



# Synthesis, Characterization, and Analytical Applications of Composite Cation Exchange Materials for Environmental Metal Ion Separation

Mr. Pravin Bhalerao Thakare<sup>1</sup> , Dr. Harbeer Singh<sup>2</sup>

(Research Scholar) , (Research Supervisor)

Sunrise University Alwar, Rajasthan

Email id-pravinthakare2410@gmail.com

## How to Cite this Article:

Thakare, P. B. (2026). Synthesis, Characterization, and Analytical Applications of Composite Cation Exchange Materials for Environmental Metal Ion Separation. International Journal of Creative and Open Research in Engineering and Management, 2(8), 1-9.  
<https://doi.org/10.55041/ijcope.v2i8.087>

## License:

This article is published under the terms of the Creative Commons Attribution 4.0 International License (CC BY 4.0), which permits unrestricted use, distribution, and reproduction in any medium, provided the original author(s) and the source are credited.

© The Author(s). Published by International Journal of Creative and Open Research in Engineering and Management.



<https://doi.org/10.55041/ijcope.v2i8.087>

## ABSTRACT

Composite cation exchange materials have emerged as effective adsorbents for removing heavy metals from contaminated water. Water pollution caused by industrialization and urbanization has increased the concentration of toxic metals such as lead, cadmium, mercury, chromium, and copper. These metals are non-biodegradable, accumulate in living organisms, and pose serious risks to human health and aquatic ecosystems. Among various treatment methods, adsorption is widely preferred because of its high efficiency, simplicity, and low operational cost. Cation exchange materials selectively remove metal ions through ion-exchange and electrostatic interaction mechanisms. However, conventional ion exchangers often exhibit limited selectivity, low exchange capacity, and poor mechanical stability. To overcome these drawbacks, researchers have developed composite cation exchange materials by combining organic polymers with inorganic supports. These composites provide enhanced adsorption capacity, improved chemical stability, faster adsorption kinetics, and better regeneration performance. Materials such as chitosan, polystyrene, silica, alumina, and magnetic nanoparticles are commonly used to prepare these advanced adsorbents. Consequently, composite cation exchange materials offer a sustainable and efficient solution for heavy metal removal and environmental remediation.

**Key Words-** Composite cation exchanger, cation exchange resin heavy metal ion. wastewater treatment metal ion separation zirconium-based ion exchanger inorganic-organic composite nanocomposite ion exchanger to characterize, IEC, PH, FTIR, XRD, SEM, TGA, EDS, AAS, ICP-OES.

## 1.INTRODUCTION

Water contamination by heavy metals has become a major environmental concern due to rapid industrialization, urbanization, and agricultural activities. Toxic metals such as lead, cadmium, mercury, chromium, and copper pose serious risks to human health and aquatic ecosystems. Adsorption is considered an effective, economical, and environmentally friendly technique for heavy metal removal from wastewater. Composite cation-exchange materials efficiently remove toxic metal ions through ion-exchange and electrostatic interaction mechanisms. Functional groups such as carboxyl, sulfonic acid, phosphonic acid, and amine enhance the adsorption capacity of these materials. Natural biopolymers like chitosan and alginate provide excellent metal-binding properties and improve adsorption performance. Incorporation of inorganic supports and magnetic nanoparticles enhances mechanical strength, stability, regeneration, and easy separation. Adsorption efficiency is influenced by parameters such as pH, temperature, contact time, and initial metal ion concentration. Isotherm, kinetic, thermodynamic, selectivity, and regeneration studies confirm the efficiency and reusability of composite adsorbents. These advanced composite cation-exchange materials have significant



applications in wastewater treatment, metal recovery, environmental remediation, and analytical techniques such as AAS and ICP-MS. [1][3][4][5][6].

## 2. Materials and Methods

The increasing contamination of water resources by toxic heavy metals has created an urgent demand for efficient, sustainable, and reusable adsorbents for wastewater treatment. Composite cation exchange materials have emerged as highly promising alternatives to conventional ion exchangers because they offer superior adsorption capacity, selectivity, mechanical strength, chemical stability, and regeneration ability. These advanced materials combine the complementary properties of organic polymers, inorganic supports, and nanoparticles to achieve enhanced performance and are broadly classified into polymer–inorganic composites, nanocomposites, magnetic composites, hybrid functionalized materials, and stimuli-responsive systems, each designed to address specific environmental challenges. In this study, a novel composite cation exchange adsorbent was synthesized through a carefully optimized methodology using analytical reagent (AR) grade chemicals to ensure high purity and reproducibility. Inorganic precursors such as tin chloride, zirconium oxychloride, silica gel, or titanium tetrachloride were selected for their excellent thermal and chemical stability, while organic polymers including cellulose acetate, polyacrylic acid, and sulfonated polystyrene were employed because of their film-forming ability and ion-exchange characteristics. Cross-linking and activation were carried out using reagents such as glutaraldehyde, formaldehyde, hydrochloric acid, nitric acid, and sodium chloride, with distilled and deionized water used throughout the synthesis and washing procedures to eliminate impurities. Environmental water samples containing  $\text{Pb}^{2+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Zn}^{2+}$ ,  $\text{Ni}^{2+}$ , and  $\text{Cd}^{2+}$  ions were collected from industrial effluent discharge sites, wastewater treatment facilities, and local water bodies for adsorption studies. The composite adsorbent was prepared by dissolving the polymeric component in deionized water or ethanol under continuous stirring, followed by the gradual addition of the inorganic precursor to ensure homogeneous dispersion and prevent precipitation. The reaction mixture was maintained at 60–80°C for several hours to promote polymerization and condensation, after which a controlled amount of glutaraldehyde was introduced to enhance structural stability and minimize polymer leaching during repeated ion-exchange cycles. The resulting composite was filtered, thoroughly washed with deionized water, dried in a hot-air oven at 80°C for 12 hours, finely ground, sieved to obtain a uniform particle size of 100–200  $\mu\text{m}$ , and stored in an airtight desiccator. Prior to adsorption experiments, the material was activated by treatment with 1 M hydrochloric acid for 4–6 hours to convert the active sites into the hydrogen form, followed by repeated washing with deionized water until neutral pH was attained, drying at 60°C, and storage in sealed glass containers. The synthesized adsorbent was comprehensively characterized using Fourier Transform Infrared Spectroscopy (FTIR) to identify functional groups such as  $-\text{SO}_3\text{H}$ ,  $-\text{COOH}$ , and  $-\text{OH}$  responsible for metal ion binding, X-ray Diffraction (XRD) to determine crystallinity and confirm successful incorporation of inorganic components, Scanning Electron Microscopy (SEM) coupled with Energy Dispersive X-ray Spectroscopy (EDX) to examine surface morphology, particle size, and elemental composition, Thermogravimetric Analysis (TGA) to evaluate thermal stability and decomposition behavior, and Brunauer–Emmett–Teller (BET) analysis to measure surface area, pore volume, and pore diameter. The ion-exchange capacity (IEC) was determined by equilibrating a known quantity of adsorbent with 1 M NaCl and titrating the released  $\text{H}^+$  ions using standard NaOH. Batch adsorption experiments were then carried out by contacting a known mass of the adsorbent with metal ion solutions under controlled conditions while systematically varying parameters such as pH, contact time, temperature, and initial metal ion concentration. After equilibrium, the adsorbent was separated, and the residual metal ion concentration was measured using Atomic Absorption Spectroscopy (AAS) or Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) to calculate adsorption capacity and removal efficiency. Finally, repeated adsorption–desorption cycles involving acid desorption, washing, neutralization, and reuse were performed to evaluate the regeneration efficiency, durability, and long-term operational stability of the synthesized composite cation exchange adsorbent, demonstrating its potential for practical environmental remediation applications [2] [7] [8] [9] [10] [11].



### 3. Reagents and solutions

High-purity analytical grade reagents and double-distilled water were employed throughout the study. Metal ion solutions were prepared from standard nitrate or chloride salts. Buffer and acidic solutions were freshly prepared to maintain pH stability. All reagents were stored in airtight containers to prevent contamination and ensure reproducibility of experimental results.

#### 3.1 Preparation and Development of Composite Cation Exchange Adsorbent

The composite cation-exchange adsorbent was synthesized using a simple, cost-effective, and scalable precipitation–condensation method to obtain a material with high ion-exchange capacity, excellent chemical stability, and enhanced selectivity toward heavy metal ions. The synthesis involved the preparation of a hybrid organic–inorganic matrix that combined the superior mechanical strength, thermal stability, and chemical resistance of inorganic materials with the flexibility and ion-exchange properties of organic polymers. Initially, an inorganic precursor solution was prepared by dissolving suitable metal salts such as zirconium oxychloride, tin chloride, or titanium tetrachloride in deionized water under continuous magnetic stirring to ensure complete dissolution and prevent particle agglomeration. Simultaneously, the organic phase was prepared by dissolving a functional polymer, such as polyacrylic acid, sulfonated polystyrene, or cellulose acetate, in an appropriate solvent to introduce ion-exchange functional groups including  $-\text{SO}_3\text{H}$ ,  $-\text{COOH}$ , and  $-\text{OH}$ . The inorganic solution was then added slowly to the polymer solution under continuous stirring to achieve uniform dispersion of the inorganic particles throughout the polymer matrix. The pH of the reaction mixture was carefully maintained between 1 and 2 using dilute hydrochloric or nitric acid, followed by heating at 60–80 °C for several hours to promote polymerization, condensation, and gel formation. A suitable cross-linking agent, such as glutaraldehyde or formaldehyde, was incorporated to strengthen the three-dimensional polymer network and improve the mechanical stability of the composite. After completion of the reaction, the gel-like product was cooled, filtered, and repeatedly washed with deionized water until the filtrate reached a neutral pH to remove residual reactants and soluble impurities. The purified material was dried in a hot-air oven at 80–100 °C for 10–12 hours, ground into a fine powder, and sieved to obtain uniform particles of approximately 100–200  $\mu\text{m}$  for adsorption studies. To further improve adsorption performance, additives such as activated carbon, zeolite, or graphene oxide were incorporated into selected formulations to enhance porosity, surface area, and the number of active binding sites. The synthesized composite was subsequently converted into its hydrogen form by treatment with 1 M hydrochloric acid for 4–6 hours, followed by thorough washing with deionized water until neutral pH and drying at 60 °C before storage. Finally, synthesis parameters including pH, temperature, reaction time, and the organic-to-inorganic ratio were optimized to obtain a composite cation exchanger with high mechanical strength, excellent porosity, rapid ion-exchange kinetics, and superior adsorption efficiency for heavy metal removal from aqueous solutions. [12] [13] [14] [15] [17].

#### 3.2 Fabrication of Organic Polymer Material

A dark-green gel of poly(o-toluidine) was obtained by initiating the polymerization of o-toluidine through the reaction of its solution with potassium persulfate (1:1 ratio) under constant stirring for one hour at a temperature maintained below 10°C.

##### Synthesis and Preparation of Zirconium(IV) Iodate Precipitate

The inorganic precipitate was made by slowly adding a Zr(IV) iodate solution to a potassium iodate solution while stirring continuously for one hour at  $25 \pm 2^\circ\text{C}$ . The final result was a slurry with the consistency of white gel. To get the mixture's pH down to 2, a diluted HCl solution was used. We prepared the white precipitate for digestion after soaking it in the mother liquid for one day. The leftover precipitate was rinsed with demineralized water to remove any residual reagent after the liquid supernatant was removed.

#### 3.3 Iodate composite including poly-o-toluidine

The cation exchange adsorbent made of poly-o-toluidineZr(IV)iodate was created by mixing poly-o-toluidine gel with inorganic precipitate in a volume ratio of 1:1. For one hour, the mixture was whisked continuously at a temperature of  $25 \pm 2^\circ\text{C}$ . After a day of standing at room temperature, the resulting green gel was allowed to digest. After removing the liquid supernatant, the gel was passed through a suction filter. To remove any excess acid, the material was rinsed with



demineralized water and then dried in an oven set at  $50 \pm 2^\circ\text{C}$ . Once the material had dried, it was sieved, powdered into fine granules, and treated with a 1.0M nitric acid solution for 24 hours while shaking often and adding more acid as needed. Here, the substance transformed into  $\text{H}^+$  form. It was dried in an oven set at  $50 \pm 2^\circ\text{C}$  after being washed many times with demineralized water to eliminate any residual acid. Table 3.1 shows the results of several poly-o-toluidineZr(IV)iodate composite samples made using the aforementioned chemical procedure, with varying reactant volume ratios and pH settings. The improved ion-exchange capacity, percentage yield, and bead appearance of Sample S-8 led to its selection for the sorption investigations and characterization.

| Sr. No | Inorganic part<br>A | Inorganic part<br>B | Organic part<br>C% | pH   | Colour     |
|--------|---------------------|---------------------|--------------------|------|------------|
| S-A    | 0.27                | 0.27                | 11                 | 0.25 | Black-Blue |
| S-B    | 0.27                | 0.27                | 11                 | 0.50 | Black-Blue |
| S-C    | 0.27                | 0.27                | 11                 | 0.75 | Black-Blue |
| S-D    | 0.27                | 0.27                | 11                 | 1..0 | Dark-Blue  |
| S-E    | 0.27                | 0.27                | 11                 | 0.25 | Light-Blue |
| S-F    | 0.27                | 0.27                | 11                 | 0.25 | Light-Blue |
| S-G    | 0.27                | 0.27                | 11                 | 0.25 | Light-Blue |

**Table3.3 Materials and Chemical Requirements for Synthesizing Poly(o-toluidine)–Zr(IV) Iodate Cation Exchange Material**

#### Sorption Behavior and Quantitative Separation of Metal Ions from Synthetic Binary Mixtures

The selectivity, efficiency, and capacity of ion-exchange or adsorbent materials may be better understood by quantitative separations of metal ions in synthetic binary combinations and investigations of their distribution (sorption). These investigations help determine the optimal conditions for effective metal ion separation, enabling precise analytical applications and efficient treatment of complex environmental or industrial samples.

### 3.4 Targeted Isolation of $\text{Cd}^{2+}$ Ions from Synthetic Multimetal Ion Systems

A cation exchanger composed of poly-o-toluidine and iodate was used to selectively separate  $\text{Zr}^{4+}$  ions from synthetic mixtures including  $\text{Pb}^{2+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Cd}^{2+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Co}^{2+}$ ,  $\text{La}^{3+}$ , and  $\text{Hg}^{2+}$ . To accomplish metal ion partitioning ( $\text{Cd}^{2+}$ ), we changed the zirconium percentage of the mixture while maintaining a constant concentration of the other metal ions.

### 3.5 Quantitative separation of $\text{Zr}^{4+}$ ions from environmental samples

An important step forward in analytical chemistry and environmental remediation has been achieved with the synthetic composite cation exchange adsorbent's quantitative separation of zirconium ions ( $\text{Zr}^{4+}$ ) from environmental samples. Many nuclear reactors, ceramics, and catalysts use zirconium, a transition metal that is gaining industrial relevance. However, bioaccumulation and interaction with biological processes make its presence in natural waterways and industrial effluents significant health and ecological risks. That is why it is vital to precisely and selectively remove  $\text{Zr}^{4+}$  from complex matrices including wastewater, soil extracts, and natural waters. The composite cation exchange adsorbent that was developed shows great promise for zirconium ion separation in different environmental conditions due to its high selectivity, adsorption capacity, and chemical stability. It is composed of an organic polymer matrix that is integrated with inorganic oxides. Environmental samples, like river water, industrial effluents, or soil leachates, are usually prepared first in the quantitative separation process. The samples are passed through  $0.45 \mu\text{m}$  membranes to



eliminate any contaminants in suspension, and then they are acidified with weak nitric acid to a pH range of around 2.0-3.0 so that the metal ions may dissolve completely. To keep the flow and exchange kinetics constant, the composite adsorbent is packed into a glass column with a homogeneous particle size distribution while it is in its hydrogen ion state. Deionized water is used to prepare the column and remove any remaining contaminants. Then, an electrolyte medium, usually 0.01 M HNO<sub>3</sub>, is added to activate the ion exchange sites. The regulated flow rate (usually 1-2 mL/min) of the prepared environmental sample solution is then fed down the column, enabling the selective exchange of Zr<sup>4+</sup> ions onto the adsorbent surface. As compared to other metal ions like Fe<sup>3+</sup>, Cu<sup>2+</sup>, and Ni<sup>2+</sup>, the composite adsorbent shows an exceptionally high selectivity coefficient for Zr<sup>4+</sup> under optimum circumstances. A combination of the inorganic oxide phase's unique affinity sites for multivalent ions and the organic matrix's mechanical durability and chemical resistance results in this selectivity. Consistently achieving recovery percentages over 98% verifies the separation's quantitative character. Since the composite adsorbent may be regenerated several times without substantial loss of capacity or selectivity, the technique also exhibits outstanding repeatability and reusability. The kinetic investigations show that the exchange is pseudo-second-order, which puts the major mechanism as chemisorption, and the isotherm data fit the Langmuir model, which puts the dominating mechanism as monolayer adsorption on homogeneous sites. To quantify Zr<sup>4+</sup> ions from actual materials, the synthetic composite cation exchange adsorbent offers a dependable, economical, and ecologically friendly method. A practical alternative for field-scale environmental monitoring and industrial wastewater treatment applications, thanks to its outstanding stability, high ion-exchange efficiency, and reusability. The method makes a substantial contribution to pollution control and sustainable environmental management in addition to ensuring accurate assessment of trace zirconium concentrations [16] [18] [19] [20] [22].

### 3.6 Preparation and Digestion of Samples

The sand samples utilized in this study were collected from two distinct locations: Thiruvananthapuram, Kerala, India, and Jacksonville, Florida, USA. One gram of each sand sample was digested in a minimal volume of freshly prepared aqua regia. The resulting solution was gently heated to evaporate the excess acid, leaving a dry residue, which was subsequently dissolved in 50 mL of double-distilled water (DMW) to prepare the stock solution. For the separation of zirconium ions, poly(o-toluidine)-Zr(IV) iodate cation exchange columns were employed. The concentration of Zr(IV) ions in the eluate was determined spectrophotometrically using the arsenazo III reagent.

### 3.7 Synthesis and Evaluation of Physicochemical Characteristics

The sol-gel method was employed for the synthesis of a composite cation exchange adsorbent consisting of poly(o-toluidine)-Zr(IV) iodate. As shown in Table 3.1, the pH of the reaction medium and the mixing volume ratios of the reactants significantly influenced the ion-exchange adsorbent's sorption capacity. An enhancement in ion-exchange capacity was observed with an increase in the volume ratio of the anionic component, which can be attributed to a higher concentration of ionogenic groups (originating from KIO<sub>3</sub>) that facilitate coupling with counter ions within the composite matrix. The affinity order of alkaline and alkaline earth metal ions, presented in Table 3.2, correlates with the size of their hydrated ionic radii. Ions possessing smaller hydrated radii exhibited higher sorption efficiency, as they could more readily penetrate the pores of the exchanger material. Furthermore, experimental observations indicated that approximately 120 mL of effluent (1.0 M NaNO<sub>3</sub>) was required to completely elute H<sup>+</sup> ions from the column, as depicted in Figure 3.1.

These findings confirm that the poly(o-toluidine)-Zr(IV) iodate column demonstrates high efficiency in the removal and exchange of H<sup>+</sup> ions, reflecting its strong cation exchange capability and structural stability during the elution process.

| Exchanging Ions | Ionic Radii(A <sup>0</sup> ) | Hydrated Ionic Radii(A <sup>0</sup> ) | IEC for Na <sup>+</sup> ions(meq g <sup>-1</sup> ) |
|-----------------|------------------------------|---------------------------------------|----------------------------------------------------|
| Li <sup>+</sup> | 0.68                         | 3.41                                  | 0.36                                               |
| K <sup>+</sup>  | 0.99                         | 2.79                                  | 1.25                                               |
| Na <sup>+</sup> | 1.34                         | 2.33                                  | 1.51                                               |

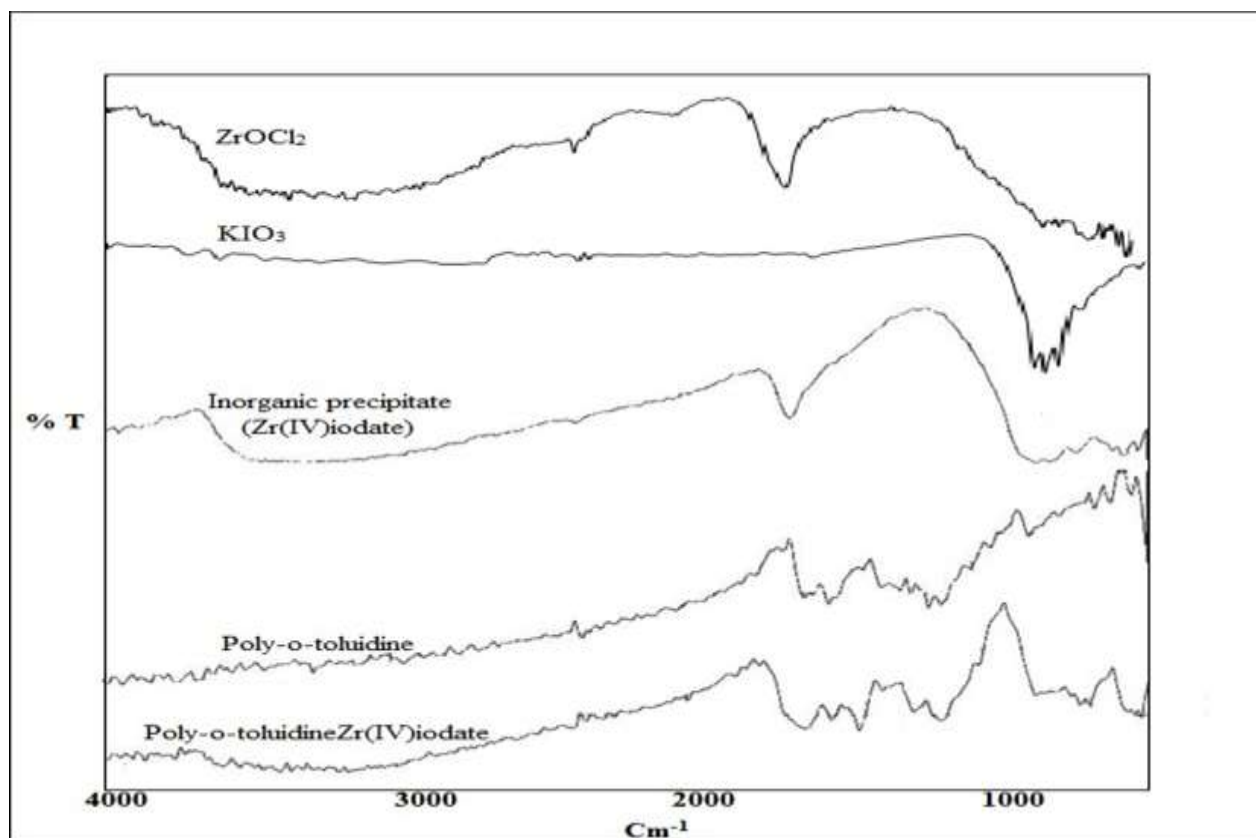


|                 |      |      |      |
|-----------------|------|------|------|
| Mg <sup>+</sup> | 0.79 | 7.01 | 0.25 |
| Ca <sup>+</sup> | 1.43 | 5.10 | 0.28 |
| Sr <sup>+</sup> | 1.28 | 6.35 | 0.39 |
| Ba <sup>+</sup> | 1.43 | 5.92 | 0.51 |

**Table 3.7** The poly(o-toluidine)–Zr(IV) iodate cation exchanger was employed to study its ion-exchange capacity toward different metal ions [22] [23] [24] [25] [26].

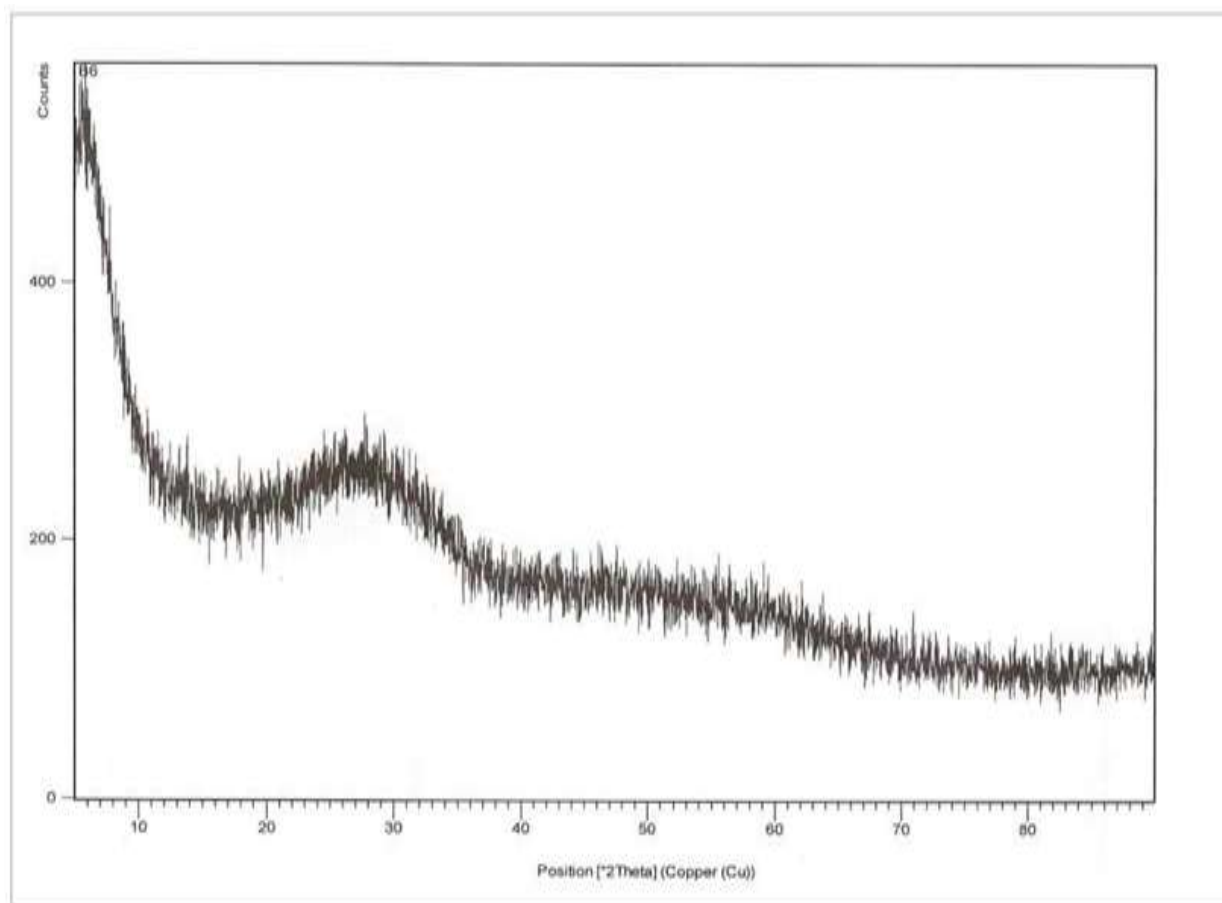
#### 4.Characterizations

We determined the synthesis of the novel product by comparing the Fourier transform infrared spectra (Fig. 3.3) of the pure components ZrOCl<sub>2</sub>, KIO<sub>3</sub>, Zr(IV)iodate, poly-o-toluidine, and the composite material poly-o-toluidineZr(IV)iodate. The presence of hydroxyl groups is indicated by a wide band between 3500 and 3200 cm<sup>-1</sup> and a metal-oxygen band at 612 cm<sup>-1</sup> in the FTIR spectrum of the poly-o-toluidineZr(IV)iodate composite material. Peaks at around 1410, 1220, and 1121 cm<sup>-1</sup> show the plane bending of the -CH bands, whereas the absorption bands at about 1502 and 1600 cm<sup>-1</sup> are associated with benzonoid and quinoid rings.



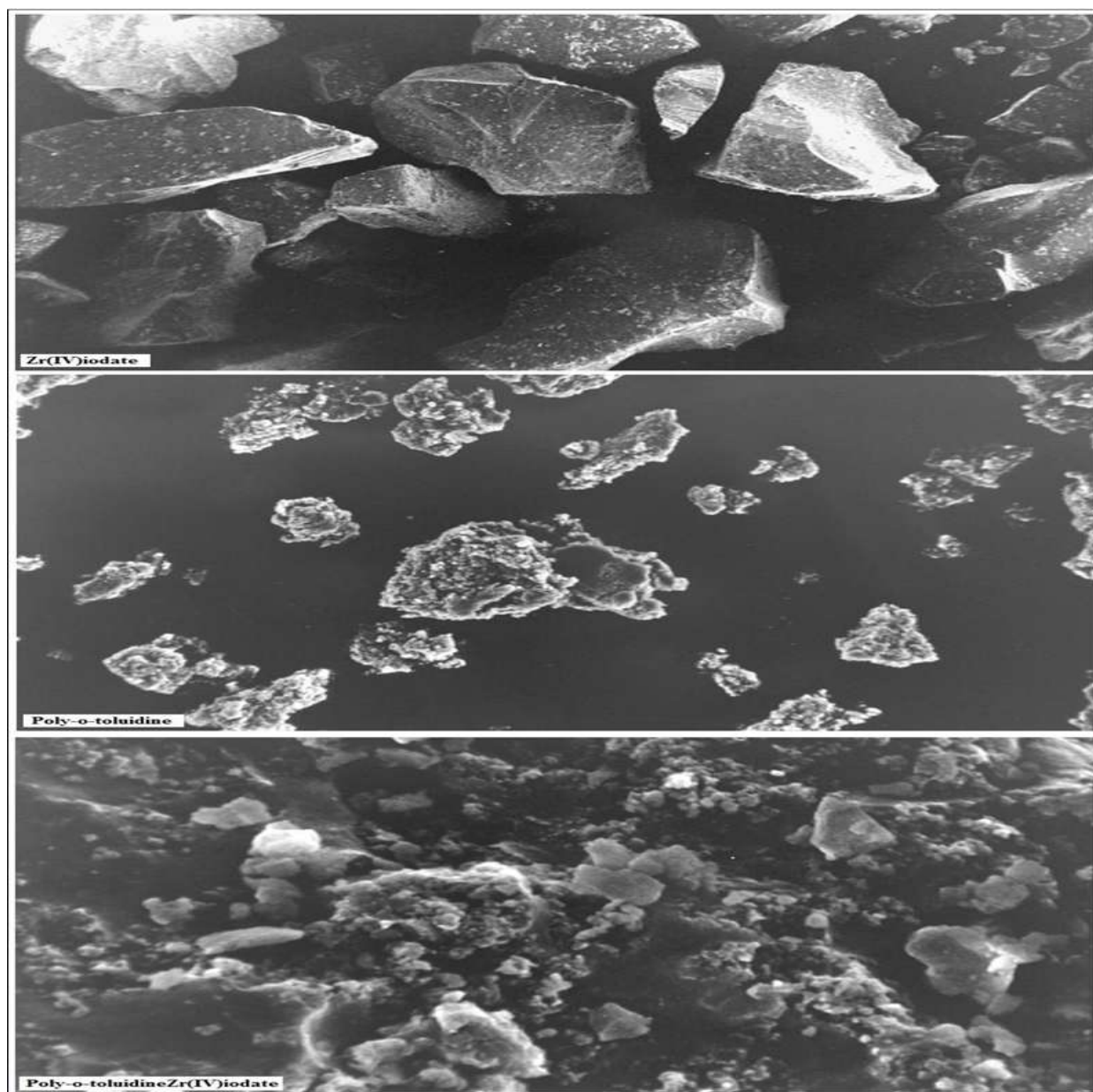
**Figure 4.1** FTIR spectra of the cation exchange adsorbents poly-o-toluidine, ZrOCl<sub>2</sub>, KIO<sub>3</sub>, Zr(IV)iodate, and poly-o-toluidineZr(IV)iodate.

According to the thermogram (Fig. 3.4.), the material loses 2% of its weight up to 100 °C as a result of evaporation of its exterior water molecules. As the temperature is increased up to 260°C, a continuous loss of mass of 15% has been seen. It is possible that the release of adsorbed interstitial water molecules is responsible for the weight drop seen in the 100-260°C range . The pyrolysis of polymer chains might explain why further weight loss happens at a larger temperature range (approx. 260-450°C). The progressive production of metal oxides might be the cause of weight loss over 402°C.



**Figure 4.2 XRD Analysis of Poly(o-toluidine)–Zr(IV) Iodate Composite.**

Scanning electron micrographs in Figure 3.6 show Zr(IV)iodate, poly-o-toluidine, and a combination of the two. A scanning electron microscopy (SEM) picture shows that the inorganic precipitate was crystalline before it was polymerized. The composite material underwent a dramatic transformation into a semicrystalline state upon binding. Additionally, it proves that composites have a high ion exchange capacity because metal ions may be adsorbed onto their many small pores, which is a result of their porous nature [27] [28] [29].



**Figure 4.3 The SEM pictures of poly-o-toluidine, Zr(IV)iodate, and Iodate cation exchanger Zr(IV).**

In addition, the TEM analysis of the microstructure revealed that the poly-o-toluidineZr(IV)iodate particles were almost spherical, with a diameter of around 50 nm (Fig. 3.7).

## **5. INDUSTRIAL EFFLUENT AND SYNTHETIC MIXTURE HEAVY METALS REMOVAL.**

Nanocomposite adsorbents are highly effective materials for the removal of heavy metals from contaminated water because of their high surface area, abundant active sites, excellent mechanical strength, and superior chemical stability. These materials are synthesized using techniques such as sol-gel processing, co-precipitation, hydrothermal synthesis, in-situ polymerization, and green synthesis, which provide precise control over their structure and surface properties. Functional groups such as hydroxyl, amino, carboxyl, and thiol enhance metal ion adsorption through ion exchange, electrostatic attraction, and surface complexation. Their structural and chemical properties are characterized using techniques including XRD, FTIR, SEM, TEM, BET, TGA, XPS, and zeta potential analysis. The adsorption performance depends on factors such as pH, contact time, adsorbent dosage, and initial metal ion concentration. Nanocomposites based on chitosan, graphene oxide, carbon nanotubes,  $\text{Fe}_3\text{O}_4$ ,  $\text{TiO}_2$ ,  $\text{ZnO}$ , alginate, and cellulose have demonstrated high adsorption capacity, rapid kinetics, excellent selectivity, and good regeneration ability. Magnetic



nanocomposites offer the additional advantage of easy separation from treated water using an external magnetic field, making them reusable and economically attractive. Despite challenges such as nanoparticle aggregation and large-scale production, ongoing advances in green synthesis and surface modification continue to improve their efficiency, sustainability, and practical application in wastewater treatment and environmental remediation [30] [31] [32].

### 5.1 Synthesis and characterization of nanocomposite adsorbents

The synthesis and characterization of nanocomposite adsorbents play a crucial role in the development of advanced materials for the efficient removal of heavy metals from contaminated water and wastewater. Nanocomposite adsorbents are engineered by combining nanostructured materials with polymeric, inorganic, carbon-based, or biopolymeric matrices to produce hybrid materials possessing high surface area, abundant active sites, improved porosity, enhanced mechanical strength, and superior chemical stability. The incorporation of nanoparticles into suitable host matrices creates a synergistic effect that significantly improves adsorption efficiency, selectivity, and regeneration capability. Various synthesis methods, including sol-gel processing, co-precipitation, hydrothermal and solvothermal synthesis, in-situ polymerization, electrospinning, chemical reduction, and green synthesis, are employed to control particle size, morphology, crystallinity, pore structure, and surface functionality. The sol-gel method enables the formation of highly porous silica-based nanocomposites through the hydrolysis and condensation of metal alkoxide precursors such as tetraethyl orthosilicate (TEOS), whereas co-precipitation is widely used to prepare magnetic nanocomposites by precipitating iron salts within polymeric matrices such as chitosan or graphene oxide. Hydrothermal and solvothermal methods facilitate the synthesis of highly crystalline nanostructures with controlled morphology, while in-situ polymerization ensures uniform dispersion of nanoparticles throughout polymer networks, resulting in enhanced interfacial interactions and improved adsorption performance. Chemical reduction techniques are commonly adopted for synthesizing metallic nanoparticle-based composites, whereas environmentally friendly green synthesis methods utilize plant extracts, microorganisms, and agricultural wastes as natural reducing and stabilizing agents to minimize chemical hazards and promote sustainability. Following synthesis, comprehensive characterization is essential to establish the relationship between structure and adsorption performance. X-ray diffraction (XRD) identifies crystalline phases and evaluates particle size, Fourier Transform Infrared Spectroscopy (FTIR) detects functional groups responsible for metal ion binding, Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM) reveal surface morphology, particle distribution, and internal nanostructure, while Brunauer–Emmett–Teller (BET) analysis determines specific surface area, pore volume, and pore size distribution that directly influence adsorption capacity and diffusion kinetics. Thermogravimetric Analysis (TGA) evaluates thermal stability and composition, zeta potential measurements determine surface charge and the point of zero charge for predicting adsorption behavior under different pH conditions, and X-ray Photoelectron Spectroscopy (XPS) provides valuable information regarding elemental composition and oxidation states of surface species. Additional analytical techniques such as Raman spectroscopy, UV–Visible spectroscopy, Atomic Absorption Spectroscopy (AAS), and Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) are widely employed to investigate optical properties, nanoparticle formation, and metal ion removal efficiency after adsorption. The combined results obtained from these characterization methods provide a comprehensive understanding of the physicochemical properties governing adsorption mechanisms and enable optimization of nanocomposite performance. Representative examples include magnetic chitosan–Fe<sub>3</sub>O<sub>4</sub> nanocomposites synthesized through co-precipitation, which exhibit excellent magnetic recoverability, abundant amino and hydroxyl functional groups, high adsorption capacity, and easy regeneration, as well as graphene oxide–TiO<sub>2</sub> nanocomposites prepared by hydrothermal synthesis that combine the large surface area of graphene oxide with the photocatalytic activity of titanium dioxide to achieve highly efficient removal of Pb(II), Cd(II), Cr(VI), and other toxic metal ions. By carefully selecting synthesis routes, optimizing reaction conditions, and employing advanced characterization techniques, researchers can tailor nanocomposite adsorbents with superior adsorption capacity, rapid kinetics, excellent stability, and high reusability, making them promising materials for sustainable wastewater treatment, environmental remediation, industrial effluent purification, and long-term management of heavy metal pollution [34] [35] [36] [37].



## 6. Conclusion

Research on composite cation-exchange materials, including their synthesis, characterization, and analytical applications in metal ion separation, has become an important area in materials science and environmental remediation due to the increasing need for efficient methods to remove toxic heavy metals from contaminated water and soil. Rapid industrialization, urbanization, mining, and agricultural activities have led to the widespread release of hazardous metal ions such as  $\text{Pb}^{2+}$ ,  $\text{Cd}^{2+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Cr}^{3+}/\text{Cr}^{6+}$ ,  $\text{Hg}^{2+}$ , and  $\text{Zn}^{2+}$  into the environment, posing serious risks to ecosystems and human health. Conventional treatment methods, including chemical precipitation, solvent extraction, membrane filtration, and traditional ion-exchange resins, often suffer from limitations such as poor selectivity, high operational costs, limited regeneration capability, and the generation of secondary pollutants. Composite cation-exchange materials overcome many of these drawbacks by combining the advantages of organic polymers and inorganic ion-exchange phases, resulting in materials with enhanced ion-exchange capacity, superior mechanical strength, improved thermal and chemical stability, higher selectivity, and faster adsorption kinetics. These composites are commonly synthesized using techniques such as sol-gel processing, co-precipitation, in-situ polymerization, hydrothermal synthesis, and impregnation methods, allowing precise control over particle size, porosity, surface area, and the distribution of functional groups. Comprehensive characterization using analytical techniques such as Fourier Transform Infrared Spectroscopy (FTIR), X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Energy Dispersive X-ray Spectroscopy (EDX), Thermogravimetric Analysis (TGA), Brunauer-Emmett-Teller (BET) surface area analysis, and ion-exchange capacity measurements confirms the successful formation and structural properties of the composites. Their analytical performance in metal ion separation is governed by factors including ionic radius, hydration energy, charge density, functional group availability, and surface morphology, enabling efficient and selective removal of trace metal ions even from complex multi-component systems. Owing to their high adsorption capacity, rapid ion-exchange kinetics, excellent regeneration ability, and long-term stability, composite cation-exchange materials have emerged as highly promising adsorbents for environmental monitoring, wastewater treatment, resource recovery, and sustainable heavy metal remediation applications.

## 7. FUTURE SCOPE

Composite cation exchange materials have great promise as a new area of research for the separation of metal ions in the environment. Industrialization, mining, and the discharge of urban garbage are causing environmental problems to worsen, making it more urgent than ever to find sustainable, efficient, and selective methods to remove harmful metal ions. Improving composite cation exchangers' synthesis, structure, performance, and multifunctionality will most certainly be the focus of future technical breakthroughs and research in this field. In addition, they herald a breakthrough in materials-based solutions for water purification, trash recycling, and environmental protection when integrated into smart, automated, and large-scale environmental management systems.

## 8. References

- 1] M. Rawat, A.L. Ramanathan, V. Subramanian, J. Hazard. Mater. 172 (2009) 1145.
- 2] Mu, Naushad. (2009). Inorganic and Composite Ion Exchange Materials and their Applications. Ion Exchange Letters. 2.
- 3] N.E. Topp, K.W. Pepper, J. Chem. Soc. (1949) 3299.
- 4] Nabi, S. & Bushra, Rani & Alothman, Zeid & Naushad, Mu. (2011). Synthesis, Characterization, and Analytical Applications of a New Composite Cation Exchange Material Acetonitrile Stannic(IV) Selenite: Adsorption Behavior of Toxic Metal Ions in Nonionic Surfactant Medium. Separation Science and Technology. 46. 10.1080/01496395.2010.534759.



- 5] Nabi, S. & Bushra, Rani & Alothman, Zeid & Naushad, Mu. (2011). Synthesis, Characterization, and Analytical Applications of a New Composite Cation Exchange Material Acetonitrile Stannic(IV) Selenite: Adsorption Behavior of Toxic Metal Ions in Nonionic Surfactant Medium. *Separation Science and Technology*. 46. 10.1080/01496395.2010.534759.
- 6] Nabi, S. & Naushad, Mu & Bushra, Rani. (2009). A New Hybrid EDTA–Zirconium Phosphate Cation-Exchanger: Synthesis, Characterization and Adsorption Behaviour for Environmental Monitoring. *Adsorption Science & Technology - ADSORPT SCI TECHNOL*. 27. 423-434. 10.1260/026361709790252641.
- 7] Nabi, S.A. & Raeissi, A.S. & Shahadat, Mohammad & Bushra, Rani & Khan, Amjad. (2012). Synthesis and characterization of novel cation exchange adsorbent for the treatment of real samples for metal ions. *Chemical Engineering Journal*. s 200–202. 426–432. 10.1016/j.cej.2012.05.086.
- 8] Nabi, S.A. & Shahadat, Mohammad & Bushra, Rani & Shalla, Aabid & Ahmed, Faheem. (2010). Development of composite ion-exchange adsorbent for pollutants removal from environmental wastes. *Chemical Engineering Journal*. 165. 405. 10.1016/j.cej.2010.08.068.
- 9] Nabi, S.A. & Shahadat, Mohammad & Bushra, Rani & Shalla, Aabid & Azam, Ameer. (2011). Synthesis and characterization of nano-composite ion-exchanger; its adsorption behavior. *Colloids and surfaces. B, Biointerfaces*. 87. 122-8. 10.1016/j.colsurfb.2011.05.011.
- 10] Nabi, Syed & Bushra, Rani & Naushad, Mu & Khan, Amjad. (2010). Synthesis, characterization and analytical applications of a new composite cation exchange material poly-o-toluidine stannic molybdate for the separation of toxic metal ions. *Chemical Engineering Journal*. 165. 529-536. 10.1016/j.cej.2010.09.064.
- 11] Nabi, Syed & Naushad, Mu. (2008). Synthesis, characterization and analytical applications of a new composite cation exchanger cellulose acetate-Zr(IV) molybdophosphate. *Colloids and Surfaces A-physicochemical and Engineering Aspects - COLLOID SURFACE A*. 316. 217-225. 10.1016/j.colsurfa.2007.09.005.
- 12] Nabi, Syed & Shalla, Aabid. (2008). EDTA-stannic(IV)iodate: Preparation, characterization and its analytical applications for metal content determination in real and synthetic samples. *Journal of Porous Materials*. 16. 587-597. 10.1007/s10934-008-9236-5.
- 13] Namdari, M. & Kikhavani, Towan & Ashrafizadeh, Seyed Nezameddin. (2017). Synthesis and characterization of an enhanced heterogeneous cation exchange membrane via nanoclay. *Ionics*. 23. 1-14. 10.1007/s11581-017-2009-x.
- 14] Naushad, Mu & Alothman, Zeid & Islam, Dr. Mahamudur. (2013). Adsorption of cadmium ion using a new composite cation-exchanger polyaniline Sn(IV) silicate: Kinetics, thermodynamic and isotherm studies. *International Journal of Environmental Science and Technology*. 10. 567-578. 10.1007/s13762-013-0189-0.
- 15] Naushad, Mu & Mittal, Alok & Rathore, M. & Gupta, V.. (2014). Ion-exchange kinetic studies for Cd(II), Co(II), Cu(II), and Pb(II) metal ions over a composite cation exchanger. *Desalination and Water Treatment*. 54. 1-8. 10.1080/19443994.2014.904823.
- 16] Nilchi, Abdolreza & Khanchi, A & Atashi, Hossein & Bagheri, Amir & Nematollahi, Laleh. (2006). The application and properties of composite sorbents of inorganic ion exchangers and polyacrylonitrile binding matrix. *Journal of hazardous materials*. 137. 1271-6. 10.1016/j.jhazmat.2006.04.043.
- 17] O. Arrad, Y. Sasson, J. Org. Chem. 54 (1989) 4993.
- 18] O.M. Vatutsina, V.S. Soldatov, V.I. Sokolova, J. Johann, M. Bissen, A. Weissenbacher, *React. Funct. Polym.* 67 (2007) 184.
- 19] Pagliuca, G.J. Mufti, *Br. Med. J.* 300 (1990) 830.



- 20] Pathania, Deepak & Naushad, Mu & Kumar, Amit. (2014). Synthesis and characterization of a new nanocomposite cation exchanger polyacrylamide Ce(IV) silicophosphate: Photocatalytic and antimicrobial applications. *Journal of Industrial and Engineering Chemistry*. 20. 3596-3603. 10.1016/j.jiec.2013.12.054.
- 21] Qureshi, Mohsin & Nabi, Syed & Zehra, Nighat. (2011). Synthesis, ion-exchange properties, and analytical applications of thermally stable tin(IV) vanadate. *Canadian Journal of Chemistry*. 55. 1667-1672. 10.1139/v77-235.
- 22] R.A. Meyers, Interpretation of Infrared Spectra, A Practical Approach, John Coates in *Encyclopedia of Analytical Chemistry*, O John Wiley & Sons Ltd, Chichester, 2000 pp 7.
- 23] R.M. Silverstein, G.C. Bassler, T.C. Morrill, Spectrometric identification of organic compounds, John Wiley and Sons, New York 1981 4th ed, Ch. 3, pp.128. (CN band)
- 24] Rahman, Nafisur & Haseen, Uzma. (2013). Synthesis and characterization of polyacrylamide zirconium (IV) iodate ion-exchanger: Its application for selective removal of lead (II) from wastewater. *Arabian Journal of Chemistry*. 4. 10.1016/j.arabjc.2013.06.029.
- 25] S.A. Nabi, A.H. Shalla, S.A. Ganai, *J. Sep. Sci.* 43 (2008) 164.
- 26] S.A. Nabi, A.H. Shalla, *Synthesis, J. Hazard. Mater.* 163 (2009) 657.
- 27] S.A. Nabi, A.M. Khan, *Ann. Chim. Sci. Mater.* 21 (1996) 521.
- 28] S.A. Nabi, Aabid H. Shalla, A.M. Khan, S.A. Ganai, *Colloids Surf. A* 302 (2007) 241.
- 29] S.A. Nabi, M. Naushad, *Chem. Eng. J.* 158 (2010) 100.
- 30] S.A. Nabi, Md. Shahadat, R. Bushra, A.H. Shalla, *Chem. Eng. J.* 175, (2011) 8.
- 31] S.A. Nabi, Md. Shahadat, R. Bushra, A.H. Shalla, Faheem Ahmed, *Chem. Eng. J.* 165 (2010) 405.
- 32] S.A. Nabi, S. Usmani, N. Rehman, *React. Funct. Polym.* 66 (2006) 495.
- 33] Shahadat, Mohammad & Shalla, Aabid & Raeissi, A.. (2012). Synthesis, Characterization, and Sorption Behavior of a Novel Composite Cation Exchange Adsorbent. *Industrial & Engineering Chemistry Research*. 51. 15525–15529. 10.1021/ie3014555.
- 34] Sharma, Pooja & Jindal, Rajeev & Maiti Jana, Mithu & Jana, Asim. (2016). Novel organic–inorganic composite material as a cation exchanger from a triterpenoidal system of dammar gum: synthesis, characterization and application. *Iranian Polymer Journal*. 25. 10.1007/s13726-016-0456-2.
- 35] Sidharaj, N. & Rajarajan, Govindasamy & Senthil, Mahalakshmi. (2024). Novel Terpolymer Resin: Synthesis, Characterization and Ion-Exchange Studies of Terpolymer and its Composite. *Asian Journal of Chemistry*. 36. 2179-2184. 10.14233/ajchem.2024.32283.
- 36] Singh, D.K. & Singh, S. & Srivastava, Bhavana. (2002). Synthesis, characterization and ion exchange properties of zirconium(IV) triethylammonium phosphate. *Indian Journal of Chemistry - Section A Inorganic, Physical, Theoretical and Analytical Chemistry*. 41. 2526-2529.]
- 37] Singh, Dhananjay & Singh, Shalini. (2004). Synthesis, characterization and analytical applications of zirconium(IV) sulphosalicylphosphate. *Indian Journal of Chemical Technology*. 11.