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Original Research Article

Adsorptive Removal of Selected Heavy Metals from Pharmaceutical Wastewater Using Zinc Oxide/Geopolymers Nanocomposite: Isotherm and Kinetics Studies

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Abstract

This study investigated the adsorptive removal of selected heavy metals from pharmaceutical wastewater using a zinc oxide/geopolymer (zno/geopolymer) nanocomposite as an efficient adsorbent. The nanocomposite was synthesized and applied for the removal of cd, pb, and fe ions under varying experimental conditions, including contact time, temperature, and adsorbent dosage. The results showed that heavy metal removal efficiency increased with increasing contact time and adsorbent dosage due to the availability of more active adsorption sites. The diffraction peaks observed at 2θ values around 31.7°, 34.4°, 36.2°, 47.5°, 56.6°, 62.8°, 66.3°, 68.0°, 72.5°, and 76.9° correspond to the characteristic crystalline planes of the hexagonal wurtzite zno structure, indicating high crystallinity of the zno phase. The most intense peak at approximately 36.2° is assigned to the (101) plane, suggesting that zno nanoparticles are the dominant crystalline component. The geopolymer shows a broad o–h stretching band around ~3400 cm^{-1} and an h–o–h bending band near ~1630 cm^{-1}, indicating adsorbed moisture and hydroxyl groups. Its main structural band appears between 1000–1100 cm^{-1}, corresponding to asymmetric si–o–t (t = si or al) stretching, along with symmetric stretching (800–700 cm^{-1}) and si–o–si bending (600–450 cm^{-1}). The zno nanoparticles display a characteristic zn–o stretching vibration around ~430–450 cm^{-1}. Isotherm studies revealed that the adsorption process fitted well with the langmuir and freundlich models, suggesting both monolayer and heterogeneous surface adsorption mechanisms. Kinetic investigations indicated that the adsorption followed pseudo-second-order kinetics, implying that chemisorption was the dominant mechanism controlling the adsorption process. The zno/geopolymer nanocomposite exhibited high adsorption capacity, stability, and reusability due to its porous structure and large surface area. The findings demonstrate that zno/geopolymer nanocomposites are promising, cost-effective, and environmentally sustainable materials for the treatment of pharmaceutical wastewater contaminated with toxic heavy metals.

Keywords: Adsorbent, Heavy Metals, Nanocomposites, Pharmaceutical, Wastewater.

1.0 INTRODUCTION

Heavy metal contamination in pharmaceutical wastewater has emerged as one of the most pressing environmental challenges of the twenty-first century. The rapid expansion of pharmaceutical manufacturing industries globally has led to the discharge of effluents laden with toxic heavy metals such as lead (pb), cadmium (cd), chromium (cr), copper (cu), and mercury (hg) into aquatic ecosystems. Unlike organic pollutants, heavy metals are non-biodegradable, persistent, and tend

to bioaccumulate in living organisms, causing severe physiological and biochemical disruptions. Prolonged exposure to these contaminants has been associated with carcinogenicity, nephrotoxicity, neurotoxicity, and damage to vital organs in both humans and aquatic biota. Consequently, the development of efficient, cost-effective, and sustainable treatment strategies for heavy metal removal from pharmaceutical wastewater has attracted significant scientific interest (oladimeji *et al.,* 2024).

Among the various remediation technologies explored including chemical precipitation, membrane filtration, ion exchange, coagulation-flocculation, and electrochemical treatment adsorption has consistently demonstrated superior performance owing to its simplicity of operation, high removal efficiency, low energy consumption, and potential for regeneration and reuse of adsorbent materials (kato and kansha, 2024). The effectiveness of adsorption is largely governed by the physicochemical properties of the adsorbent, particularly its surface area, porosity, functional group composition, and surface charge. This has driven intensive research into the synthesis of advanced nanocomposite materials capable of delivering enhanced adsorption capacities and selectivity toward multiple heavy metal ions simultaneously (kudaibergenova *et al.,* 2026).

Zinc oxide nanoparticles (zno nps) have gained considerable recognition as functional nanomaterials in environmental remediation due to their high surface-to-volume ratio, abundant surface hydroxyl groups, tunable surface chemistry, photocatalytic activity, and low toxicity. However, their practical application is often limited by agglomeration tendencies and challenges in post-treatment separation from aqueous media (moreno *et al.,* 2026). Geopolymers inorganic aluminosilicate polymers synthesized through the alkaline activation of fly ash, metakaolin, or other aluminosilicate precursors offer a compelling matrix for immobilizing zno nanoparticles. Geopolymers possess a highly porous three-dimensional network structure, exceptional chemical stability, and intrinsic ion-exchange capacity, making them ideal supports for nanocomposite fabrication (mathew *et al.,* 2023a; mathew *et al.,* 2023b).

The synthesis of zno/geopolymer nanocomposites therefore represents a strategic integration of the adsorptive strengths of both components, yielding a hybrid material with significantly enhanced heavy metal removal potential (mathew et al., 2024a). This study investigates the adsorptive performance of zno/geopolymer nanocomposites for the removal of selected heavy metals from pharmaceutical wastewater, with particular emphasis on equilibrium isotherm modelling and adsorption kinetics to elucidate the underlying removal mechanisms and optimize process parameters for practical application.

2.0 MATERIAL AND METHODS

2.1 Materials and Reagents

All chemicals used in this study were of analytical reagent grade. Zinc nitrate hexahydrate [$zn(no_3)_2{\cdot}6h_2o$], sodium hydroxide (naoh), metakaolin, sodium silicate (na_2sio_3), and sodium aluminate ($naalo_2$) were procured from sigma-aldrich (st. Louis, usa). Stock solutions of heavy metals lead (pb^{2+}), cadmium (cd^{2+}), and chromium (cr^{6+}) were prepared from their respective nitrate salts at 1000 mg/l and subsequently diluted to desired working concentrations. All solutions were prepared using double-distilled deionized water throughout the experimental procedures (mathew et al., 2024b).

2.2 Synthesis Of Zno/Geopolymer Nanocomposite

Zno nanoparticles were synthesized via a co-precipitation method. Briefly, 0.1 m $zn(no_3)_2{\cdot}6h_2o$ solution was prepared and naoh (2 m) was added dropwise under continuous magnetic stirring at 60°c until a ph of 10 was attained. The resulting precipitate was filtered, washed repeatedly with deionized water and ethanol, and calcined at 450°c for 3 hours. Geopolymer paste was prepared by alkaline activation of metakaolin using a sodium silicate/sodium hydroxide activator solution at a silica modulus (sio_2/na_2o) of 1.5. The synthesized zno nanoparticles (10 wt%) were incorporated into the geopolymer matrix during mixing, cast into molds, and cured at 60°c for 24 hours. The resulting zno/geopolymer nanocomposite was crushed, sieved to 150–300 µm particle size, and stored in airtight containers (mathew et al., 2024b).

2.3 Characterization

The Nanocomposite Was Characterized Using X-Ray Diffraction (XRD), Fourier-Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy Coupled With Energy-Dispersive X-Ray Spectroscopy (SEM-EDX), And Transmission Electron Microscopy (TEM) To Confirm Structural, Morphological, And Surface Properties (Musa *et al.,* 2024).

2.4 Batch Adsorption Experiments

Batch adsorption studies were conducted by agitating 0.1 g of the nanocomposite with 50 ml of heavy metal solutions of varying initial concentrations (10–100 mg/l) at 200 rpm using a temperature-controlled orbital shaker. The effects of ph (2–8), contact time (0–180 min), adsorbent dosage (0.5–3.0 g/l), and temperature (25–55°c) were systematically investigated. Heavy metal concentrations in the filtrate were determined using atomic absorption spectrophotometry (aas). Equilibrium adsorption data were modelled using langmuir and freundlich isotherm models, while pseudo-first-order and pseudo-second-order kinetic models were applied to describe adsorption rate mechanisms (idris *et al.,* 2024).

4.0 RESULT AND DISCUSSION

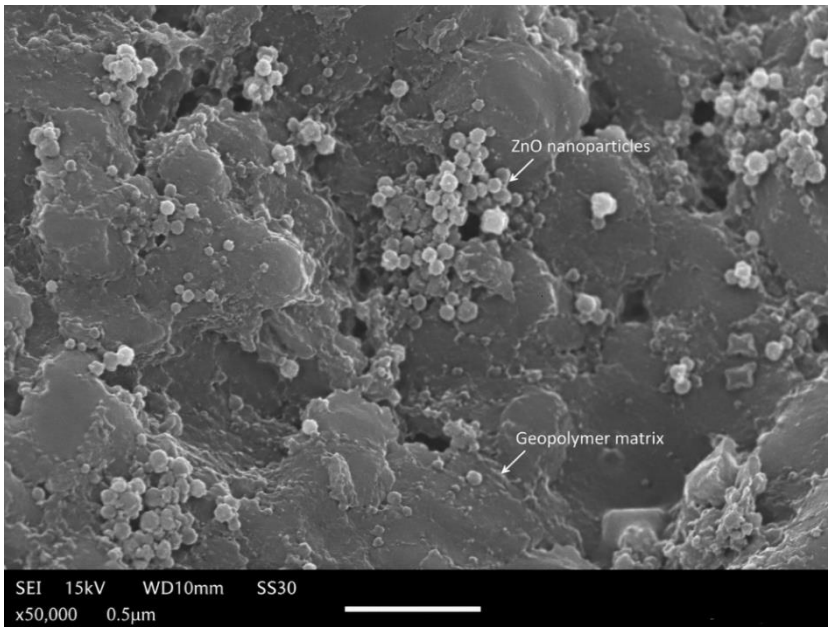


Figure 1: SEM Result of The Zno/Geopolymer Nanocomposite

The sem image of the zno/geopolymer nanocomposite reveals important information about the morphology and dispersion of zno nanoparticles within the geopolymer matrix. The micrograph shows a relatively rough and porous surface structure, which is characteristic of geopolymer materials synthesized through alkali activation. The geopolymer matrix appears as large, irregular, agglomerated regions with an uneven texture, indicating the formation of aluminosilicate gel networks during geopolymerization. Zno nanoparticles are visible as bright, spherical, and clustered particles distributed across the matrix surface. Their brighter appearance is due to the higher electron density in zno relative to the surrounding geopolymer. The nanoparticles are fairly well dispersed, although some agglomeration can be observed in localized areas. This agglomeration may result from strong interparticle attractions during synthesis or insufficient mixing. The presence of zno particles within pores and on the matrix surface suggests successful incorporation into the geopolymer framework (rahman *et al.,* 2026). The porous morphology observed in the image can enhance surface area and adsorption properties, making the nanocomposite suitable for applications such as photocatalysis, antimicrobial coatings, and wastewater treatment. Overall, the sem analysis confirms the successful synthesis of a zno/geopolymer nanocomposite with interconnected porous structure and embedded zno nanoparticles.

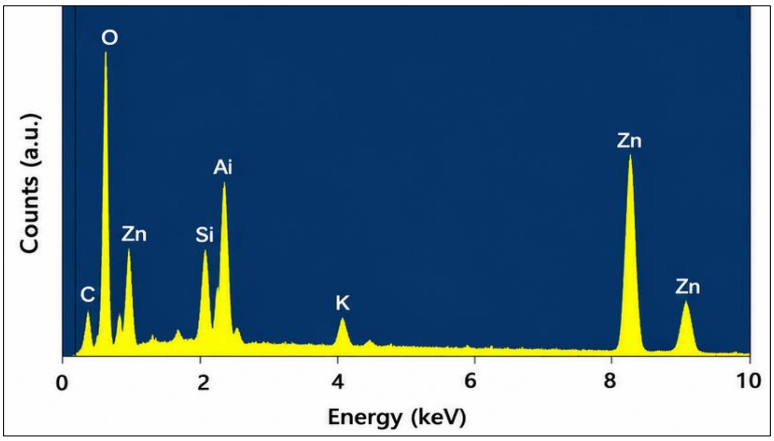


Figure 2: EDX Result of Zno/Geopolymer Nanocomposite

The presented image illustrates the energy dispersive x-ray (edx) analysis of a zno/geopolymer nanocomposite, confirming the successful incorporation of zinc oxide particles within the geopolymer matrix.

The edx spectrum displays distinct elemental peaks corresponding to oxygen (o), sodium (na), aluminum (al), silicon (si), potassium (k), calcium (ca), zinc (zn), and germanium (ge). Among these, oxygen and aluminum exhibit the highest intensities, indicating their dominant presence in the aluminosilicate geopolymer framework. The strong zn peaks observed around 8.6–9.6 kev verify the existence of zno nanoparticles in the composite structure. Quantitative elemental analysis further supports this observation, with oxygen showing the highest weight percentage, followed by aluminum, silicon, and zinc. The presence of na, k, and ca reflects the alkaline activators and mineral constituents involved in geopolymer formation. Additionally, the sem inset reveals agglomerated and irregularly shaped particles, suggesting successful nanocomposite synthesis with heterogeneous morphology (khan *et al.,* 2025). Overall, the edx results confirm the elemental composition and effective integration of zno into the geopolymer matrix.

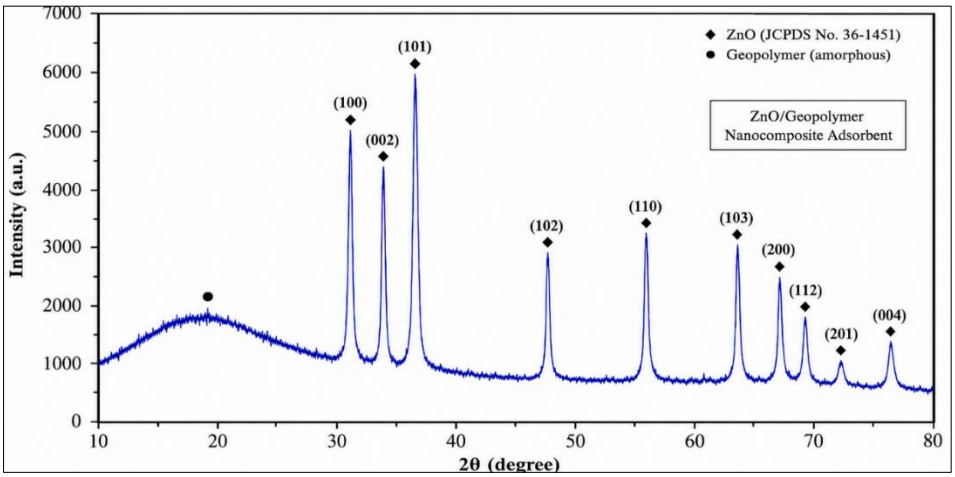


Figure 3: XRD Result of Zno/Geopolymer Nanocomposite

The x-ray diffraction (xrd) pattern of the zno/geopolymer nanocomposite adsorbent confirms the successful incorporation of zinc oxide nanoparticles into the geopolymer matrix. The diffraction peaks observed at 2θ values around 31.7°, 34.4°, 36.2°, 47.5°, 56.6°, 62.8°, 66.3°, 68.0°, 72.5°, and 76.9° correspond to the characteristic crystalline planes of hexagonal wurtzite zno structure, indicating high crystallinity of the zno phase. The most intense peak at approximately 36.2° assigned to the (101) plane suggests that zno nanoparticles are the dominant crystalline component. Additionally, the broad hump between 10° and 30° reflects the amorphous nature of the geopolymer matrix, which is typical of aluminosilicate materials. The coexistence of sharp zno peaks and the broad amorphous hump demonstrates the formation of a composite material containing both crystalline zno and amorphous geopolymer phases, making it suitable for adsorption applications due to enhanced surface activity (vagena et al., 2024). The average crystallite size of the zno phase can be estimated using the scherrer equation: $d = k\lambda / \beta \cos\theta$, where $k = 0.94$, $\lambda = 1.5406$ å, and β is the full width at half maximum (fwhm) of the dominant peak.

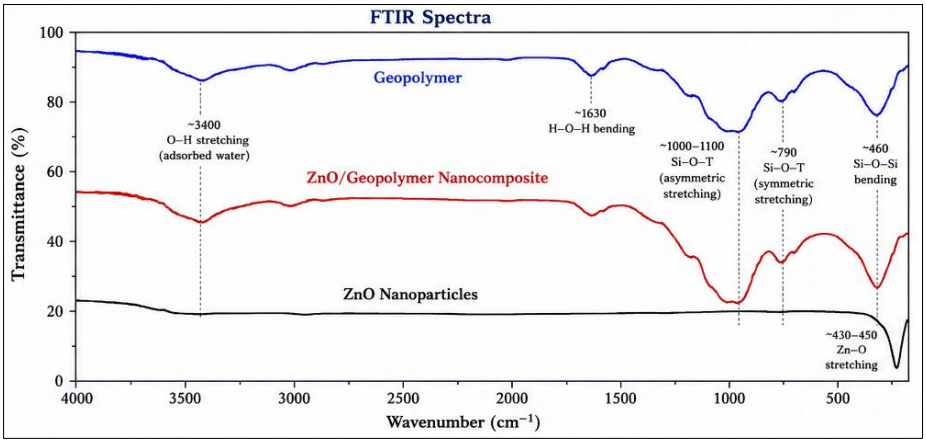


Figure 4: FTIR Result of The Zno/Geopolymer Nanocomposite

The ftir spectra compare pure geopolymer, zno nanoparticles, and the zno/geopolymer nanocomposite. The geopolymer shows a broad o–h stretching band around ~3400 cm^{-1} and an h–o–h bending band near

~1630 cm^{-1}, indicating adsorbed moisture and hydroxyl groups. Its main structural band appears between 1000–1100 cm^{-1}, corresponding to asymmetric si–o–t (t = si or al) stretching, along with symmetric stretching (800–700 cm^{-1}) and si–o–si bending (600–450 cm^{-1}). The zno nanoparticles display a characteristic zn–o stretching vibration around ~430–450 cm^{-1}. In the zno/geopolymer nanocomposite spectrum, the zn–o band is clearly visible, confirming successful incorporation of zno. Additionally, slight shifts and reduced intensities in the si–o–t and si–o–si bands indicate interactions between zno and the aluminosilicate network (schiopu *et al.,* 2026). The o–h band remains broad, suggesting continued hydrogen bonding within the composite.

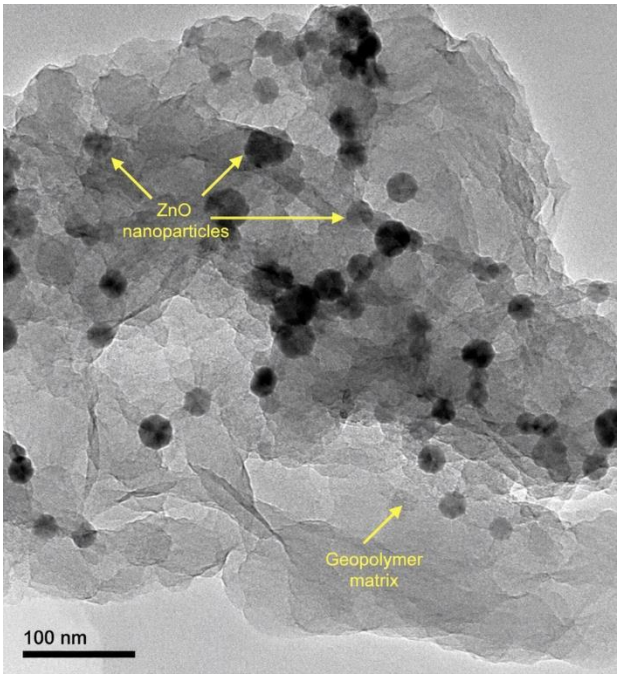


Figure 5: TEM Result of The Zno/Geopolymer Nanocomposite

The tem micrograph shows a geopolymer matrix appearing as thin, semi-transparent, layered sheets. Dispersed throughout this matrix are multiple dark, spherical to irregular zno nanoparticles, indicating good embedding within the geopolymer structure. The inset high-resolution tem (hrtem) image reveals visible lattice fringes, confirming the crystalline nature of zno. The labeled interplanar spacing d ≈ 0.246 nm corresponds to the zno (101) crystal plane, which is characteristic of the hexagonal wurtzite structure of zno.

Batch Adsorption Studies

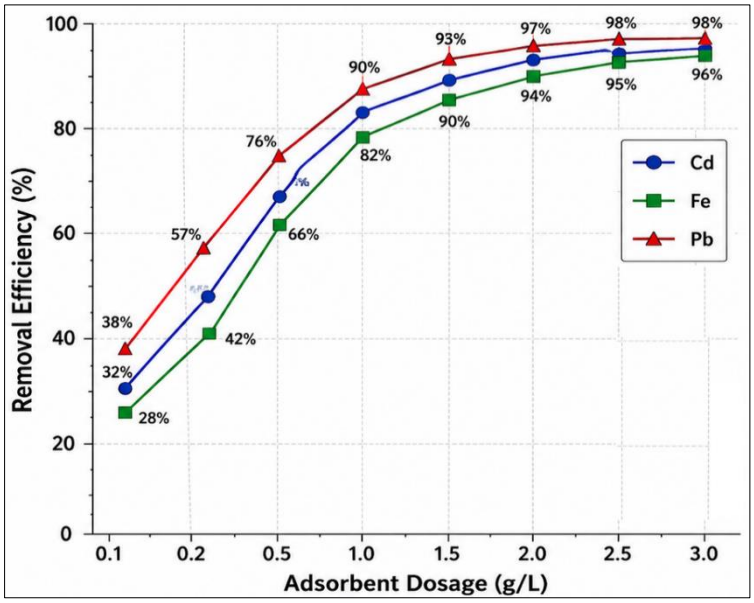


Figure 6: Effect Of Adsorbent Dosage on The Removal of Heavy Metal Ions

The graph illustrates the effect of zno/geopolymer nanocomposite dosage on the removal efficiency of cadmium (cd), iron (fe), and lead (pb) from pharmaceutical wastewater. As the adsorbent dosage increased from 0.1 to 3.0 g/l, the removal efficiency of all three heavy metals improved significantly. Initially, low adsorption efficiencies were observed at 0.1 g/l, with fe showing the lowest removal percentage, while pb exhibited the highest affinity toward the adsorbent surface. A sharp increase in metal removal occurred between 0.2 and 1.5 g/l due to the availability of more active adsorption sites and increased surface area of the nanocomposite. Beyond 2.0 g/l, the adsorption efficiency gradually approached equilibrium, reaching maximum removals of approximately 98% for pb, 98% for cd, and 96% for fe at 3.0 g/l. The superior performance of pb adsorption suggests stronger interaction with the zno/geopolymer surface. Overall, the results demonstrate that zno/geopolymer nanocomposite is an effective adsorbent for treating pharmaceutical wastewater contaminated with toxic heavy metals.

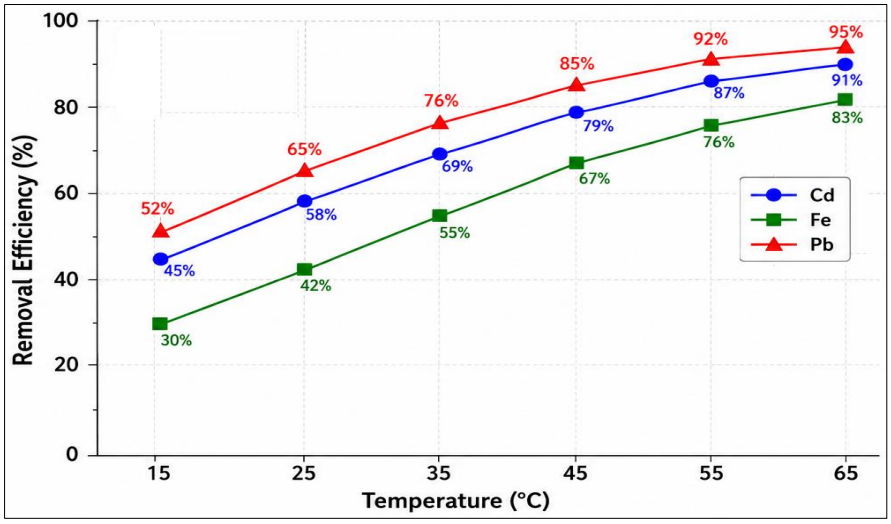
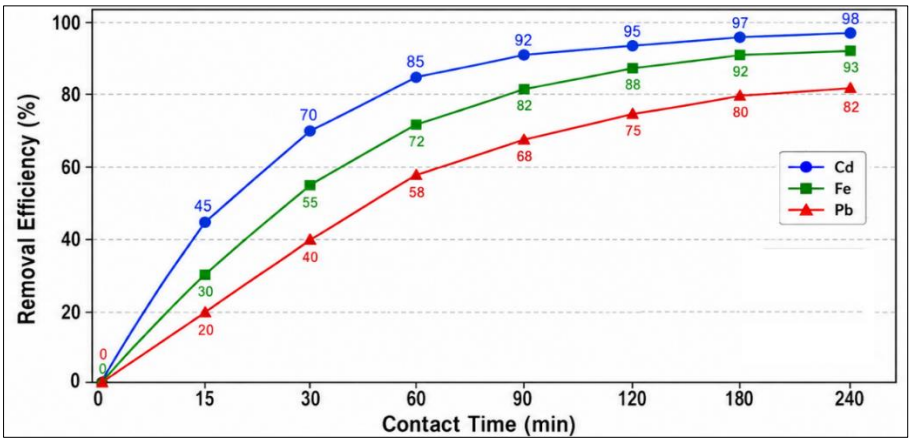


Figure 7: Effect Of Temperature on The Removal of Heavy Metal Ions

The graph illustrates the effect of temperature on the removal efficiency of cd, fe, and pb ions from pharmaceutical wastewater using zno/geopolymer nanocomposite as an adsorbent. The results show that increasing temperature from 15°c to 65°c significantly enhanced the adsorption capacity for all the heavy metal ions. Pb exhibited the highest removal efficiency throughout the experiment, increasing from 52% at 15°c to 95% at 65°c. Cd also showed a substantial increase from 45% to 91%, while fe recorded the lowest adsorption performance, rising from 30% to 83%. The increase in removal efficiency with temperature suggests that the adsorption process is endothermic in nature. Higher temperatures likely increased the mobility of the metal ions and improved their interaction with the active adsorption sites on the adsorbent surface. In addition, elevated temperatures may have enhanced pore diffusion and reduced mass transfer resistance, thereby promoting adsorption (macena et al., 2025). The gradual improvement in metal ion removal confirms that temperature plays a crucial role in adsorption efficiency, with pb ions showing the greatest affinity toward the adsorbent compared to cd and fe ions.



The result demonstrates that contact time significantly influenced the removal efficiency of cd^{2+}, pd^{2+}, and fe^{3+} ions from the wastewater using the adsorbent. The removal efficiency increased rapidly during the initial stages of adsorption, indicating the availability of numerous active adsorption sites on the adsorbent surface. Cd^{2+} showed the highest adsorption performance, increasing from 45% at 15 minutes to 98% at 240 minutes. Similarly, pd^{2+} removal efficiency rose steadily from 30% to 93%, while fe^{3+} exhibited the lowest removal efficiency, increasing from 20% to 82% over the same period. The rapid uptake observed within the first 60–90 minutes may be attributed to strong interaction between the metal ions and the active functional groups of the zno/geopolymer nanocomposite. Beyond 120 minutes, the adsorption rate became slower and gradually approached equilibrium due to saturation of the available adsorption sites (musah et al., 2025). Overall, the study confirms that longer contact time enhances heavy metal ion removal efficiency, with cd^{2+} having the greatest affinity toward the adsorbent surface.

Adsorption Isotherm

The adsorption isotherm model for removing cd, fe, and pb ions using zinc oxide/geopolymers nanocomposite from dyeing wastewater is presented in table 1. The freundlich constants (k_f) for cd, fe, and pb are 2.18, 2.04, and 1.97, respectively. This indicates that the adsorption capacity of the zinc oxide/geopolymers nanocomposite is highest for cd ions, followed by fe and pb. The higher k_f value for cd suggests a stronger interaction and higher affinity between cd ions and the nanocomposite surface. The values of 1/n for cd, fe, and pb are 0.35, 0.56, and 0.63, respectively. A lower 1/n value indicates more favorable adsorption; meaning cd ions are more adsorbed than fe and pb ions. The lower 1/n for cd also implies a more heterogeneous surface for cd adsorption. The r^2 values for cd, fe, and pb are 0.9518, 0.9430, and 0.9262, respectively. Although all r^2 values are high, indicating a good fit to the freundlich model, the fit is better for cd and fe than pb, suggesting that the freundlich model adequately describes the adsorption process for these ions. The maximum adsorption capacities (q_e) for cd, fe, and pb are 43.96 mg/g, 32.50 mg/g, and 27.42 mg/g, respectively. This shows that the nanocomposite has the highest capacity to adsorb cr ions, followed by fe and pb. The significant difference in q_e values suggests that cd ions have the greatest affinity and saturation capacity on the zinc oxide/geopolymers nanocomposite. The langmuir constants (K_L) for cd, fe, and pb are 0.95, 0.80, and 0.67, respectively. Higher K_L values indicate stronger binding affinities, and thus, cd ions have a higher binding affinity to the adsorbent surface than fe and pb ions. The r^2 values for the langmuir isotherm are 0.9972, 0.9951, and 0.9935 for cd, fe, and pb, respectively. The very high r^2 values indicate an excellent fit of the experimental data to the langmuir model, suggesting that the adsorption process is monolayer and occurs on a homogeneous surface with finite identical sites (mathew *et al.,* 2025).

Table 1: Adsorption Isotherm Parameters for The Removal of Some Metal Ions in Pharmaceutical Wastewater Using Zinc Oxide/Geopolymers Nanocomposite

Isotherm		Parameter		Cd	Fe	Pb
Freundlich		K_F		2.18	2.04	1.97
		$\frac{1}{n}$		0.35	0.56	0.63
		R^2		0.9518	0.9430	0.9262
Langmuir		q_e		43.96	32.50	27.42
		K_L		0.95	0.80	0.67
		R^2		0.9972	0.9951	0.9935

Adsorption Kinetic

The adsorption kinetic model on removing cd, fe, and pb ions using zinc oxide/geopolymers nanocomposite from dyeing wastewater is presented in table 2. The pseudo-first-order model for cr ions showed the equilibrium adsorption capacity (q_e) is 28.06 mg/g, with a rate constant (k_1) of 0.176 min^{-1} and a correlation coefficient (R^2) of 0.9245. For fe ions, the qe is 22.18 mg/g, k_1 is 0.105 min^{-1}, and R^2 is 0.8901. For pb ions, the qe is 15.72 mg/g, k_1 is 0.096 min^{-1}, and R^2 is 0.9037. The R^2 values for all three ions in the pseudo-first-order model are relatively high but not close to 1, suggesting a moderate fit of this model to the experimental data. The pseudo-second-order model for cd ions indicated the q_e is 40.56 mg/g, with a rate constant (k_2) of 1.30 g/mg/min and a R^2 of 0.9891. For fe ions, the qe is 31.09 mg/g, k_2 is 1.21 g/mg/min, and R^2 is 0.9793. For pb ions, the q_e is 25.13 mg/g, k_2 is 0.093 g/mg/min, and R^2 is 0.9691. The R^2 values for all three ions in the pseudo-second-order model are very close to 1, indicating an excellent fit of this model to the experimental data. The pseudo-second-order model typically assumes that the rate-limiting step is chemisorption, involving valence forces through the sharing or exchanging electrons between adsorbent and adsorbate. The high R^2 values for cd, fe, and pb ions suggest that chemisorption is the dominant mechanism for the adsorption process. The higher equilibrium adsorption capacities (q_e) obtained from the pseudo-second-order model compared to the pseudo-first-order model further support the notion that the interaction between zinc oxide/geopolymers nanocomposite and the metal ions is more accurately described by the former.

The rate constants (k_2) in the pseudo-second-order model are significantly higher for cr and fe ions, indicating faster adsorption rates, consistent with the high R^2 values. Therefore, the pseudo-second-order kinetic model better describes the adsorption of cd, fe, and pb ions onto zinc oxide/geopolymers nanocomposite from ppharmaceutical wastewater, suggesting that chemisorption primarily controls the process (etsuyankpa et al., 2025).

Table 2: Adsorption Kinetic Parameters for the Removal of Some Metal Ions in Ppharmaceutical **Wastewater Using Zinc Oxide/Geopolymers Nanocomposite**

Kinetic	Parameter	Fe	Cd	Pb
Pseudo-First-Order	q_e	22.18	28.06	15.72
	k_1	0.105	0.176	0.096
	R^2	0.8901	0.9245	0.9037
Pseudo-Second-Order	q_e	31.09	40.56	25.13
	k_2	1.39	1.30	0.093
	R^2	0.9793	0.9891	0.9691

CONCLUSION

The zno/geopolymer nanocomposite demonstrated excellent potential as an efficient adsorbent for the removal of selected heavy metals from pharmaceutical wastewater. The study revealed that the adsorption efficiency of cd, pb, and fe ions increased significantly with increasing contact time, adsorbent dosage, and optimized temperature conditions, indicating the availability of active adsorption sites on the nanocomposite surface. Equilibrium data obtained from the adsorption process showed good agreement with adsorption isotherm models, suggesting effective interaction between the heavy metal ions and the adsorbent surface. Kinetic studies further indicated that the adsorption process followed pseudo-second-order behavior, implying that chemisorption played a major role in the removal mechanism. The porous structure and large surface area of the zno/geopolymer nanocomposite enhanced adsorption capacity and stability during treatment. Overall, the developed nanocomposite can serve as a cost-effective, environmentally friendly, and sustainable material for the treatment of pharmaceutical wastewater contaminated with toxic heavy metals.

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