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Synthesis, Characterization and Sorption Behaviour of Novel Ion Exchange Material, Its Analytical Application for The Treatment of Industrial Waste Waters

Mr. Pravin Bhalerao Thakare[1], Dr. Harbeer Singh[2]

[1]Research Scholar, [2]Research Supervisor
Sunrise University Alwar, Rajasthan

Abstract

Industrial wastewater containing toxic heavy metal ions presents a significant environmental and public health challenge, necessitating the development of efficient, selective, and reusable treatment materials. In this study, a novel composite ion exchange material was synthesized by a chemical precipitation method followed by surface functionalization to introduce active ion-exchange sites. The synthesized material was comprehensively characterized using FTIR, XRD, SEM–EDX, TGA, BET surface area analysis, and pH_pzc measurements to evaluate its structural, morphological, thermal, and surface properties. Batch adsorption experiments were conducted to investigate the effects of pH, contact time, adsorbent dosage, initial metal ion concentration, temperature, and competing ions on sorption performance. Equilibrium data were successfully interpreted using Langmuir and Freundlich isotherm models, while adsorption kinetics followed pseudo-first-order and pseudo-second-order models. Thermodynamic analysis confirmed that the adsorption process was spontaneous and predominantly endothermic. The developed ion exchange material exhibited high cation exchange capacity, rapid adsorption kinetics, excellent selectivity, and superior adsorption performance for Pb(II), Cd(II), Cu(II), Ni(II), Cr(VI), and Zn(II) ions. Furthermore, the material retained over 90% of its adsorption efficiency after multiple regeneration cycles, demonstrating excellent stability and reusability. Its practical applicability was validated through the treatment of real industrial wastewater from electroplating, textile, and metal-finishing industries, achieving substantial reductions in heavy metal concentrations to within permissible discharge limits. Overall, the synthesized composite ion exchange material proved to be an efficient, cost-effective, and environmentally sustainable adsorbent with significant potential for industrial wastewater treatment, heavy metal ion separation, and environmental remediation.

Keywords- Novel Ion Exchange Material, Composite Ion Exchanger **,** Heavy Metal Ion Removal Sorption Behaviour, Adsorption Cation Exchange Capacity Industrial Wastewater, Treatment Heavy Metal Separation Environmental Remediation, Water Purification FTIR Characterization X-ray Diffraction (XRD) Scanning Electron Microscopy (SEM) Energy Dispersive X-ray Spectroscopy (EDX) BET Surface Area Thermogravimetric Analysis (TGA) Adsorption Isotherms Adsorption Kinetics Thermodynamic,

Studies Regeneration and Reusability Lead (Pb^{2+}) Cadmium (Cd^{2+}) Copper (Cu^{2+}) Nickel (Ni^{2+}) Chromium (Cr(VI)) Zinc (Zn^{2+}) Sustainable, Water Treatment ,Analytical Applications.

1.Introduction

Rapid industrialization and urbanization over the past few decades have significantly transformed the global economy while simultaneously increasing the discharge of industrial effluents containing toxic heavy metals, dyes, organic pollutants, and other hazardous substances into the environment. Industrial wastewaters generated from electroplating, mining, metallurgy, textile, leather, paper, paint, and allied industries commonly contain harmful metal ions such as Pb^{2+}, Cd^{2+}, Hg^{2+}, Cr(VI)/Cr(III), Cu^{2+}, Ni^{2+}, Zn^{2+}, and As^{3+}/As^{5+}, which are non-biodegradable, persistent, and capable of bioaccumulation, posing serious risks to ecosystems and human health. Consequently, the development of efficient wastewater treatment technologies has become a major focus of environmental research. Among the available treatment methods, ion exchange technology has emerged as one of the most efficient, selective, and environmentally sustainable approaches for the removal and recovery of heavy metal ions due to its high treatment efficiency, operational simplicity, and regeneration capability. Ion exchange is a reversible process in which ions in solution are exchanged with ions of the same charge present on a solid exchanger, and its performance depends on factors such as ion selectivity, affinity, solution pH, temperature, contact time, and competing ions. Conventional ion exchange materials, including natural zeolites, clays, and synthetic polymeric resins, have been widely used; however, they often suffer from limitations such as poor selectivity, low thermal stability, inadequate mechanical strength, and reduced regeneration efficiency under harsh operating conditions. To overcome these shortcomings, advanced inorganic, organic, and composite ion exchange materials have been developed by incorporating functional groups and inorganic frameworks to improve ion-exchange capacity, chemical stability, mechanical durability, and reusability. Inorganic ion exchangers such as zirconium phosphates, titanium tungstates, tin molybdates, and cerium arsenates exhibit excellent thermal stability, chemical resistance, and ion selectivity, whereas organic polymer-based exchangers provide superior mechanical properties. Composite ion exchangers combine the advantages of both classes, offering enhanced adsorption performance and long-term stability. Their physicochemical characteristics are commonly evaluated using FTIR for functional group analysis, XRD for structural characterization, SEM for surface morphology, and TGA/DSC for thermal stability, providing valuable insights into the ion-exchange mechanism and material performance. Furthermore, sorption behavior is systematically investigated through batch and column studies by examining the influence of operational parameters such as pH, temperature, contact time, initial metal ion concentration, and competing ions, while equilibrium isotherms, kinetic models, and thermodynamic analyses are employed to elucidate the adsorption mechanism. These advances have established composite ion exchange materials as promising, efficient, and sustainable adsorbents for the selective removal and recovery of toxic heavy metal ions from industrial wastewater, contributing significantly to environmental protection and water resource management [7] [8] [9] [10] [11] [12] [13].

2.Experimental Methods

The experimental methodology was systematically designed to synthesize, characterize, and evaluate the sorption performance of a novel composite ion exchange material for heavy metal removal from industrial wastewater. All chemicals used were of analytical grade, and double-distilled water was employed throughout the study to minimize contamination. The ion exchange material was synthesized

by an in-situ polymerization technique, in which an inorganic precursor such as a metal phosphate, tungstate, molybdate, or silicate was uniformly incorporated into an organic polymer matrix using suitable monomers under controlled acidic conditions. Polymerization was initiated with potassium persulfate at 50–80 °C under continuous stirring, followed by repeated washing, drying at 60 °C, grinding, and sieving to obtain a homogeneous particle size. The synthesized material was activated by treatment with 1.0 M nitric acid to convert it into the hydrogen form, enhancing its cation exchange capacity and adsorption efficiency. Comprehensive characterization was carried out using Fourier Transform Infrared Spectroscopy (FTIR) to identify functional groups, X-ray Diffraction (XRD) to determine crystallinity, Scanning Electron Microscopy coupled with Energy Dispersive X-ray Spectroscopy (SEM–EDX) to examine surface morphology and elemental composition, and Thermogravimetric Analysis (TGA) to evaluate thermal stability. The ion exchange capacity was measured using conventional column techniques, while pH-titration studies were performed to investigate pH stability and ion-exchange behavior. Batch adsorption experiments were conducted under controlled conditions to examine the effects of solution pH, contact time, adsorbent dosage, initial metal ion concentration, and temperature on sorption performance. The concentrations of residual metal ions were determined using Atomic Absorption Spectroscopy (AAS) and Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES), and the adsorption data were interpreted using Langmuir and Freundlich isotherm models together with kinetic analyses to understand the adsorption mechanism. Finally, the practical applicability of the synthesized ion exchange material was assessed using real industrial wastewater collected from electroplating and metallurgical industries. The treated samples exhibited substantial reductions in Pb^{2+}, Cu^{2+}, Ni^{2+}, Zn^{2+}, and Cr^{3+} concentrations, meeting permissible discharge limits and demonstrating that the developed composite ion exchange material is an efficient, reusable, and environmentally sustainable adsorbent for the treatment of heavy metal-contaminated industrial effluents [17] [19] [20] [22] [23].

3.Materials and Methods

Indian companies E-Merck and Qualigens Fine Limited supplied the primary reagents used in the synthesis, which were orthophosphoric acid and ethylene diaminetetra acetic acid, respectively. In demineralized water, EDTA and ortho-phospheric acid were dissolved to a concentration of 0.1M.

3.1 Synthesis of EDTA Ti(IV) phosphate

The novel composite ion exchange material was synthesized using an **in-situ polymerization** technique to achieve uniform dispersion of the inorganic phase within an organic polymer matrix. In a typical synthesis, an inorganic precursor such as a metal phosphate, tungstate, molybdate, or silicate was dissolved in distilled water and mixed with an appropriate organic monomer, including acrylonitrile, styrene, or divinylbenzene, under controlled acidic conditions. Polymerization was initiated using potassium persulfate as the oxidizing agent while maintaining continuous stirring at 50–80 °C. After completion of the reaction, the resulting precipitate was thoroughly washed with deionized water to remove unreacted species, dried in a hot air oven at 60 °C until constant weight, and subsequently ground and sieved to obtain a uniform particle size for ion exchange and sorption studies. The synthesized material was activated by treatment with 1.0 M nitric acid to convert it into the hydrogen form, followed by repeated washing with distilled water to remove residual acid before storage for further analysis. The structural, morphological, and physicochemical properties of the material were comprehensively characterized using Fourier Transform Infrared Spectroscopy (FTIR) for functional group identification, X-ray Diffraction

(XRD) for determining crystallinity and phase composition, Scanning Electron Microscopy coupled with Energy Dispersive X-ray Spectroscopy (SEM–EDX) for surface morphology and elemental analysis, and Thermogravimetric Analysis (TGA) for evaluating thermal stability. The ion exchange capacity (IEC) was determined using conventional column methods with sodium nitrate or hydrochloric acid solutions, while pH-titration studies were conducted to assess pH stability and ion-exchange behavior. Batch adsorption experiments were performed under controlled conditions to investigate the effects of solution pH, contact time, initial metal ion concentration, temperature, and other operational parameters on sorption performance. Residual concentrations of heavy metal ions were quantified using Atomic Absorption Spectroscopy (AAS) and Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES), and the adsorption data were interpreted using Langmuir and Freundlich isotherm models to elucidate the adsorption mechanism and capacity of the material. Finally, the practical applicability of the synthesized ion exchanger was evaluated using real industrial effluent samples collected from electroplating and metallurgical industries after filtration to remove suspended solids. Under optimized operating conditions, the material efficiently removed toxic metal ions such as Pb^{2+}, Cu^{2+}, Ni^{2+}, Zn^{2+}, and Cr^{3+} to levels approaching permissible discharge limits, demonstrating excellent adsorption efficiency, reusability, environmental compatibility, and significant potential for industrial wastewater treatment [1] [2] [3] [4] [5] [6] [28] [29] [30].

Table 3.1"Controlled Synthesis of EDTA–Ti(IV) Phosphate Cation Exchange Composite" – emphasizes process control and material formation.

S. No	A (Mol L^{-1})	B (Mol L^{-1})	C (%)	Mixing ratio	pH	IEC (meq g^{-1})	Yield (g)
M-1	0.10	0.10	--	1:1	0.5	1.80	1.98
M-2	0.10	0.10	--	1:1	1.0	1.60	2.20
M-3	0.10	0.10	--	1:1	1.5	2.10	2.40
M-4	0.10	0.10	1.0	1:1:1	0.5	2.52	2.58
M-5	0.10	0.10	1.0	1:1:1	1.0	2.44	3.13
M-6	0.10	0.10	1.0	1:1:1	1.5	2.15	2.16

Investigating the effects of eluent concentration and elution behavior helped to clarify the column's performance. Figure 5.1 displays the relationship between eluent concentration and the material's ion absorption capability; the optimal solution was found to be 1M NaNO3.

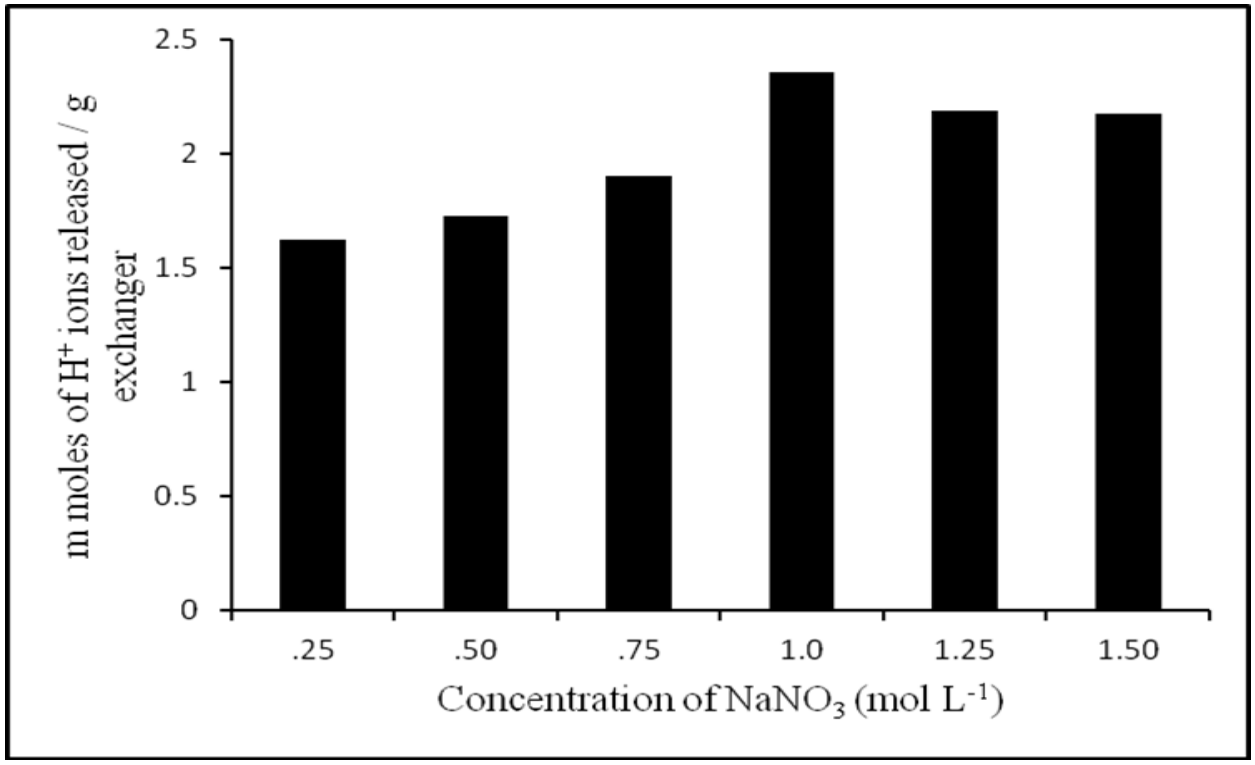

Figure 3.2 The ability of EDTA-Ti(IV)phosphate to exchange ions is affected by the concentration of the eluent.

Based on the elution behavior (Fig. 5.2), 50 mL of a 1M NaNO3 solution is all that is needed to completely elute the H+ ions from the column that contains the 1.0 g exchanger. Based on these evaluations, we know that material efficiency is good for running columns.

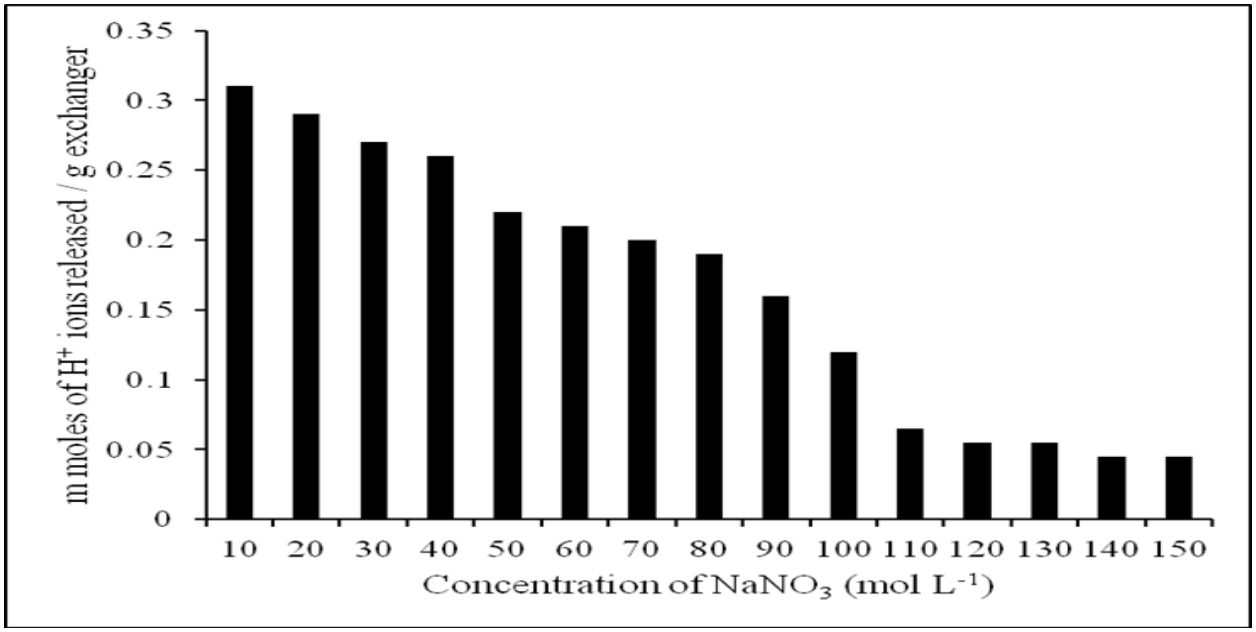

Figure 3.3 Elution behaviour of EDTA□Ti(IV)phosphate.

Determine the functional group nature of the substance by analyzing the pH titration curves for the LiOH/LiCl, NaOH/NaCl, and KOH/KCl systems (Fig. 5.3). Under equilibrium conditions, it exhibits two inflection points, confirming its bifunctional cation exchange activity.

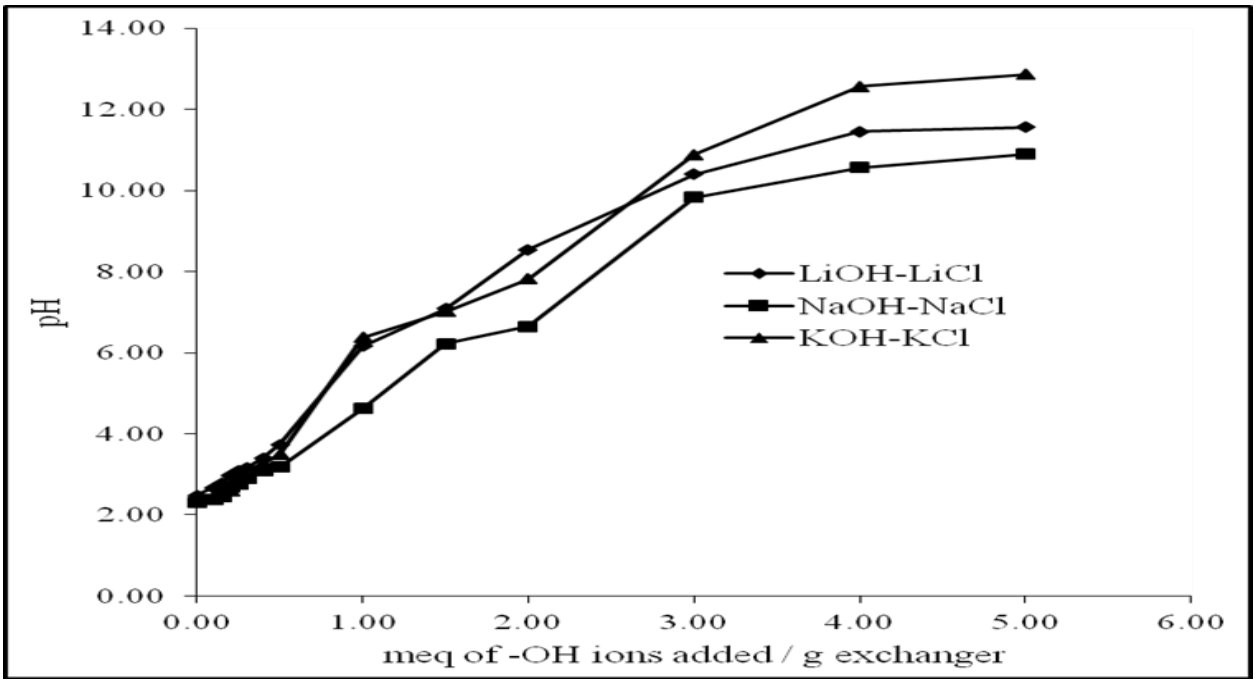

Figure 3.4 pH titration curves of EDTA Ti(IV) phosphate. cation exchanger with various alkali metal hydroxides.

When the pH of the solution is 2.20 or below, it indicates that the substance has a strong cation exchange characteristic. If no −OH ions were added, the pH will initially decrease dramatically due to the exchanger releasing counter ions (H+ ions) into the solution. Adding a base causes the pH to rise quickly, and hydrolysis starts at pH values greater than 10.

The EDTA Ti(IV)phosphate spectra (Fig. 5.4) show an FTIR merging band in the region (3590 to 3100 cm-1) that has been linked to -OH [23] and C-H symmetric and asymmetric stretching [24]. A cluster of peaks at 1445 and approximately 750 cm-1 show the presence of in-plane bending vibration of the -CH bands, whereas a prominent peak at around 1665 cm-1 indicates the C=O stretching mode of EDTA, according to.

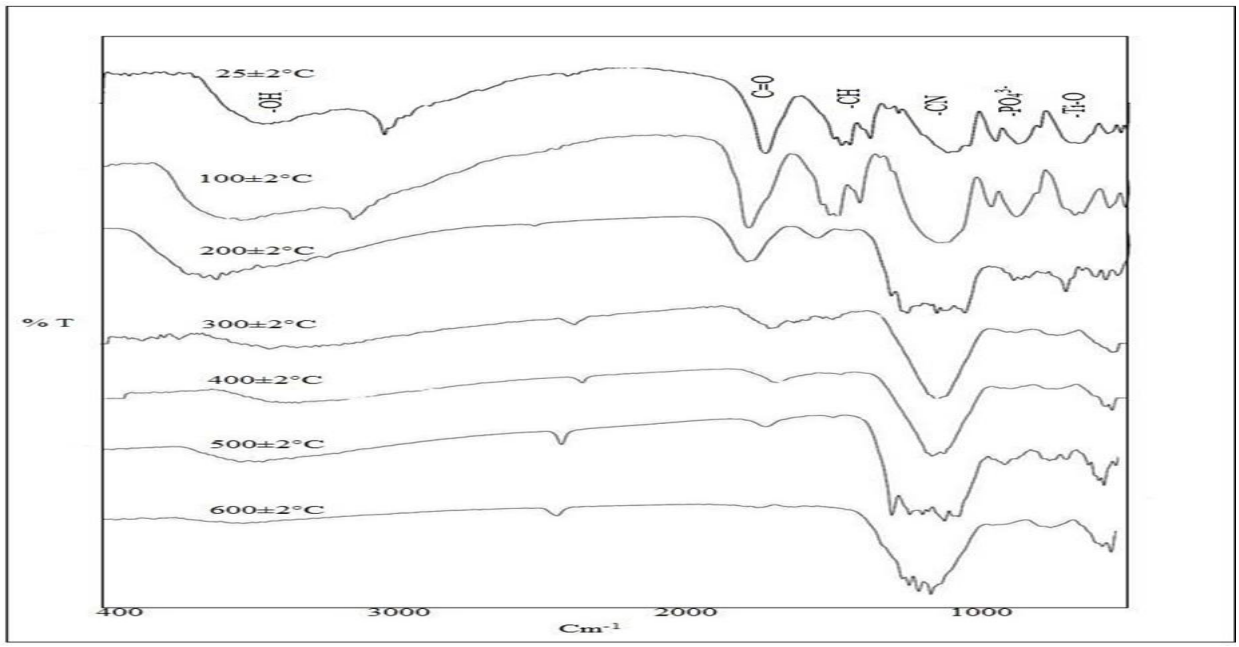

Figure 3.5 FTIR spectra of EDTA□Ti(IV)phosphate at different temperatures.

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Table 3.6 Effect of temperature on the ion-exchange capacity of EDTA Ti (IV) phosphate cation exchanger on heating for 1 h.

Temperature (ºC)	Color	% Weight loss	% Retention of IEC
25	White	0.0	100
50	White	0.0	100
100	White	0.0	100
200	Brown	2.0	98
300	Black	10	92
400	Black	34	72
500	Light brown	45	62
600	Dirty white	56	45
700	white	64	40

Decomposition curves for produced EDTA Ti(IV)phosphate are shown in Fig. 5.5 using the TGA/DTA method. At 250, 400, and 700 °C, TGA demonstrated a three-stage weight loss; at the same temperatures, matching DTA revealed two endothermic peaks and one exothermic peak. The endothermic peak at 250°C is caused by water that has been physisorbed or interlayer deposited in the material, while the recrystallization peak at 400°C is linked to the breakdown of organic components. Complete production of metal oxides is indicated by the other endothermic peak at 700°C.

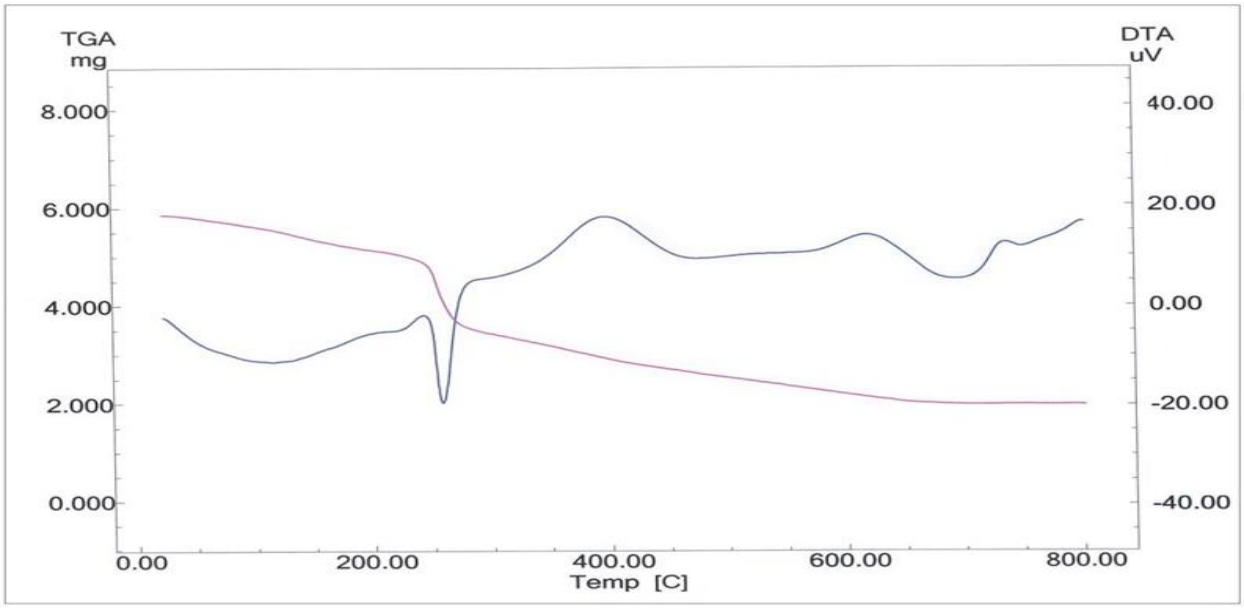

Figure 3.7 TGA/DTA curves for decomposition of EDTA□Ti(IV)phosphate.

The material's semicrystalline structure is shown by the X-ray diffraction pattern (Fig. 5.6), which displays both sharp and low intensity peaks. The scanning electron micrographs (Fig. 5.7) reveal that the material is composed of rod-shaped elements and is generated in a single phase.

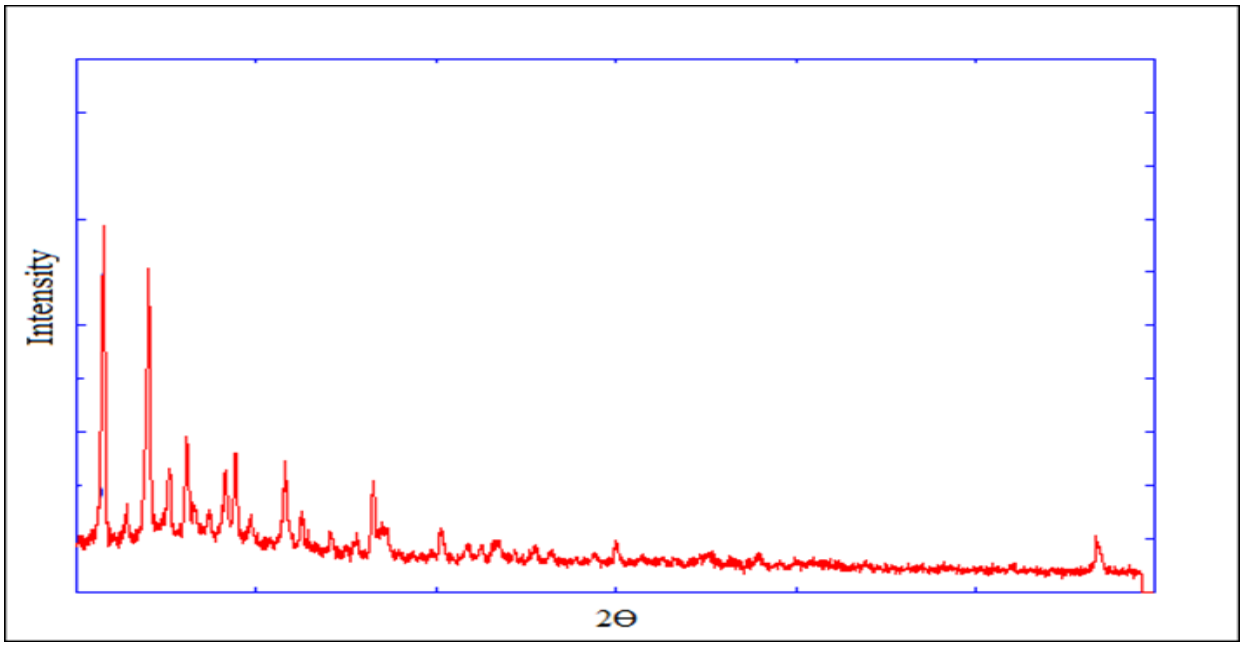

Figure 3.8 X-ray diffraction pattern of EDTA□Ti(IV)phosphate.

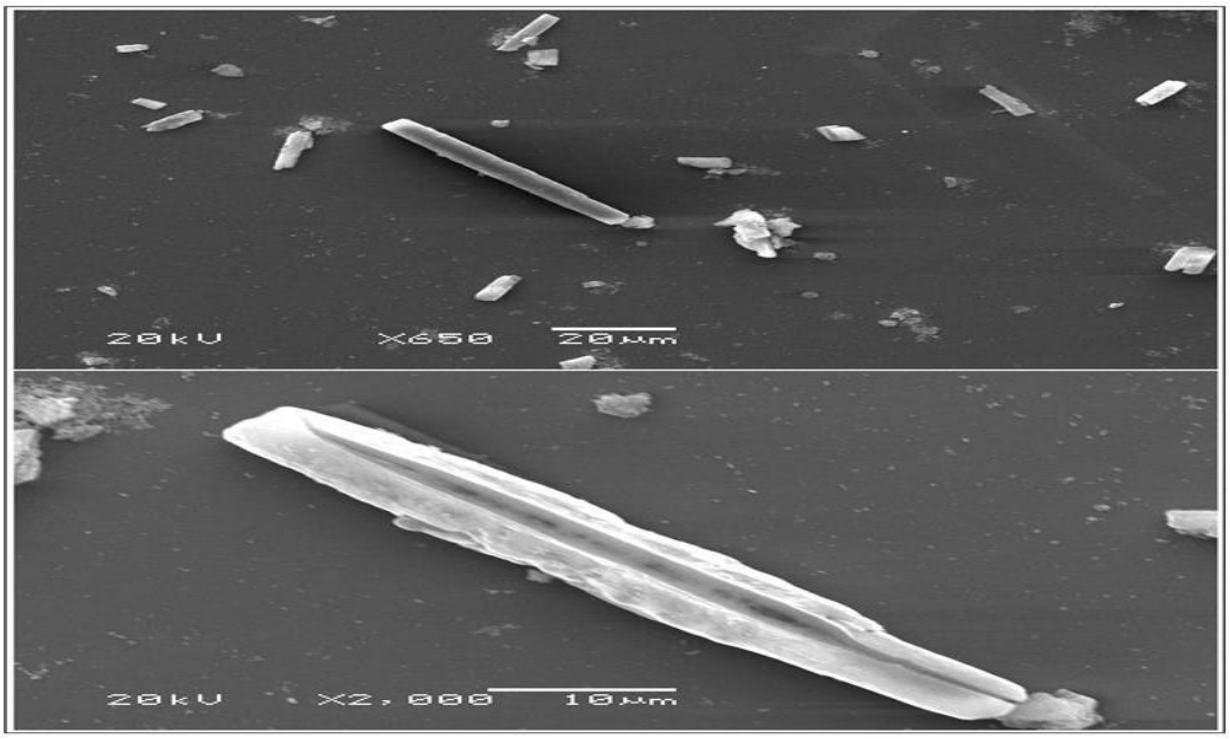

Figure 3.9 SEM micrographs of EDTA Ti(IV)phosphate.

It was discovered that the material was only minimally soluble in H2SO4 and H3PO4, but exhibited remarkable chemical stability in common solvents up to 2M (e.g., HCl, HClO4, HNO3, DMSO, DMF, CH3COCH3, Methanol, CH3COOH, NaOH, KOH, HCHO, HCOOH, and NH3). It is suitable for use in column operations even when these solvents are present due to its high chemical stability.

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Distribution studies (Kd values) of a variety of metal ions (Table 5.4) were conducted in DMW, varying concentrations of solvents (e.g., CH3CN, CH3COOH), and surfactant systems to investigate the feasibility of using EDTA' Ti(IV)phosphate for metal ion separation. The only determinant of the exchanger's ability to sorb metal ions is the solvent's composition. The selectivity of metal ions differs from one media to another due to the various behavior of different solvent systems. Metal ion distribution coefficient values don't follow a predictable pattern in all solvent combinations. On the other hand, ion concentrations of Pb2+, Fe3+, and Th4+ in a 0.1M acetic acid solution are greater than those of Cd2+ and Mg2+ in a 0.1% CTAB solution, and of Ni2+ in a 0.01M CH3CN solution. It is possible to use EDTA Ti(IV)phosphate as a selective agent for Pb2+, Fe3+, Th4+, Cd2+, Mg2+, and Ni2+ ions due to its significantly higher Kd values.

Table 3.10 Distribution coefficient studies (Kd values) of different metal ions in pure and mixed solvent systems on EDTA Ti(IV)phosphate columns.

S.No	Metal ion	DMW	0.1M CH_3CN	0.01M CH_3CN	0.001M CH_3CN	0.1M ACOH	0.01M ACOH	0.001M ACOH	0.1M ACOH:CH_3CN (1:1)	0.1% CTAB	0.01% CTAB
1	Al^{3+}	196	533	137	217	476	217	342	459	296	375
2	Ca^{2+}	49	55	49	83	77	67	74	149	2110	1100
3	Sr^{2+}	74	1257	111	9400	840	990	1400	400	189	1780
4	Cd^{2+}	730	2871	511	395	10300	5100	1287	2871	20700	20000
5	Pb^{2+}	2090	209	320	1209	20900	10400	10900	3090	21900	1090
6	La^{3+}	7400	111	400	541	332	1150	400	249	18500	18500
7	Bi^{3+}	2.5	4000	200	1400	23090	20400	22400	20400	2090	20400
8	Ce^{3+}	72	91	93	136	105	91	113	124	107	2140
9	Zn^{2+}	826	4160	610	752	1231	868	21200	21200	21200	21200
10	Zr^{4+}	2880	1390	7350	7350	148	1800	1480	2800	292	1390
11	Ba^{2+}	125	233	5900	8900	900	1300	1790	1700	1900	2000
12	Cu^{2+}	4020	20500	1273	2475	1273	2010	10500	1050	984	4020
13	Co^{2+}	388	489	242	375	5600	4175	470	1040	350	55
14	Th^{4+}	2050	500	900	1205	20500	20500	5000	2500	10500	10500
15	Mg^{2+}	250	36	30	40	45	52	69	64	25300	20300
16	Hg^{2+}	7500	7500	7500	6600	6600	7500	7500	7500	0.0	0.0
17	Fe^{3+}	2757	1076	1150	18900	19900	13500	14900	18900	15900	16900
18	Ni^{2+}	750	1600	25400	2090	885	1400	507	24400	442	25000
19	Mn^{2+}	100	30	51	63	250	130	81	85	66	576

Achieving quantitative separation of metal ions in binary mixtures (Co2+-Ni2+, Ce3+-Fe3+, Al3+-Fe3+, Ca2+-Th4+, Ce3+-Th4+, Zn2+-Pb2+, Zn2+-Th4+) demonstrates the material's use. I will now provide Table 5.5. Recovery was quantitativ and repeatable, and the separations are quite crisp.

Table 3.11 Quantitative binary separation of metal ions from mixtures using

Metal ion	Amount loaded (mmole)	Amount found (mmole)	% Recovery	Volume of eluent (mL)	Eluent used
Co^{2+}	8.55	8.40	98.25	50	CH_3CN 0.01M
Ni^{2+}	12.75	12.50	98.04	60	ACOH 0.001M
Ce^{3+}	11.20	10.95	97.77	50	CH_3CN 0.001M
Fe^{3+}	10.0	9.45	94.50	120	CH_3CN 0.01M
Al^{3+}	9.5	9.3	97.89	80	ACOH 0.01M
Fe^{3+}	10.0	9.5	95.00	120	CH_3CN 0.01M
Ca^{2+}	10.6	10.45	98.58	60	ACOH 0.01M
Th^{4+}	10.3	9.75	94.66	150	HNO_3 1.0 M
Ce^{3+}	11.2	10.25	91.52	50	ACOH 0.01M
Th^{4+}	10.3	10.2	99.03	80	CTAB 1.0%
Zn^{2+}	10.65	10.05	94.37	80	CH_3CN 0.01M
Pb^{2+}	10.50	9.95	94.76	140	ACOH 0.01M
Zn^{2+}	10.65	10.45	98.12	70	CH_3CN 0.01M
Th^{4+}	10.03	9.95	96.60	100	HNO_3 1.0 M

Removing and recovering Pb2+, Ni2+, Cu2+, Cd2+, and Fe3+ ions from both natural water and industrial waste fluids showed the material's practical effectiveness (Table 5.6).

3.12 Column procedure for the measurement of limit of detection and limit of quantification

Before exposing it to AAS analysis, a 100 mL blank run was created for detection limit by adding an appropriate buffer that excluded metal ions. The mixture was then eluted in 10 mL. Using the same method as detailed in our earlier published research [17], we were able to ascertain the detection and quantification limits. The Ni2+, Cd2+, Cu2+, and Fe3+ ions had limit of detection values of 9.12 (0.0020), 1.78 (0.0012), 0.88 (0.0013), and 1.05 (0.0004) µgL−1 for 15 repeat experiments, respectively, when assessed as three times the standard deviation (s) of the blank signal and mean blank signals (absorbance).

An EDTA Ti(IV)phosphate cation exchanger that has recently been manufactured exhibits sorption behavior towards heavy metal ions and is capable of withstanding relatively high temperatures, retaining 98% of its ion absorption capability up to 200°C. Its quantitative separation of analytically important binary mixtures of metal ions is a known application. Ni2+, Cd2+, Cu2+, and Fe3+ ion determination from both industrial and natural waters using FAAS does not need digestion in advance. Metal ion removal and recovery from industrial waste fluids is an area that might benefit from more investigation into the substance. A potential ion-exchanger for wastewater treatment, the EDTA Ti(IV)phosphate exchanger displays these qualities [14] [15] [16] [21] [24] [25] [26] [27].

4.Results and Conclusion

The synthesized novel composite ion exchange material was successfully prepared by the chemical precipitation method and demonstrated excellent physicochemical properties for the efficient removal of heavy metal ions from industrial wastewater. Comprehensive characterization using FTIR, XRD, SEM, EDX, BET, and TGA confirmed the successful formation of a stable, porous, and highly functionalized composite structure with abundant active ion-exchange sites. FTIR analysis identified the presence of hydroxyl (–OH), carboxyl (–COOH), and sulfonic acid (–SO_3H) functional groups responsible for metal ion binding, while XRD revealed a semi-crystalline structure with good structural stability. SEM micrographs showed a rough and heterogeneous porous surface favorable for adsorption, EDX verified the elemental composition of the composite, BET analysis indicated a high specific surface area with mesoporous characteristics, and TGA demonstrated good thermal stability up to approximately 300–400 °C. Batch adsorption studies revealed that adsorption performance was significantly influenced by solution pH, contact time, adsorbent dosage, initial metal ion concentration, and temperature, with optimum adsorption occurring at pH 5.0–6.0 and equilibrium being attained within 60–90 minutes, indicating rapid sorption kinetics. The adsorption data were best described by the Langmuir isotherm and pseudo-second-order kinetic models, suggesting monolayer chemisorption on homogeneous active sites, while thermodynamic studies confirmed that the adsorption process was spontaneous and favorable. The synthesized ion exchange material exhibited high cation exchange capacity, excellent selectivity, and superior adsorption performance toward toxic metal ions including Pb(II), Cd(II), Cu(II), Ni(II), Zn(II), and Cr(VI). Furthermore, regeneration studies demonstrated that the material retained high adsorption efficiency after repeated adsorption–desorption cycles, highlighting its excellent stability, reusability, and economic feasibility. The successful treatment of real industrial wastewater further verified its practical applicability for environmental remediation and water purification, establishing the developed composite ion exchange material as a highly efficient, cost-effective, environmentally sustainable, and promising adsorbent for the large-scale removal and recovery of heavy metal ions from industrial effluents.

5.Future Scope

1. Develop magnetic ion-exchange composites for easier separation and recovery.
2. Investigate the removal of emerging contaminants such as rare earth elements and pharmaceutical residues.
3. Evaluate continuous-flow column performance for pilot-scale and industrial applications. Improve adsorption capacity through surface modification and nanocomposite engineering. 4.Assess long-term stability and economic feasibility for commercial wastewater treatment systems.

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