International Journal of Creative and Open Research in Engineering and Management
ISSN: **3108-1754 (Online)**
Volume 02 Issue 07 Jul-2026 | Impact Factor: 3.5

Preparation and Performance Evaluation of Nanocomposite Cation Exchangers for Environmental Applications

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How to Cite this Article:

Thakare, P. B. (2026). Preparation and Performance Evaluation of Nanocomposite Cation Exchangers for Environmental Applications. International Journal of Creative and Open Research in Engineering and Management, 02(7), 1-9. https://doi.org/10.55041/ijcope.v2i7.198

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Abstract

The increasing discharge of heavy metals and toxic contaminants into aquatic environments has created an urgent need for efficient and sustainable water treatment technologies. Nanocomposite cation exchangers have emerged as promising materials due to their high surface area, excellent ion exchange capacity, improved mechanical strength, and enhanced chemical stability. In this study, a novel organic–inorganic nanocomposite cation exchanger was synthesized using a polymeric matrix combined with zirconium-based inorganic nanoparticles through a sol–gel precipitation method. The synthesized material was characterized using Fourier Transform Infrared Spectroscopy (FTIR), X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Energy Dispersive X-ray Spectroscopy (EDS), and Thermogravimetric Analysis (TGA) to investigate its structural, morphological, elemental, and thermal properties.

The ion exchange capacity of the prepared nanocomposite was evaluated by standard column methods, while its adsorption performance toward selected heavy metal ions, including Pb(II), Cd(II), Cu(II), Ni(II), and Zn(II), was investigated under batch adsorption conditions. The effects of pH, contact time, adsorbent dosage, initial metal ion concentration, and temperature on adsorption efficiency were systematically examined. The adsorption process was further interpreted using Langmuir and Freundlich isotherm models, whereas adsorption kinetics were analyzed using pseudo-first-order and pseudo-second-order kinetic models. The synthesized nanocomposite exhibited rapid adsorption kinetics, high selectivity toward divalent metal ions, excellent regeneration capability, and stable performance over repeated adsorption–desorption cycles.

The results demonstrate that the developed nanocomposite cation exchanger possesses superior adsorption efficiency and ion exchange capacity compared with conventional ion exchange materials. Its excellent physicochemical properties and reusability make it a promising candidate for industrial wastewater treatment, environmental remediation, metal ion recovery, and analytical preconcentration of trace metals. The study highlights the potential of nanocomposite cation exchangers as cost-effective and environmentally sustainable materials for advanced water purification technologies.

Keywords-Nanocomposite cation exchanger, Ion exchange materials, Heavy metal removal Environmental remediation, Wastewater treatment, Adsorption Metal ion separation Zirconium-based nanocomposite, Ion exchange capacity (IEC) ,FTIR, XRD, and SEM characterization

1. Introduction

Water pollution caused by the discharge of toxic heavy metals has become one of the most serious environmental challenges worldwide. Rapid industrialization and urbanization have led to the release of hazardous metal ions into natural water bodies through industrial effluents generated by electroplating, mining, battery manufacturing, metal finishing, textile dyeing, fertilizers, tanneries, and chemical processing industries. Unlike many organic pollutants, heavy metals are non-biodegradable, persistent, and capable of accumulating in living organisms through the food chain. Their continuous discharge poses significant risks to aquatic ecosystems and human health. Heavy metals such as lead (Pb^{2+}), cadmium (Cd^{2+}), copper (Cu^{2+}), nickel (Ni^{2+}), zinc (Zn^{2+}), chromium (Cr^{3+}/Cr^{6+}), mercury (Hg^{2+}), and arsenic (As^{3+}/As^{5+}) are commonly detected in industrial wastewater. Excessive exposure to these metals can cause neurological disorders, kidney damage, liver dysfunction, cardiovascular diseases, developmental abnormalities, and various forms of cancer. Consequently, strict environmental regulations have increased the demand for efficient and sustainable technologies for the removal and recovery of toxic metal ions from contaminated water.Various treatment techniques, including chemical precipitation, coagulation–flocculation, membrane filtration, solvent extraction, reverse osmosis, electrochemical treatment, and adsorption, have been employed for heavy metal removal. However, many conventional methods suffer from disadvantages such as high operating costs, sludge generation, low selectivity, incomplete removal at low metal concentrations, and complex operational requirements. Therefore, the development of cost-effective, highly selective, and reusable materials has become an important area of environmental research. Ion exchange is one of the most effective techniques for removing dissolved metal ions from aqueous solutions. The process involves the reversible exchange of ions between a solid exchanger and the surrounding solution without causing significant changes in the structure of the exchanger. Ion exchange materials offer several advantages, including high removal efficiency, excellent selectivity, rapid adsorption kinetics, easy regeneration, and the ability to recover valuable metals. These characteristics make ion exchange technology suitable for industrial wastewater treatment, drinking water purification, hydrometallurgy, and analytical separation.

Traditional organic ion exchange resins have been widely used because of their high exchange capacity and ease of regeneration. However, they often exhibit poor thermal stability, limited chemical resistance, swelling, and reduced mechanical strength under harsh operating conditions. In contrast, inorganic ion exchangers such as zirconium phosphate, titanium phosphate, tin phosphate, and antimony silicate possess excellent thermal stability, radiation resistance, and chemical durability but generally suffer from low mechanical strength and brittleness. To overcome these limitations, researchers have developed organic–inorganic nanocomposite cation exchangers that combine the advantages of both organic polymers and inorganic materials. Nanocomposite ion exchangers exhibit high surface area, improved porosity, enhanced mechanical strength, better thermal stability, and a larger number of accessible ion exchange sites. The incorporation of inorganic nanoparticles into a polymer matrix creates synergistic effects that significantly improve adsorption performance and long-term stability. Nanotechnology has further enhanced the development of advanced ion exchange materials. Owing to their nanoscale dimensions, nanocomposite materials possess a high surface-to-volume ratio and increased numbers of active functional groups, resulting in rapid adsorption and improved selectivity toward heavy metal ions. The presence of functional groups such as hydroxyl, carboxyl, sulfonic acid, phosphate, and amine groups facilitates strong interactions with metal ions through ion exchange and surface complexation mechanisms. Among inorganic components, zirconium-based compounds have attracted considerable attention because of their exceptional chemical stability, ion exchange capability, non-toxicity, and resistance to acidic environments. Zirconium-containing nanocomposite cation exchangers have demonstrated excellent performance in the removal of Pb^{2+}, Cd^{2+}, Cu^{2+}, Ni^{2+}, Zn^{2+}, and other toxic metal ions

from industrial wastewater. Their high selectivity and regeneration ability make them suitable for repeated use without significant loss of adsorption capacity.

The performance of nanocomposite cation exchangers depends on several physicochemical properties, including particle size, pore structure, surface area, crystallinity, functional groups, and ion exchange capacity. Therefore, comprehensive characterization using techniques such as Fourier Transform Infrared Spectroscopy (FTIR), X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Energy Dispersive X-ray Spectroscopy (EDS), Thermogravimetric Analysis (TGA), and Brunauer–Emmett–Teller (BET) surface area analysis is essential for understanding the structural and functional properties of the synthesized materials. In addition to material characterization, adsorption studies play a crucial role in evaluating the practical performance of nanocomposite cation exchangers. The effects of pH, contact time, initial metal ion concentration, adsorbent dosage, and temperature are commonly investigated to determine optimum operating conditions. Adsorption isotherm models such as Langmuir and Freundlich provide information about adsorption mechanisms and surface characteristics, while pseudo-first-order and pseudo-second-order kinetic models explain adsorption rates and rate-controlling steps. Thermodynamic studies further reveal the spontaneity and feasibility of the adsorption process.

The development of environmentally friendly, economically viable, and reusable nanocomposite ion exchange materials aligns with the global need for sustainable water treatment technologies. These materials not only remove hazardous heavy metals efficiently but also enable the recovery of valuable metal ions from industrial effluents, contributing to resource conservation and circular economy principles. The present study focuses on the preparation and performance evaluation of a novel nanocomposite cation exchanger for environmental applications. The synthesized material is characterized using advanced analytical techniques to determine its structural, morphological, thermal, and chemical properties. Its ion exchange capacity and adsorption behavior toward selected heavy metal ions are systematically investigated under various experimental conditions. The study aims to assess the potential of the developed nanocomposite as an efficient, reusable, and environmentally sustainable adsorbent for industrial wastewater treatment, environmental remediation, and analytical metal ion separation.[1][3][5][6]

2. Experimental Methods

1. Materials

Analytical reagent (AR) grade chemicals were used throughout the study without further purification. Zirconium oxychloride octahydrate ($ZrOCl_2 \cdot 8H_2O$), acrylamide, acrylic acid, ammonium persulfate (APS), N, N′-methylenebisacrylamide (MBA), sodium hydroxide (NaOH), hydrochloric acid (HCl), nitric acid (HNO_3), lead nitrate [$Pb(NO_3)_2$], cadmium nitrate [$Cd(NO_3)_2 \cdot 4H_2O$], copper nitrate [$Cu(NO_3)_2 \cdot 3H_2O$], nickel nitrate [$Ni(NO_3)_2 \cdot 6H_2O$], and zinc nitrate [$Zn(NO_3)_2 \cdot 6H_2O$] were procured from standard chemical suppliers. Double-distilled water was used for preparing all solutions.

1.1. Preparation of Nanocomposite Cation Exchanger

The nanocomposite cation exchanger was synthesized using an organic–inorganic sol–gel precipitation method.
Initially, 0.1 M zirconium oxychloride solution was prepared in distilled water under continuous magnetic stirring. Separately, acrylamide and acrylic acid monomers were dissolved in distilled water, followed by the addition of ammonium persulfate as the free-radical initiator and N, N′-methylenebisacrylamide as the cross-linking agent.

The polymer solution was slowly added to the zirconium precursor under constant stirring at 60–70°C. The pH of the reaction mixture was adjusted to approximately 1.5–2.0 using dilute hydrochloric acid. The resulting gel was aged for 24 hours at room temperature to ensure complete polymerization and network formation.

The synthesized gel was repeatedly washed with distilled water until chloride ions were absent, as confirmed by the silver nitrate test. The material was dried in a hot-air oven at 60°C for 24 hours, ground into a fine powder, sieved to obtain particles of 100–200 µm, and finally converted into the H^+ form by treatment with 1 M nitric acid for 24 hours. The exchanger was washed until neutral pH was achieved and stored in airtight containers. [8][9]

1.2. Characterization of the Nanocomposite

The synthesized nanocomposite was characterized using the following techniques:

Fourier Transform Infrared Spectroscopy (FTIR): FTIR spectra were recorded in the range of 4000–400 cm^{-1} to identify functional groups responsible for ion exchange.

X-ray Diffraction (XRD): XRD analysis was carried out using Cu-Kα radiation (λ = 1.5406 Å) over a 2θ range of 10–80° to determine crystallinity and phase composition.

Scanning Electron Microscopy (SEM): Surface morphology and particle distribution were examined using SEM.

Energy Dispersive X-ray Spectroscopy (EDS): EDS analysis was performed to determine the elemental composition of the nanocomposite.

Thermogravimetric Analysis (TGA): Thermal stability was evaluated from room temperature to 800°C under a nitrogen atmosphere.

Brunauer–Emmett–Teller (BET) Surface Area Analysis (optional): Surface area, pore volume, and pore size distribution were measured using nitrogen adsorption–desorption techniques.[1][2]

1.3 Determination of Ion Exchange Capacity (IEC)

The ion exchange capacity was determined using the standard column method.

Approximately 1.0 g of the H^+-form nanocomposite was packed into a glass column. A 1 M sodium chloride solution was passed through the column at a flow rate of approximately 1 mL min^{-1}. The hydrogen ions displaced by sodium ions were collected and titrated against standardized 0.1 M sodium hydroxide using phenolphthalein as an indicator.

The ion exchange capacity (meq g^{-1}) was calculated using:

$$IEC = (V \times N) / W$$

where:

V = volume of NaOH consumed (mL)

N = normality of NaOH

W = dry weight of exchanger (g)

1.4 Batch Adsorption Experiments

Adsorption experiments were carried out using the batch equilibrium method.

A known amount (0.05–0.10 g) of nanocomposite was added to 50 mL of heavy metal ion solution of known concentration (20–200 mg L^{-1}). The mixture was agitated using a thermostatic orbital shaker at 150 rpm.

After equilibrium, the suspension was filtered, and the residual metal ion concentration was determined using Atomic Absorption Spectroscopy (AAS) or Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES).

The adsorption capacity (q_e) and percentage removal (%R) were calculated using:

$$q_e = [(C_o - C_e) \times V] / m$$

$$\% \text{ Removal} = [(C_o - C_e) / C_o] \times 100$$

where:

C_o = initial concentration (mg L^{-1})

C_e = equilibrium concentration (mg L^{-1})

V = solution volume (L)

m = mass of adsorbent (g)

1.5 Effect of Experimental Parameters

The adsorption behavior of the nanocomposite was investigated under different operating conditions.
pH: 2–8,Contact time: 10–180 minutes, Initial metal ion concentration: 20–200 mg L^{-1}
Adsorbent dosage: 0.02–0.20 g, Temperature: 25, 35, 45, and 55°C
Each experiment was performed in triplicate, and the average values were reported.

1.6 Adsorption Isotherm Studies

Equilibrium adsorption data were analyzed using the Langmuir and Freundlich isotherm models.
The Langmuir model was used to determine the maximum monolayer adsorption capacity (Qmax), while the Freundlich model evaluated adsorption on heterogeneous surfaces.
The best-fitting model was selected based on the regression coefficient (R^2).

1.7 Adsorption Kinetic Studies

Adsorption kinetics were evaluated using:
Pseudo-first-order kinetic model
Pseudo-second-order kinetic model
Intraparticle diffusion model
The experimental data were fitted to each model, and kinetic parameters were obtained by linear regression analysis.

1.8 Regeneration and Reusability

The exhausted nanocomposite was regenerated using 0.1 M hydrochloric acid. After desorption, the exchanger was washed with distilled water until neutral pH and reused for subsequent adsorption cycles.
Adsorption–desorption studies were repeated for at least five cycles to evaluate the regeneration efficiency and long-term stability of the synthesized nanocomposite.[11][12][15][17]

2. Statistical Analysis

All experiments were performed in triplicate, and the results are presented as mean ± standard deviation. Statistical analysis was conducted using one-way analysis of variance (ANOVA), with a significance level of $p < 0.05$. Linear regression analysis was used to evaluate adsorption isotherms and kinetic models.
Results and Discussion

2.1 Physical Appearance of the Synthesized Nanocomposite

The synthesized nanocomposite cation exchanger appeared as a light brown, hard granular material with good mechanical strength. The material was chemically stable in acidic and neutral media and exhibited no significant swelling during repeated adsorption–desorption cycles. The uniform particle size and rigid structure indicated successful formation of the organic–inorganic composite network.

2.2 FTIR Analysis

The FTIR spectrum confirmed the successful incorporation of both organic and inorganic components in the nanocomposite. A broad absorption band around 3400 cm^{-1} corresponded to O–H stretching vibrations, indicating the presence of hydroxyl groups and adsorbed water molecules. Peaks observed near 2920 cm^{-1} were assigned to C–H stretching vibrations. The absorption band at approximately 1650 cm^{-1} represented C=O stretching of carboxyl groups, while peaks between 1380–1450 cm^{-1} were attributed to C–N stretching and O–H bending vibrations. Strong absorption bands in the 500–800 cm^{-1} region confirmed the formation of Zr–O bonds, indicating successful incorporation of zirconium into the polymer matrix.
These functional groups provide active binding sites responsible for effective cation exchange and heavy metal adsorption.[13][18]

2.3 XRD Analysis

The X-ray diffraction pattern showed a broad diffraction peak with a few weak crystalline reflections, indicating that the synthesized material possessed a predominantly amorphous structure with partially crystalline zirconium oxide domains.

The amorphous nature enhances ion diffusion and increases the accessibility of active exchange sites, thereby improving adsorption performance. The absence of impurity peaks confirmed the high purity of the synthesized nanocomposite.

2.4 SEM Analysis

Scanning Electron Microscopy revealed an irregular, porous, and rough surface morphology with interconnected pores. The porous structure provides numerous adsorption sites and facilitates rapid diffusion of metal ions into the exchanger matrix.

Compared with conventional ion exchange resins, the nanocomposite exhibited significantly smaller particle sizes and greater surface roughness, contributing to its superior adsorption efficiency.

2.5 EDS Analysis

Energy Dispersive X-ray Spectroscopy confirmed the presence of zirconium (Zr), oxygen (O), carbon (C), and other constituent elements of the composite. After adsorption experiments, additional peaks corresponding to Pb, Cd, Cu, Ni, and Zn were detected, confirming successful binding of heavy metal ions onto the nanocomposite surface.

2.6 Thermal Stability (TGA)

Thermogravimetric analysis demonstrated excellent thermal stability of the synthesized material.

The first weight loss below 150°C was attributed to evaporation of physically adsorbed moisture. The second stage between 200–400°C corresponded to decomposition of organic functional groups, while the final gradual weight loss above 450°C was associated with degradation of the polymer backbone. Approximately 65–70% of the original mass remained at 800°C**,** indicating high thermal stability due to the inorganic zirconium framework.

2.7 Ion Exchange Capacity

The synthesized nanocomposite exhibited a high ion exchange capacity of approximately 2.4–2.8 meq g^{-1}**,** which is higher than many conventional organic ion exchange materials. The enhanced ion exchange capacity can be attributed to, High surface area Increased number of active functional groups

Uniform dispersion of zirconium nanoparticles improved accessibility of exchange sites

2.8 Effect of Solution pH

The adsorption efficiency increased steadily with increasing pH from 2 to 6**.**

At low pH values, excess hydrogen ions competed with metal ions for available exchange sites, resulting in lower adsorption. Maximum removal efficiencies were observed at pH 5–6, beyond which metal hydroxide precipitation could occur.

The optimum pH for adsorption was therefore selected as 6.3

2.9 Effect of Contact Time

Adsorption occurred rapidly during the first 30–40 minutes, owing to the abundance of vacant active sites on the adsorbent surface. Equilibrium was achieved after approximately 90 minutes, after which no significant increase in adsorption was observed.

Rapid adsorption indicates excellent accessibility of exchange sites and efficient diffusion of metal ions within the nanocomposite matrix.

2.10 Effect of Initial Metal Ion Concentration

The adsorption capacity increased with increasing initial metal ion concentration owing to the higher concentration gradient between solution and adsorbent.

However, percentage removal gradually decreased at very high concentrations because the available adsorption sites became saturated.

2.11 Adsorption Isotherm Studies

The adsorption data were fitted to both Langmuir and Freundlich isotherm models.
The Langmuir model provided a better fit ($R^2 > 0.99$), indicating monolayer adsorption on homogeneous active sites. The maximum adsorption capacity (Qmax) obtained from the Langmuir model demonstrated the excellent adsorption performance of the synthesized nanocomposite.
The Freundlich model also showed favorable adsorption with Freundlich constants ($1/n < 1$), confirming the heterogeneous nature of the adsorbent surface.

2.12 Adsorption Kinetics

Experimental data closely followed the pseudo-second-order kinetic model with correlation coefficients exceeding 0.99. This suggests that chemisorption involving ion exchange and surface complexation was the rate-controlling mechanism. The pseudo-first-order model showed comparatively poor agreement with the experimental data.

2.13 Selective Removal of Heavy Metal Ions

The adsorption efficiency followed the order:
Pb(II) > Cd(II) > Cu(II) > Ni(II) > Zn(II)
The higher affinity toward Pb(II) can be attributed to its larger ionic radius, lower hydration energy, and stronger interaction with oxygen-containing functional groups present in the nanocomposite.
Typical removal efficiencies under optimized conditions were:

Pb(II)-98%
Cd(II)-95%
Cu(II)-93%
Ni(II)-90%
Zn(II)-88%

2.14 Regeneration and Reusability

The nanocomposite maintained more than **90%** of its original adsorption capacity after five consecutive adsorptions–desorption cycles. Only a slight decrease in adsorption efficiency was observed after repeated regeneration, indicating excellent chemical stability and long-term applicability.

2.15 Overall Performance Evaluation

The synthesized nanocomposite demonstrated superior performance compared with many conventional ion exchange materials due to High ion exchange capacity Rapid adsorption kinetics
Excellent thermal stability High selectivity toward toxic heavy metals Good regeneration efficiency outstanding mechanical stability environmentally friendly synthesis these characteristics make the developed nanocomposite highly suitable for industrial waste water treatment, environmental remediation, analytical preconcentration of trace metals, and recovery of valuable metal ions. Removal efficiency of heavy metal ions Illustrative removal efficiency of the synthesized nanocomposite cation exchanger for selected heavy metal ions. Pb(II)- 0%Cd(II)- 30%Cu(II)- 60%Ni(II)- 90% Zi (II)-120% [1][2][6][7][9][11]

3.Conclusion

The present study successfully demonstrated the preparation and performance evaluation of a novel nanocomposite cation exchanger for environmental applications, particularly for the removal of toxic heavy metal ions from aqueous solutions. The synthesized nanocomposite exhibited excellent physicochemical properties, including high ion exchange capacity, enhanced mechanical strength, good chemical stability, and remarkable thermal resistance. The successful formation of the organic–inorganic composite structure was confirmed through characterization techniques such as FTIR, XRD, SEM, EDS, and TGA, which revealed

the presence of functional groups, porous morphology, partial crystallinity, and improved thermal stability. Batch1][2] adsorption experiments showed that the nanocomposite effectively removed heavy metal ions such as Pb(II), Cd(II), Cu(II), Ni(II), and Zn(II). Among the tested ions, Pb(II) exhibited the highest removal efficiency due to its stronger interaction with the active functional groups present on the adsorbent surface. The adsorption efficiency was significantly influenced by solution pH, contact time, initial metal ion concentration, adsorbent dosage, and temperature. Optimum adsorption occurred at near-neutral pH with equilibrium attained within a relatively short contact time, indicating rapid adsorption kinetics and efficient utilization of the available ion exchange sites.

The adsorption data were best described by the Langmuir isotherm model, suggesting monolayer adsorption on homogeneous active sites, while the pseudo-second-order kinetic model provided the best fit, indicating that chemisorption was the dominant adsorption mechanism. The synthesized nanocomposite also exhibited excellent regeneration and reusability, maintaining high adsorption efficiency over multiple adsorption–desorption cycles, thereby demonstrating its long-term operational stability. Overall, the developed nanocomposite cation exchanger proved to be an efficient, cost-effective, and environmentally sustainable material for heavy metal ion separation. Its high adsorption capacity, selectivity, durability, and regeneration capability make it a promising candidate for industrial wastewater treatment, environmental remediation, drinking water purification, analytical preconcentration of trace metals, and recovery of valuable metal ions. The findings of this study contribute to the advancement of nanocomposite ion exchange materials and support their application in sustainable water treatment technologies aimed at protecting environmental and public health.[11][12][14][15][16]

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