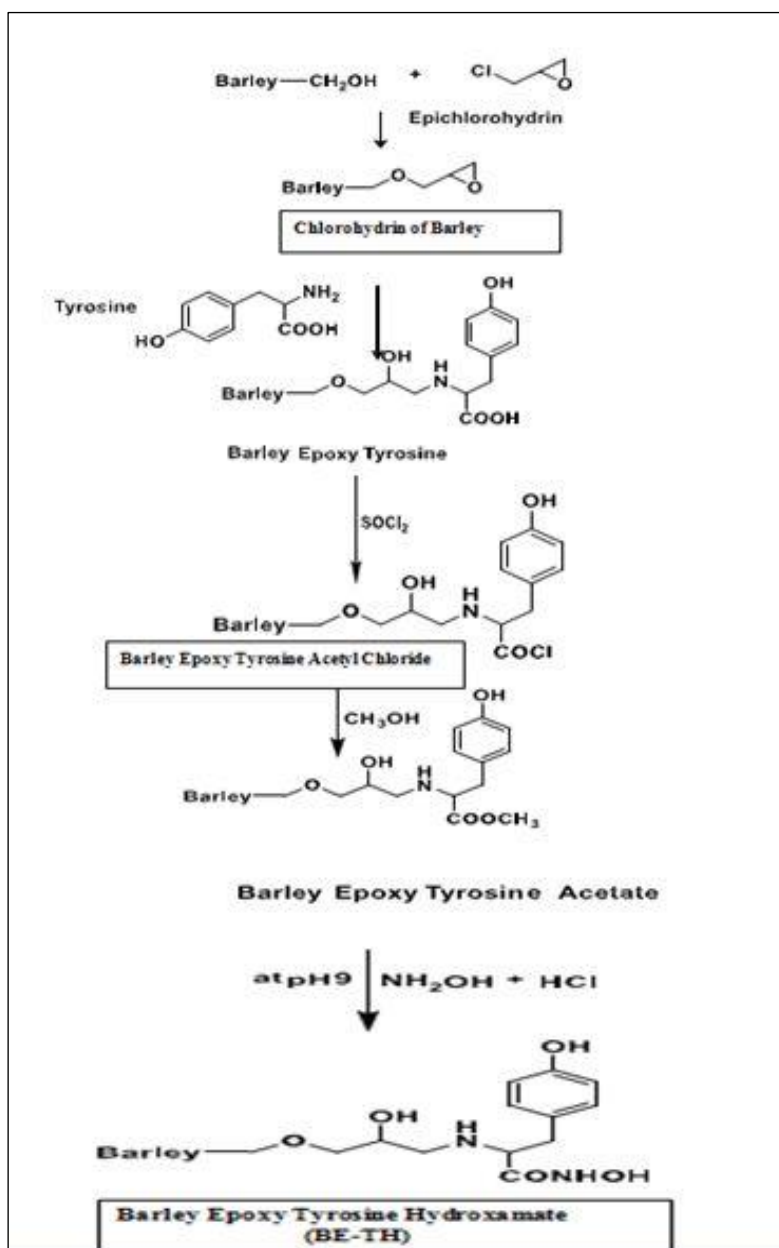
and washed with methanol and nitric acid to remove impurities.

3. Synthesis of Barley Epoxy Tyrosine Hydroxamate Resin (BE-TH):

Barley Epoxy Tyrosine was reacted with thionyl chloride, hydroxylamine hydrochloride, and sodium bicarbonate at pH 9. The product was filtered, washed, and dried to obtain BE-TH resin. The synthesized green chelating BE-TH resin is shown in the figure-1. The methodology for the synthesis of BE-TH resin is shown in the scheme-I



Figure 1: BE-TH resin



Scheme-I: Synthesis of BE-TH resin

Result and discussion:-**Analysis of Physicochemical characteristics of BE-TH resin:**

The Physicochemical characteristics of BE-TH resin are shown in Table 1. BE-TH exhibited the lowest nitrogen content (2.24%). Studies by Abdullah N et al. has demonstrated that higher nitrogen content in resins generally correlates with better ion exchange and

chelation properties due to the presence of nitrogen-based donor sites [21]. BE-TH exhibited the highest bulk density (0.922 g/cm³), reflecting a compact and denser structure, which can influence resin packing in industrial applications. BE-TH displayed the highest ion exchange capacity (5.96 meq/g). This makes BE-TH the most effective for applications requiring high exchange efficiency indicating its suitability for applications requiring higher ion exchange rates.



Table 1: Physicochemical characteristics of BE-TH resin

Resin	% Nitrogen Content	Bulk density (g/cm ³)	% Moisture content	Ion exchange capacity (meq/g)
BE-TH	2.24%	0.922	8%	5.96

FT-IR Analysis:

The infrared spectrum of BE-TH (Figure 2) was recorded and it was observed that few peaks disappeared. Every peak is shifted due to changes in the chemical environment. Peak position and assignments of the IR spectra of BE-TH are the following:

- 3280.1cm⁻¹: N-H stretching in –NH-OH (Most informative peak: appeared). Indicates the formation of Hydroxamate.
- 2820.0cm⁻¹: Because of O-H stretching in –the NH-OH group.
- 1640.0cm⁻¹: Because of -CO stretching in –CONH.
- 2113.4cm⁻¹: Because of –CH₂- methylene bridge stretching.
- 1408.9cm⁻¹, 1384.2 cm⁻¹: Because of C=C

stretching in aromatic nuclei.

- 991.5cm⁻¹: Because of –C-O-C of oxirane (epoxy) group.
- 1077.2 cm⁻¹: Because of C-N stretching in primary aromatic amines.
- FT-IR analysis of all the BE-TH resin confirms the presence of the following common groups (besides many other functional groups present in individual resins):
 - (i) Hydroxamate group (a peak at approx.3280 cm⁻¹ corresponding to –OH stretching in NHOH).
 - (ii) Epoxy group (a peak at approx. 991 cm⁻¹ corresponding to the –C-O-C of oxirane (epoxy) group).

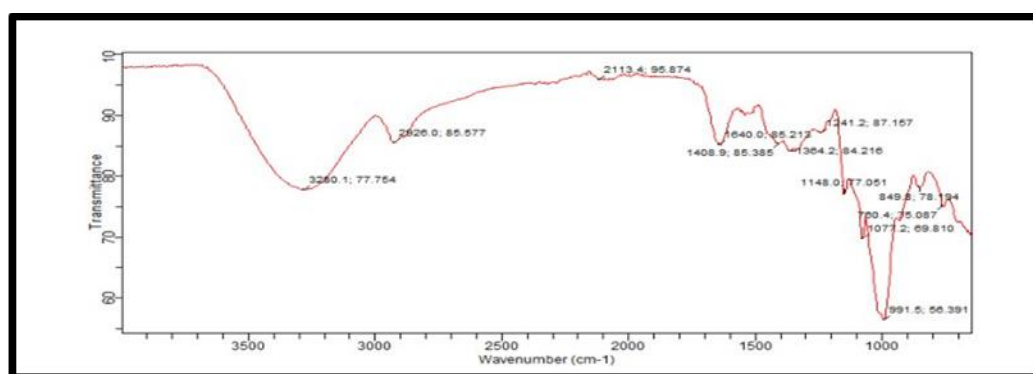


Figure 2: FT-IR Spectra of BE-TH resin

SEM Analysis:

The surface topography of BE-TH resin was studied by SEM technique, which is shown in the figure 3. The SEM analysis of BE-TH resin is shown in Figure 3 revealing a macroporous surface with irregular and uneven particle

sizes. The pores varied in size, indicating a heterogeneous texture. This macroporous structure enhances the resin's ability to adsorb metal ions by providing a larger surface area and multiple binding sites. The larger pores facilitate the diffusion of ions into



deeper layers, while smaller pores may improve surface adsorption [22]. The uneven particle size and pore distribution contribute to the resin's overall effectiveness in metal ion removal, making it suitable for wastewater treatment applications. BE-TH exhibited a macroporous

surface. This unique surface characteristic increases surface area and provides more active sites, improving adsorption efficiency. Overall, the resin's surface structure plays a crucial role in enhancing its suitability for metal ion removal applications [23].

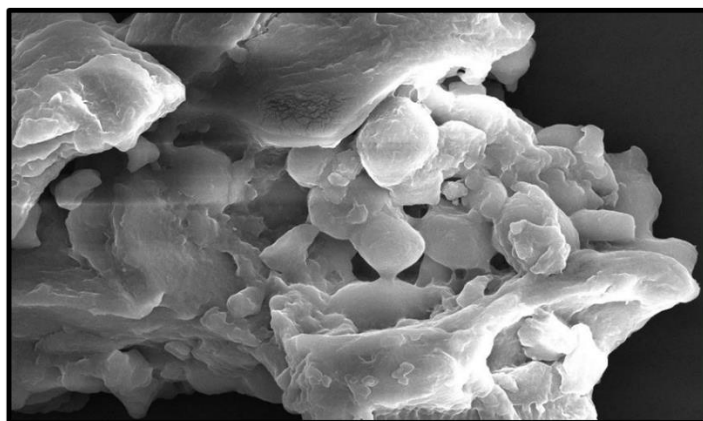


Figure 3: SEM image of BE-TH resin

TGA-DTA analysis:

The thermal stability of BE-TH was evaluated using TGA-DTA analysis. TGA-DTA curves are given in Figures 4 and 5. The TGA curve of BE-TH resin (figure 4) shows a fast weight loss of 5% up to 70°C due to the removal of external water molecules. Slow weight loss of the material is seen from a temperature of approximately 150°C which may be because of the loss of the ring and the weight loss is 3%. Fast weight loss of the material

is seen from a temperature of approximately 220°C which may be due to the decomposition of the hydroxamate group and the weight loss is 44%. The BE-TH resin shows the decomposition of barley molecules at the temperature of 560°C. In Figure 5 the DTA curve exhibited three distinct endothermic peaks at 70°C, 150°C, and 220°C. These correspond to moisture loss, decomposition of unfunctional groups, and thermal degradation, respectively [24].

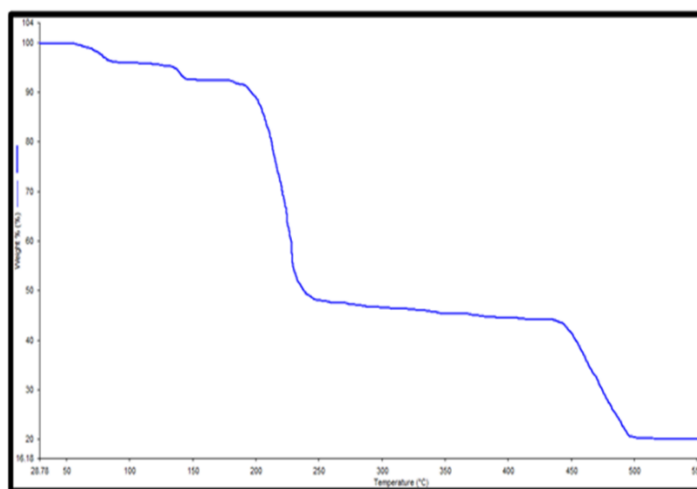


Figure 4: TGA curve for BE-TH resin

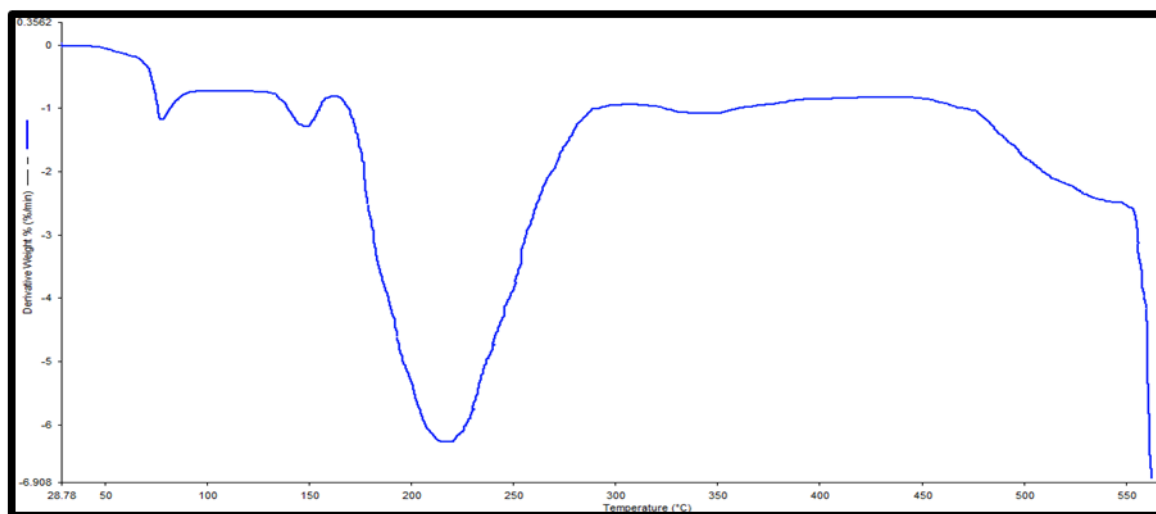


Figure 5: DTA curve for BE-TH resin

Factors affecting % removal of metal ions by the resins:

The present research focused on the factors affecting on % removal of metal ions by the resin such as effect of pH, effect of contact time and effect of resin dosage.

Effect of pH on % removal of metal ions by BE-TH resin

The effect of pH on % removal of metal ions by BE-TH resin is shown in Table 2 and figure 6. BE-TH resin

showed maximum metal ion removal at pH 4–6, with Ni(II) achieving the highest removal (82%) at pH 6, followed by Cu(II) (72%), Zn(II) (65%), and Cr(III) (54%). Pb(II) (35%) and Fe(II) (28%) showed lower efficiency as shown in the table 2 and figure 6. Lower pH reduced adsorption due to H⁺ competition, while higher pH caused metal hydroxide precipitation [25,26]. These results highlighted the critical role of pH in optimizing BE-TH resin performance for wastewater treatment.

Table 2: Effect of pH on % removal of metal ions by BE-TH resin

pH value	% Removal of metal ions					
	Cr(III)	Pb(II)	Cu (II)	Ni(II)	Zn(II)	Fe(II)
3	52	26	56	60	54	22
4	54	30	62	64	58	24
5	46	32	68	76	62	25
6	49	35	72	82	65	28
7	42	28	66	73	56	26
8	40	25	62	72	52	24

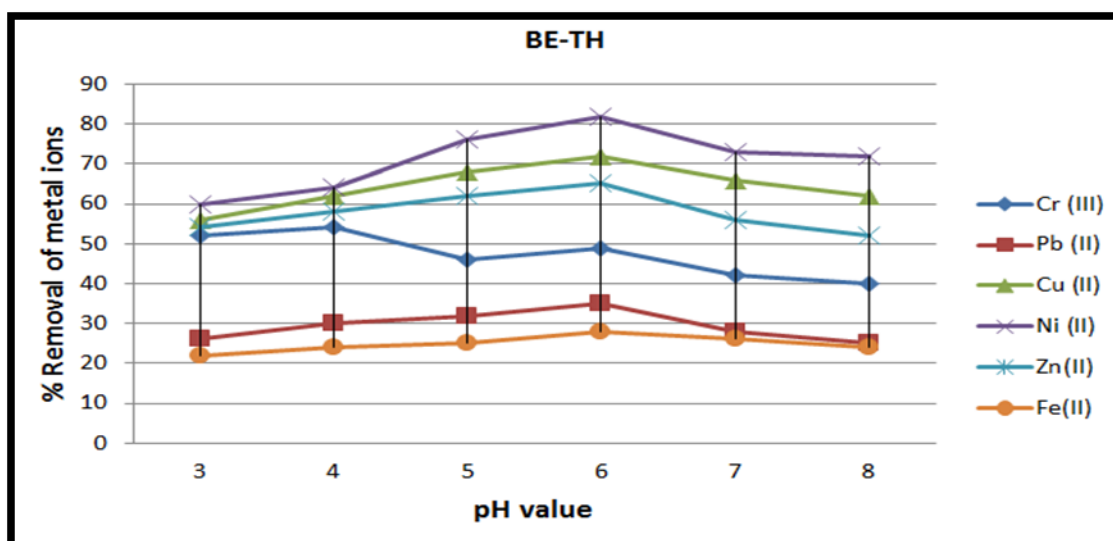


Figure 6: Effect of pH on % removal of metal ions by BE-TH resin

Effect of contact time on % removal of metal ions by BE-TH resin:

The influence of contact time on metal ion removal by BE-TH resin is summarized in table 3 and figure 7. Cr(III) removal reached 52% at 60 minutes, which is lower than the 96.7% removal of Cr(VI) achieved by ion-exchange resin in a previous study [27]. Pb(II) removal was 32% at 90–120 minutes, with slight improvement over time. Cu(II) peaked at 74% removal at 60 minutes

before declining. Ni(II) showed maximum removal efficiency of 82% at 90 minutes, followed by a decrease. Zn(II) reached 62% removal at 120 minutes, indicating a strong affinity, although this was lower than the 83% removal by zeolite X reported earlier [28]. Fe(II) exhibited a gradual increase, achieving 30% removal at 180 minutes. Overall, the results indicated that BE-TH resin demonstrated optimal removal efficiency within the initial 60–120 minutes, with a notable preference for Ni(II) and Zn(II)

Table 3: Effect of BE-TH resin contact time on % removal of metal ions:

Contact time(in min)	Cr(III)	Pb(II)	Cu(II)	Ni(II)	Zn(II)	Fe(II)
30	50	28	54	40	48	22
60	52	30	74	60	44	24
90	48	32	62	82	52	25
120	47	32	58	54	62	28
180	42	24	48	44	46	30

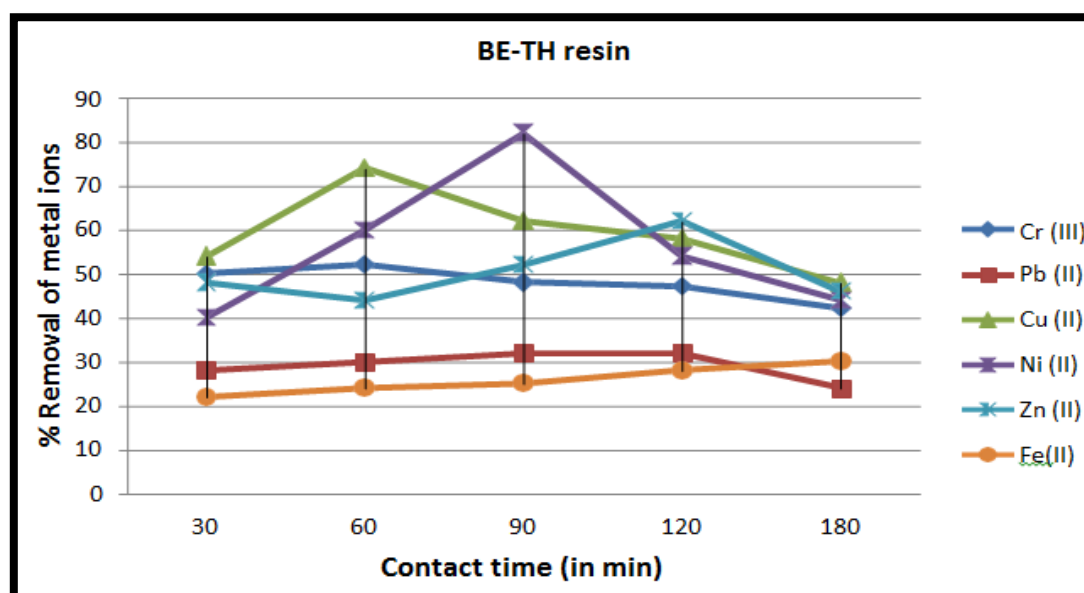


Figure 7 : Effect of contact time on % removal of metal ions by BE-TH Resin

Effect of BE-TH Resin Dosage on % Removal of Metal Ions

Increasing BE-TH resin dosage improved metal ion removal efficiency (Table 4, Figure 8). Cr(III) and Pb(II) reached around 50% removal at 5 g, with Cr(III) increasing to 58% at 5 g, comparable to tamarind seed-based adsorbents [29]. This trend aligns with increased surface area and biosorption sites, as noted by Kashefi et al. [30]. Similar

results were reported by Garg et al., where Jatropha oil cake and sugarcane bagasse achieved 97% and 92% Cr(VI) removal at 20 g/L [31]. Cu(II) achieved 80% removal at 5 g, Ni(II) reached 85%, Zn(II) peaked at 77% at 4–5 g, and Fe(II) reached 40% at 5 g. Increased resin dosage enhanced efficiency, with Ni(II) and Cu(II) showing the highest removal, followed by gradual improvements for Pb(II), Cr(III), and Fe(II).

Table 4: Effect of BE-TH resin dosage on % removal of metal ions:

Dosage (in g)	% Removal of metal ions					
	Cr(III)	Pb(II)	Cu (II)	Ni(II)	Zn(II)	Fe(II)
0.5	32	32	68	70	60	22
1	36	34	69	72	61	24
1.5	38	36	70	73	63	26
2	40	38	72	75	65	28
2.5	44	39	74	78	70	30
3	50	40	75	80	71	32



3.5	52	42	76	81	73	34
4	54	44	78	83	75	38
4.5	56	48	79	84	77	38
5	58	50	80	85	77	40

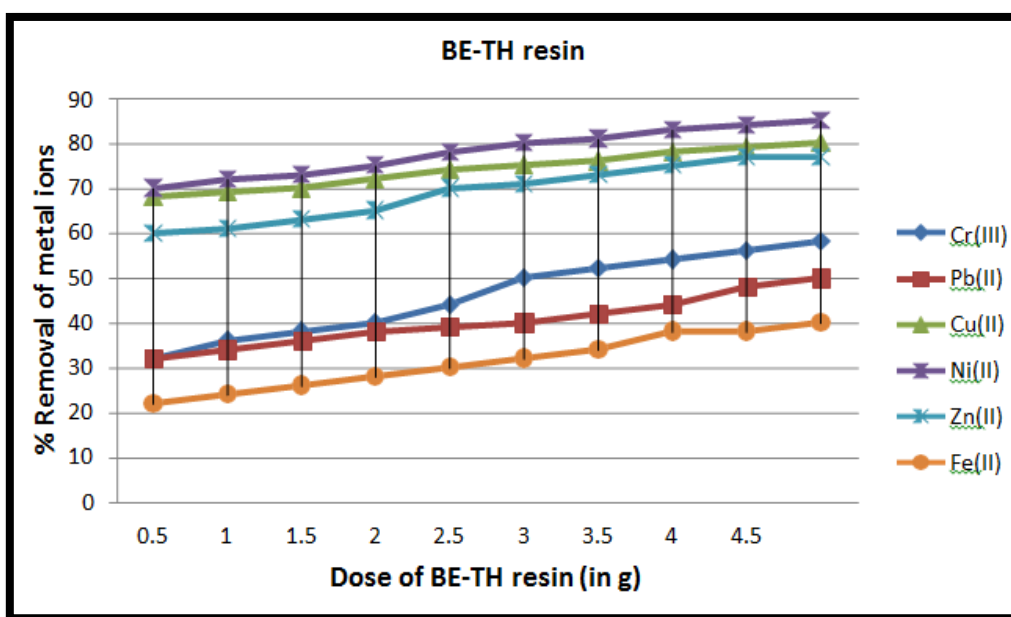


Figure 8: Effect of BE-TH resin dosage on % removal of metal ions

Applications of Synthesized BE-TH Resin for the Treatment of Industrial Effluents: The efficiency of the newly synthesized green chelating BE-TH resin in removing heavy metal ions from industrial effluents was evaluated using wastewater samples from four industries in the Jodhpur Industrial Area (Industries A, B, C, and D). The results are shown in Table 5 and Figure 10 as follows:

- **Industry A:** Highest removal: Ni(II) (85%)
- **Industry B:** Highest removal: Ni(II) (86%)

- **Industry C:** Highest removal: Cu(II) (83%), Ni(II) (82%), and Zn(II) (76%)
- **Industry D:** Highest removal: Ni(II) (82%)

BE-TH resin demonstrated consistent performance across industries, particularly for Ni(II), Cu(II), and Zn(II), with removal efficiencies exceeding 70% in most cases. Lower efficiencies for Fe(II) and Pb(II) were likely due to variations in ionic size, binding affinities, or competitive adsorption.

Table 5-Analysis of treated wastewater effluent from Industry A,B,C,D with BE-TH resin:

INDUSTRY	Metal ions	Metal Concentration (in ppm) of untreated effluents	Metal Concentration (in ppm) of effluents treated with BE-TH resin	% Removal efficiency of resin



INDUSTRY A	Cr(III)	0.05	0.025	50
	Pb(II)	0.02	0.014	30
	Cu(II)	38	11.4	70
	Ni(II)	53	7.95	85
	Zn(II)	0.65	0.26	60
	Fe(II)	26	17.68	32
INDUSTRY B	Cr(III)	0.02	0.008	60
	Pb(II)	0.07	0.0385	45
	Cu(II)	78	14.04	82
	Ni(II)	44	6.16	86
	Zn(II)	0.18	0.045	75
	Fe(II)	18	10.44	42
INDUSTRY C	Cr(III)	0.08	0.0304	62
	Pb(II)	0.35	0.1575	55
	Cu(II)	58	9.86	83
	Ni(II)	64	11.52	82
	Zn(II)	0.65	0.156	76
	Fe(II)	5.75	0.335	42
INDUSTRY D	Cr(III)	0.04	0.0156	61
	Pb(II)	0.14	0.0658	53
	Cu(II)	62	15.5	75
	Ni(II)	42	7.56	82
	Zn(II)	3.1	0.868	72
	Fe(II)	22	14.3	35

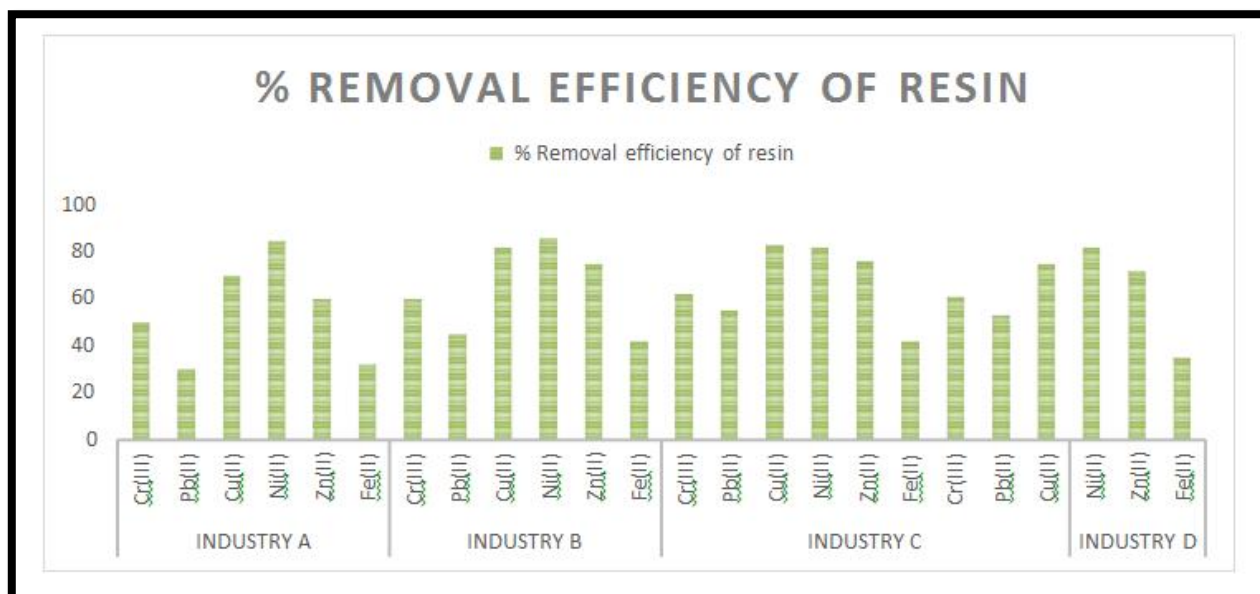


Figure 10: % Removal efficiency of metal ions from Industry A, B, C and D with BE- TH resin

Conclusion:

The study demonstrated the effectiveness of BE-TH resin in removing heavy metal ions, particularly Ni(II), Cu(II), and Zn(II), with high removal efficiencies observed in both laboratory and industrial effluent applications. The resin proved adaptable under varying pH, contact time, and dosage conditions, making it suitable for industrial wastewater treatment. The green chelating BE-TH resin provided eco-friendly, cost-effective solutions. The study also emphasized the scalability of BE-TH resin for widespread industrial use, highlighting its potential to reduce environmental pollution effectively. Collaboration between industries, research institutions, and policymakers is vital to accelerate adoption and promote sustainable wastewater management practices.

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Conflict of Interest: There is no conflict of interest.

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