



Article

Fabrication of Activated Carbon@NiFe-Layered Double Hydroxides/Polyether Sulfone Nanocomposite Beads for the Adsorptive Removal of Trace Cadmium (Cd^{2+}) and Lead (Pb^{2+}) from Wastewater

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Abstract

The removal of toxic trace metals such as cadmium (Cd^{2+}) and lead (Pb^{2+}) from wastewater is critical due to their persistence, bioaccumulation, and adverse health effects. In this study, a novel composite adsorbent was synthesized by integrating activated carbon with nickel–iron-layered double hydroxides (NiFe-LDH) and immobilizing the resulting nanocomposite within Polyether sulfone (PES) beads to improve stability, handling, and recyclability. The material was evaluated under varying pH, initial metal concentration, and contact time conditions. The adsorption behavior was investigated using four isotherm models and two kinetic models. The composite beads exhibited maximum adsorption capacities of 1.784 mg g^{-1} for Cd^{2+} and 5.882 mg g^{-1} for Pb^{2+} . The Cd^{2+} adsorption followed the Langmuir isotherm model ($R^2 = 0.995$), indicating a homogeneous monolayer adsorption, whereas Pb^{2+} adsorption was best described by the Freundlich model ($R^2 = 0.955$), suggesting heterogeneous surface interactions and multiple binding sites. The kinetic analysis showed that the adsorption of both metals followed a pseudo-second-order model, supporting chemisorption as the dominant rate-controlling mechanism. The AC@NiFe-LDH/PES beads demonstrated high efficiency, structural integrity, and ease of recovery over multiple cycles, highlighting their potential as a sustainable and environmentally friendly adsorbent for trace metal removal from contaminated water.

Keywords: Polyether sulfone (PES) beads; trace metals; AC@NiFe-LDH/PES; environmental remediation; adsorption



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1. Introduction

The presence of trace metals such as Cd^{2+} and Pb^{2+} in wastewater is a major environmental concern due to their toxic effects on human health and the environment [1–3]. These metals can enter wastewater streams through industrial processes, agricultural runoff, and domestic wastewater [4,5]. One of the primary environmental issues is the contamination of water by heavy metals such as Cd^{2+} and Pb^{2+} [6–8]. This is because these metals are found to be toxic even at very low concentrations [6–8]. On the other hand, these trace metals are non-essential and therefore pose significant health risks to humans, even at trace levels, as Cd concentrations exceeding $0.6 \mu\text{g/L}$ may adversely affect the nervous system, whilst Pb concentrations above $5 \mu\text{g/L}$ can cause high blood pressure, kidney disease, and memory issues [9,10]. Thus, according to the World Health Organization

(WHO) guidelines, the maximum permissible concentration of Cd in drinking water is 3 µg/L, whilst the maximum allowed for lead is 10 µg/L, with the aim of 0 µg/L, which is recognized as safe in drinking water [10]. As a result, numerous researchers have developed various methods to remove these harmful trace metals from the environment [11,12]. These techniques include adsorption [13,14], filtration [15–17], and coagulation [18,19]. Adsorption is the most popular technology of the aforementioned techniques since it is less expensive, adaptable, easy to use, and efficient at eliminating trace or heavy metals [20,21]. In addition to environmental remediation, it has been used to remove trace metals and other contaminants in a range of industries, including food, paper, cosmetics, textiles, and pharmaceuticals [22]. The utilization of various adsorbents, including clays, metal–organic frameworks (MOFs), silica gel, activated carbon, zeolites, activated alumina, and polymer adsorbents, is another appealing aspect of the adsorption approach [23–25].

Adsorption equilibrium data play a central role in understanding adsorption mechanisms, as they provide insight into how the adsorbent interacts with metal ions in solution at equilibrium conditions [26,27]. The equilibrium stage essentially governs the efficiency of the process, since it reflects the balance between the adsorbent surface and the adsorbate species [28,29]. To enhance this interaction, different adsorbents have been designed with the aim of selectively binding target metal ions, guided by the surface chemistry between the two [30–33]. Modifying properties such as surface area, availability of active sites, electronic conductivity, and pore structure has proven particularly effective in tailoring adsorbents for improved specificity toward trace metals [34–36]. Many adsorbents have been specifically developed to remove Cd^{2+} and Pb^{2+} , such as metal–organic frameworks [37,38], layered double hydroxides (LDHs) [22,39], and biochar [23–25]. Layered double hydroxides (LDHs) have been widely explored in wastewater treatment due to their high surface reactivity, tunable composition, and abundance of surface hydroxyl groups, which enable effective interactions with various aqueous contaminants [26]. However, the difficult separation of conventional LDH powders, especially nanopowders, and secondary pollution of their residues in the wastewater after adsorption have been shown to limit their advanced application [27]. Therefore, the development of an LDH film with an overall structure and the advantage of easy separation after adsorption should be envisaged as an effective way to overcome the limitations of conventional materials. Given the foregoing, NiFe-LDH has exceptional adsorption capabilities; nonetheless, its application in environmental remediation procedures is constrained by metal leaching and poor recyclability [28]. Therefore, NiFe-LDH embedding into self-floating PES microbeads is a promising method that provides diminished metal leaching, good recyclability, and fantastic separation. However, the adsorption capability of NiFe-LDH/PES is reduced when NiFe-LDH is impregnated into PES microbeads because this results in a decrease in surface area and an increase in agglomeration. Therefore, the appropriate way to overcome such flaws is to incorporate it with activated carbon (AC) to form AC@NiFe-LDH/PES microbeads.

The objective of this study was to develop and evaluate AC@NiFe-LDH/PES beads as a new and recyclable adsorbent for removing Cd^{2+} and Pb^{2+} from aqueous solutions. The beads were designed to selectively adsorb and retain the target metals while allowing for the passage of clean water. The study found that the fabricated beads showed high efficiency in removing Cd^{2+} and Pb^{2+} from wastewater, with removal efficiencies exceeding 90%. These results indicate that AC@NiFe-layered double hydroxides/Polyether sulfone beads have enormous potential for the effective removal of trace metals. The nanocomposite beads' surface, morphological, and structural properties were characterized using several analytical techniques.

2. Materials and Methods

2.1. Reagents and Materials

Unless otherwise specified, all chemicals used in this study were of analytical reagent grade, and ultrapure water was used in all experiments. Urea (ACE, Johannesburg, South Africa), iron(III) nitrate nonahydrate $[\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}]$, nickel(II) nitrate hexahydrate $[\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}]$, and ethanol were obtained from Sigma-Aldrich (Johannesburg, South Africa). Ammonia solution (25%) was purchased from Merck (Johannesburg, South Africa). Polyvinylpyrrolidone (K10) was also sourced from Merck and used as received. Deionized water (DI) was prepared using a Millipore Milli-Q purification system (Merck, Darmstadt, Germany). Polyether sulfone (PES) with an average molecular weight of 62,000 g/mol was supplied by Solvay Advanced Polymers (Brussels, Belgium). The single-element standards of 1000 mg L⁻¹ of Cd and Pb purchased from Sigma-Aldrich (Johannesburg, South Africa) were used to freshly prepare a synthetic sample of multi-elemental solutions at a desired concentration daily. All the samples were filtered using the 0.22 µm pore size membrane filters purchased from Sigma-Aldrich (Johannesburg, South Africa), prior to analysis.

2.2. Synthesis of the Materials

2.2.1. Hydrothermal Synthesis of AC@NiFe-LDH Composites

Based on a previously published technique, a modified hydrothermal approach was used to synthesize AC@NiFe-LDH [29]. A standard approach involved dissolving 1.33 g of $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ and 3.90 g of $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ in 80 mL of deionized water and stirring for 15 min. The nominal molar ratio of Ni²⁺ to Fe³⁺ in the precursor solution was 3:1, corresponding to the targeted LDH composition. Separately, 0.11 g of activated carbon was dispersed in 10 mL of deionized water, and then the mixture was sonicated for 5 min to create an activated carbon suspension. The suspension was then added to the metal salt solution and stirred for 30 min. Next, 1.94 g of urea was added, and the solution was stirred for 30 min before being transferred to a Teflon-lined stainless-steel autoclave. The autoclave was kept at 150 °C for 21 h. After cooling to ambient temperature, the product was centrifuged, thoroughly cleaned with deionized water, and dried overnight in an oven at 100 °C. The dried material was then ground into a fine powder for future use.

2.2.2. Fabrication of AC@NiFe-LDH/PES Nanocomposite Beads

To exfoliate the LDH, AC@NiFe-LDH powder was ultrasonically dispersed in N-Methyl-2-pyrrolidone (NMP) solutions with polyvinylpyrrolidone (PVP) (5 wt.% of LDHs' mass) as a stabilizer. Then, Polyether sulfone (PES) was added, dissolved under stirring, and degassed. The prepared solution was first left to stand at room temperature for 24 h to eliminate air bubbles. It was then transferred into a syringe and carefully dropped, drop by drop, into a coagulation bath containing deionized water, where bead formation occurred. The beads were allowed to remain in the bath for about 1 min, then soaked in fresh deionized water for 24 h to remove residual solvents. Finally, the beads were dried at room temperature. The resultant beads were labeled as B0 to B3, to represent the increase in AC@NiFe LDH loading in the dope solutions (Table 1).

Table 1. AC@NiFe-LDH/PES bead compositions.

Bead ID	PES wt. %	NMP wt. %	PVP wt. %	AC@NiFe-LDHwt. %
B0	16	82	2	0
B1	16	81.8	2	0.2
B2	16	81.6	2	0.4
B3	16	81.4	2	0.6

2.3. Instrumentation

The prepared beads were characterized using a variety of analytical methods. Scanning electron microscopy (SEM) was used to investigate element composition, cross-sectional structure, and surface morphology using an Oxford Instruments EDS detector (JEOL JSM-IT300; Tokyo, Japan). For cross-sectional imaging, the beads were freeze-fractured in liquid nitrogen. Before SEM examination, samples were mounted on stubs and gold-coated using a SCD 005 Cool Sputter Coater (Bal-Tec, Amstetten, Germany). The presence and distribution of AC@NiFe LDH nanoparticles (NPs) were investigated further using transmission electron microscopy. The functional groups and bonding properties of the beads were examined using an ATR-FTIR spectrometer (Perkin Elmer, Rodgau, Germany). All metals in the water samples were quantified using an Inductively Coupled Plasma Mass Spectrometer (ICP-MS) (Perkin Elmer Nexion 350 D). The instrument operating conditions used were those recommended by the manufacturer. Cd-114 and Cd-112; Pb-204, Pb-206, Pb-207, and Pb-208 were all measured in calculated specific isotopic ratios.

2.4. Adsorption Procedure

To gain insight into the adsorption mechanism of Cd^{2+} and Pb^{2+} , equilibrium isotherm models were employed. For this experiment, five AC@NiFe-LDH/PES beads were used for each run. Due to minor variations in bead size, the total mass was not fixed but was approximately in the range of 200–250 mg, with initial concentrations ranging from 2 to $15 \mu\text{g L}^{-1}$, and allowed to equilibrate for 30 min. The experimental data obtained from ICP-MS analysis were evaluated using four widely applied isotherm models: Freundlich, Langmuir, Temkin, and Dubinin–Radushkevich (D–R).

Finally, kinetic and isotherm investigations were carried out under optimal conditions for each metal to assess the adsorption reaction properties. Kinetic investigations were conducted with exposure times ranging from 5 to 180 min, and adsorption isotherms were investigated at initial metal concentrations of 50–200 $\mu\text{g L}^{-1}$. Table S1 shows equations of these models, and they are widely reported in the literature [30–32]. All adsorption experiments were conducted in triplicate ($n = 3$) to ensure reproducibility. The reported values represent the mean of three independent measurements, and variability is expressed as standard deviation where applicable.

2.5. Multivariate Optimization

The adsorption of Cd^{2+} and Pb^{2+} was performed by adding an adequate mass of beads into sample bottles containing 50 mL of $500 \mu\text{g L}^{-1}$ Cd^{2+} and Pb^{2+} solution (pH 3–9-adjusted with 0.01 mol L^{-1} HCl or NaOH). Ultrasonication was used to allow adsorption for 5–30 min. The ultrasonication parameters included a frequency of 20 kHz and a power of 100 W. The beads were separated from the aqueous solutions. The resultant solution was filtered using the $0.22 \mu\text{m}$ pore size membrane filters and analyzed by ICP-MS. The adsorption process was optimized by a 2-level 1/2 fractional factorial design. Sample pH and contact time (CT) and sample volume (V) were the most significant experimental variables; their low (–), middle (0), and high (+) levels are listed in Table 2. Moreover, these recoveries were imported into the Minitab 2018 statistics program to study the response of each parameter. The response of each parameter was illustrated by a Pareto chart, and these parameters indicated the critical factors.

Table 2. Experimental levels and ranges of independent variables.

Parameters	Lower Level (–)	Central Point (0)	Higher Level (+)
pH	3	6	9
Sample volume (mL)	5	10	15
Contact time (min)	5	17.5	30

2.6. Collection and Treatment of Real Environmental Water Samples

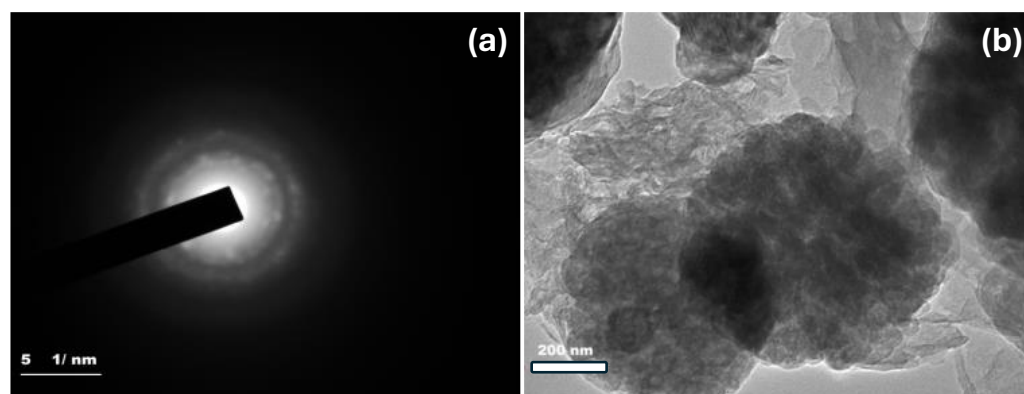
Water samples were collected from a variety of sources, including wastewater effluents, a wastewater treatment plant (WWTP), and tap water from a University of South Africa laboratory in Johannesburg. All samples were filtered over a 0.22 μm PVDF membrane and adjusted to a pH of 8 with 0.01 mol/L HCl or NaOH solutions. The optimal adsorption method was then used to remove the target metals from these samples. Each experiment was repeated three times to guarantee reliability and reproducibility.

3. Results and Discussion

3.1. Characterization of the NiFe-LDH Nanocomposite

3.1.1. High-Resolution Transmission Electron Microscopy (HR-TEM)

Figure 1 illustrates an HR-TEM image of the synthesized beads. The objective was to understand the structural and morphological characteristics of the NiFe-LDH material when incorporated into a PES matrix, particularly its structural stability. HR-TEM imaging (Figure 1b) revealed that the NiFe-LDH nanosheets, characterized by their layered structure, were observed to be uniformly distributed across the AC surface. AC nanoparticles, appearing as darker regions in the HR-TEM images, were observed to provide a conductive support that facilitates the dispersion of NiFe-LDH nanosheets [33]. The activated carbon appears as a porous structure interspersed between the LDH layers, which likely helps to increase the overall surface area of the nanocomposites and the size of LDH particles. This suggests that the synthesis conditions effectively preserved the layered architecture of the NiFe-LDH during the deposition process. To gain further information on the crystallinity of the NiFe-LDH in the nanocomposites, we employed selected area electron diffraction (SAED) (Figure 1a). The diffraction pattern revealed sharp and diffuse diffraction rings, which correspond to the crystal planes of NiFe-LDH [29,34]. The rings observed in the SAED pattern are associated with the [003], [006], [009], and [015] planes, typical of the hexagonal crystal structure of NiFe-LDH [29,34,35]. These planes confirm the highly crystalline nature of the LDH material despite being incorporated into activated carbon and PES.

**Figure 1.** SAED pattern of beads (a). HR-TEM image of beads (b).

3.1.2. Scanning Electron Microscopy (SEM)

As shown in the SEM images, there are distinct morphological changes for pristine PES beads and AC@NiFe-LDH/PES composites (Figure 2a). The clean PES beads have a comparatively smooth and barren surface, suggesting their compact polymer structure, whereas the AC@NiFe-LDH/PES beads show rougher, non-uniform surface morphology with apparent porous texture and particulate matter entrenchment in the AC@NiFe-LDH loading (Figure 2b). Cross-section micrographs reveal that the introduction of AC and LDH not only alters the surface roughness but also promotes internal porosity, which is beneficial for the diffusion and adsorption of metal ions (Figure 2c,e). The presence of NiFe-LDH exhibits uniform distribution, indicating successful embedding in the PES matrix which would be helpful in improving adsorption performance in the subsequent experiments (Figure 2d,f). The PES matrix plays a crucial supporting role in the adsorption system by immobilizing AC@NiFe-LDH within mechanically stable and recoverable beads. Although PES does not directly participate in chemical adsorption, its phase-inversion-induced porous structure enhances the accessibility of active adsorption sites by promoting efficient mass transfer of metal ions into the bead interior. The preserved morphology of the NiFe-LDH within the beads indicates that active sites remain available for interaction with metal ions, supporting adsorption through electrostatic interactions and ion-exchange processes rather than physical entrapment alone.

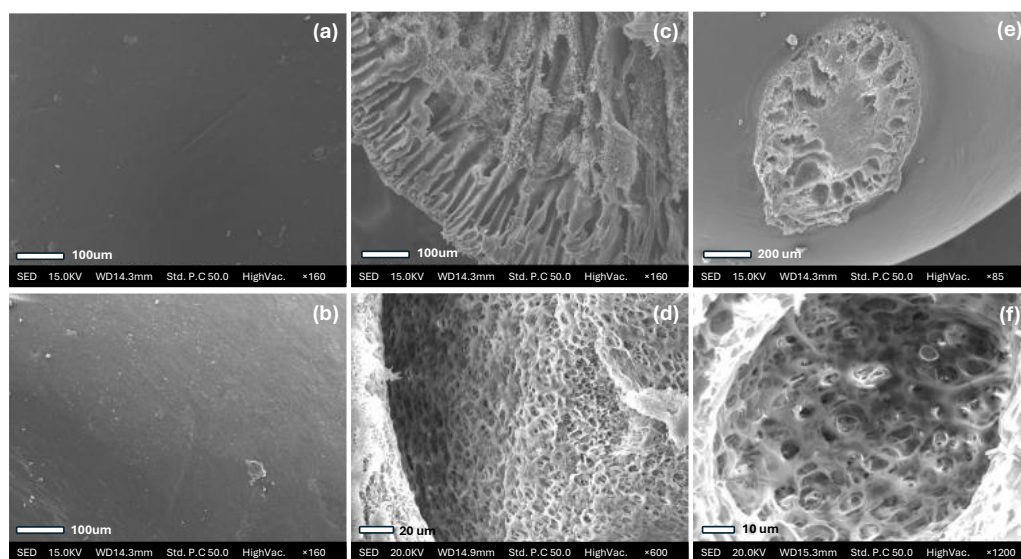


Figure 2. SEM images showing the top surface morphology (PES beads (a), AC@NiFe-LDH/PES beads (b)), the inside of the beads (d,f), and a cross-section of a bead (c,e).

3.2. Optimization Strategy

3.2.1. Selection of Suitable Bead Composition

The optimization study initially concentrated on various bead compositions (B0–B3) containing 0–0.6 wt.% AC@NiFe-LDH, as illustrated in Figure 3. The recovery efficiencies of Cd^{2+} and Pb^{2+} improved with an increase in the LDH loading. This trend can be attributed to the greater availability of active adsorption sites and larger pore volumes in beads with higher loadings. Among these, B3 (with a loading of 0.6 wt.%) achieved the highest recovery efficiency and was therefore selected for further studies. The improved adsorption performance at higher AC@NiFe-LDH loadings is interpreted as a synergistic contribution of activated carbon and NiFe-LDH, where activated carbon enhances surface area and porosity, while NiFe-LDH provides abundant metal hydroxide active sites. This interpretation is based on adsorption efficiency trends rather than direct synergy quantifica-

tion. This allows for the target compounds to be readily eluted using the desorption solvent. The incorporation of activated carbon plays a critical role in enhancing the adsorption performance of the composite beads. Activated carbon contributes to a high surface area and porous channels, which facilitate rapid diffusion of metal ions toward active sites.

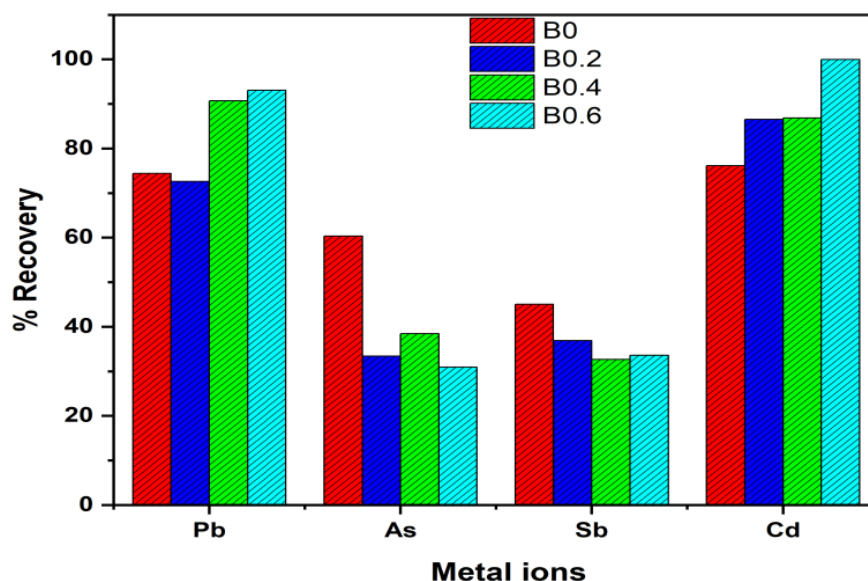


Figure 3. Selection of suitable bead compositions: B0: PES beads; B1: 0.2; B2: 0.4; and B3: 0.6. Experimental conditions: pH: 7.5; contact time: 30 min; initial concentration: $100 \mu\text{g L}^{-1}$.

3.2.2. Screening of Parameters

Following the preliminary studies, several parameters that potentially influence adsorption responses, such as solution pH, sample volume (mL), and contact time (min), were first screened using a 2-level 1/2 fractional factorial design. Figure 4 shows the design matrix and analytical response for Cd^{2+} and Pb^{2+} analytes in terms of extraction efficiency. The results of the design of experiment (DOE) yielded Pareto charts based on Analysis of Variance (ANOVA). The significant parameters are represented by the blue bars shown in Figure 4 for each factor. The red line indicates the 95% confidence level. All the assessed parameters, with the exception of pH, showed significance at the 95% confidence level. While pH exceeded the threshold, neither contact time nor sample volume did.

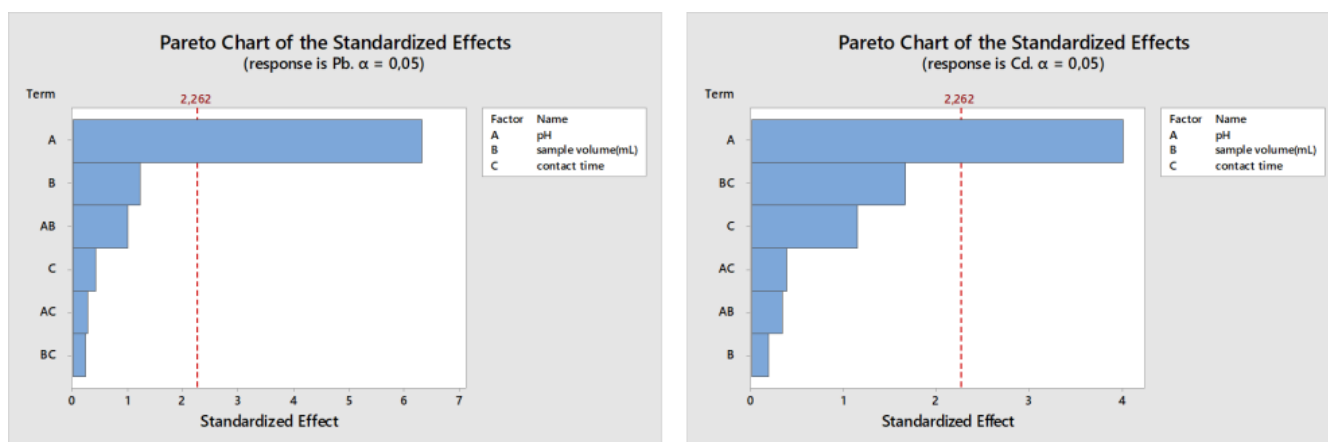


Figure 4. Two-level 1/2 fractional factorial design Pareto chart ($n = 3$) of Pb^{2+} and Cd^{2+} removal for the optimization of contact time, pH, and sample volume at the 95% confidence level.

3.2.3. Response Surface Methodology (RSM) Based on Central Composite Design

The RSM analysis in Figure 5 gave further insight into the interaction effects between process variables. (A) The Pareto chart of the results of ANOVA showed that pH significantly impacted the removal efficiencies of Cd^{2+} and Pb^{2+} beyond the 95% confidence limit. This strong effect can be attributed to the pH-dependent variations in the metal ion speciation and surface charge of AC@NiFe-LDH/PES beads, which exert a direct influence on electrostatic attraction and binding affinity. By contrast, effects of contact time, as well as their interaction, were found to be statistically insignificant within the range examined, suggesting they have lower sensitivity upon pH adjustment. The observations indicate that pH control is the governing operational parameter that maximizes the removal efficiency, consistent with what appears in the literature.

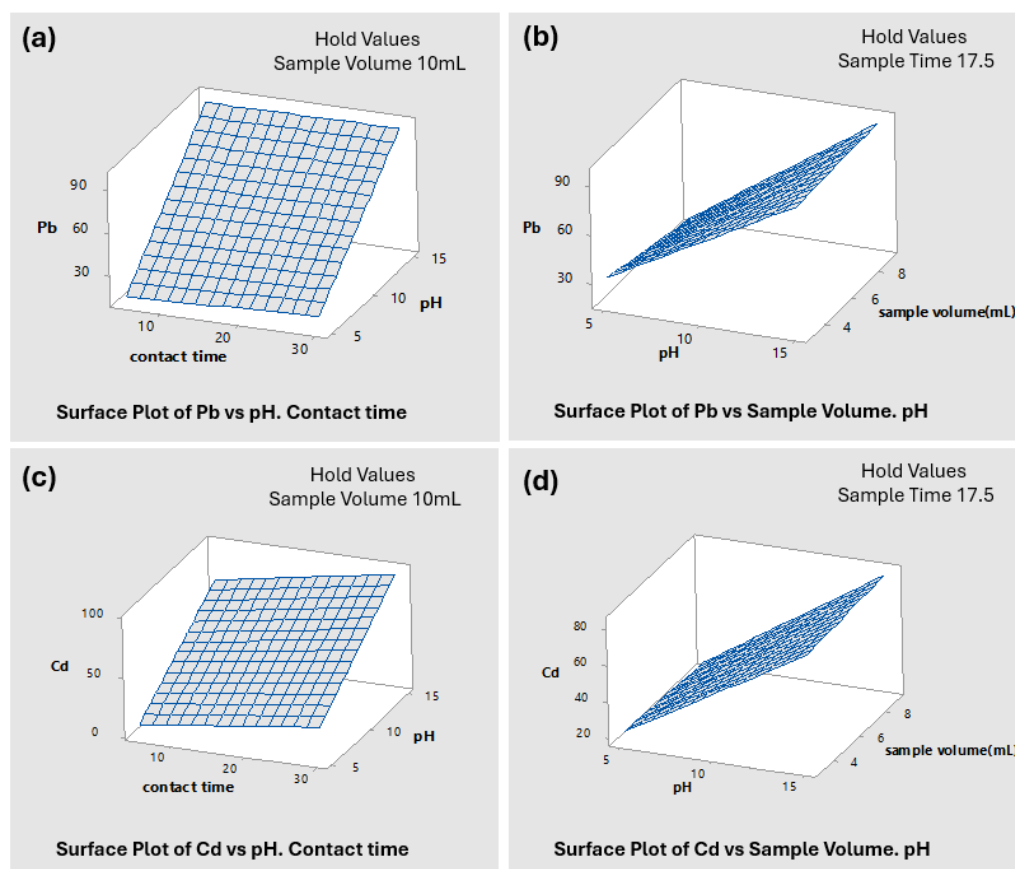


Figure 5. Response surface methodology (RSM) based on the central composite design. The response (y-axis) represents removal efficiency (%). Surface plots represent: (a) pH vs contact time for Pb; (b) Sample volume vs pH for Pb, (c) pH vs contact time for Cd, (d) Sample volume vs pH for Cd.

3.3. Adsorption Isotherms

The equilibrium adsorption data were modeled with Langmuir, Freundlich, Temkin, and Dubinin–Radushkevich (D–R) isotherm models to describe the interaction between the AC@NiFe-LDH/PES beads and the target metal ions (Cd^{2+} and Pb^{2+}). The Langmuir model was found to provide the best fitted isotherm ($R^2 = 0.991$), and the maximum monolayer adsorption capacity (Q_{max}) was 3.02 mg g^{-1} , suggesting that the adsorption process mainly occurred on the monolayer and coverage of the adsorbate on the homogeneous surface containing active sites occurred in a similar way [36]. This indicates that adsorption of Cd^{2+} (Figure 6a) occurs mainly via specific surface metal ion interactions and not as a result of multilayer deposition, which is in agreement with the large n value from the

Freundlich model ($n = 7.380$) that indicates a favorable adsorption process and that the adsorbent has a strong binding affinity [40].

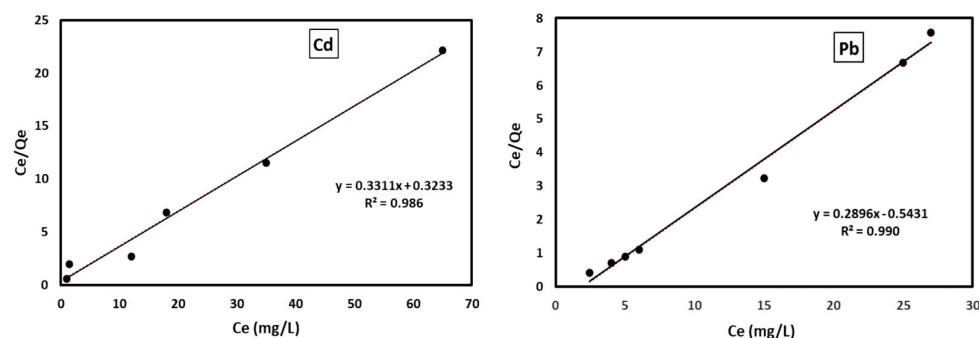


Figure 6. Langmuir isotherm model for Cd^{2+} and Pb^{2+} .

On the other hand, Pb^{2+} adsorption (Figure 6b) was best described by the Langmuir model ($R^2 = 0.987$), compared to the Freundlich ($R^2 = 0.9167$) and Temkin ($R^2 = 0.9389$) models, indicating predominant monolayer adsorption on relatively homogeneous active sites of the AC@NiFe-LDH/PES beads. Although the composite material inherently contains chemically diverse components, the adsorption behavior within the studied concentration range is dominated by uniform binding sites [41]. As summarized in Table 3, the greater $Q_{\max}$ of the Pb^{2+} (3.45 mg g^{-1}) as compared to Cd^{2+} may be attributed to the larger ionic radius of the Pb^{2+} ($1.19 \text{ \AA}$) as compared to the Cd^{2+} ($0.95 \text{ \AA}$), as well as the oxygen-containing functional groups present on the surface of the ACs and hydroxylated LDH surface [42]. The RL values were found to be between 0 and 1 in Cd^{2+} and Pb^{2+} for adsorption, which suggested the process was favorable under the range of studied concentrations. The Temkin constants also demonstrated a lower heat of adsorption of Pb^{2+} ($BT = 2.34 \times 10^3 \text{ J mol}^{-1}$) than Cd^{2+} ($BT = 3.48 \times 10^3 \text{ J mol}^{-1}$), evidencing weaker interactions for Pb^{2+} , possibly explaining the better fitting of this metal to heterogeneous surfaces. In the synthesized nickel–iron-layered double hydroxides, the mean adsorption energies (E) estimated by the D–R model were $1.118 \text{ kJ mol}^{-1}$ for Cd^{2+} and $1.290 \text{ kJ mol}^{-1}$ for Pb^{2+} , also falling within the limit of physisorption ($<8 \text{ kJ mol}^{-1}$), which suggests electrostatic forces prevailing over chemisorption. In general, isotherm results reveal that Cd^{2+} removal by AC@NiFe-LDH/PES is well represented by monolayer adsorption on the homogeneous AC@NiFe-LDH/PES active sites, while Pb^{2+} sorption is controlled by multi-site adsorption, which may be attributed to the structural and chemical heterogeneity of activated carbon and LDH nanocomposites.

Table 3. Adsorption isotherm constant values for the Freundlich and Langmuir isotherms.

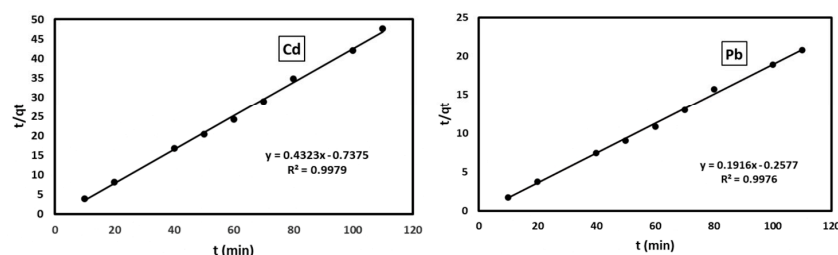
Isotherms	Parameters	Cd	Pb
Langmuir	$Q_{\max} (\text{mg g}^{-1})$	3.02	3.45
	$K_L (\text{Lmg}^{-1})$	1.024	0.533
	R_L	0.010	0.018
	R^2	0.991	0.987
Freundlich	K_f	1.971	7.441
	n	7.380	4.883
	R^2	0.391	0.9167
Temkin	$K_T (\text{L g}^{-1})$	2.19×10^{-4}	6.8×10^{-4}
	$B_T (\text{J/mol})$	3.48×10^3	2.34×10
	R^2	0.3404	0.9389

Table 3. Cont.

Isotherms	Parameters	Cd	Pb
Dubinin–Radushkevich	B ($\text{mol}^2\text{J}^{-2}$)	-2×10^{-7}	-3×10^{-7}
	R^2	0.0492	0.744
	q_m (mg g^{-1})	0.657	4.778
	E (KJmol^{-1})	1.581	1.290

3.4. Adsorption Kinetics

The adsorption kinetics of Cd^{2+} and Pb^{2+} on AC@NiFe-LDH/PES beads were modeled by pseudo-first-order and pseudo-second-order equations (Figure 7a,b). According to Table 4, the pseudo-second-order model fitted well for both metals ($R^2 = 0.997$ for Cd^{2+} and 0.998 for Pb^{2+}), and the Q_e values calculated were very close to the experimental values (2.310 mg g^{-1} for Cd^{2+} vs. 2.400 mg g^{-1} experimental value; 5.219 mg g^{-1} for Pb^{2+} vs. 5.400 mg g^{-1} experimental value). This result implies that valence forces are dominant for chemisorption with exchange or sharing of electrons between the metal ions and adsorbent [43]. Although the pseudo-second-order kinetic model suggests that surface complexation plays an important role in the adsorption process, the low mean adsorption energies obtained from the D–R isotherm indicate that electrostatic interactions (physisorption) also contribute significantly. Therefore, the adsorption of Cd^{2+} and Pb^{2+} likely involves a combination of electrostatic attraction and surface complexation mechanisms. The R^2 value of the pseudo-first-order model was much lower (0.050 for Cd^{2+} and 0.054 for Pb^{2+}), suggesting it failed to properly describe the adsorption process. The higher value of the rate constant (K_2) ($0.254 \text{ g mg}^{-1} \text{ min}^{-1}$) for Cd^{2+} than for Pb^{2+} ($0.143 \text{ g mg}^{-1} \text{ min}^{-1}$) indicates that the composite beads have the ability to capture Cd^{2+} ions more quickly, at least in the initial stage, which might be attributed to the factor of the smaller size of the hydrated ionic radius of Cd^{2+} than that of Pb^{2+} and the faster diffusion of Cd^{2+} ions through bead pores [44,45]. The initial kinetics (Tables 3 and 4) at the same [Ce] levels are consistent with those obtained for other LDH-based and AC-supported adsorbents, where the heavy metal uptake occurs via a fast adsorption step and a slow adsorption phase due to the rise in availability of active sites [46]. The strong agreement with the pseudo-second-order model indicates that the adsorption of Cd^{2+} and Pb^{2+} is governed primarily by surface reaction mechanisms, while intraparticle diffusion may contribute but is not the sole rate-limiting step [47].

Figure 7. Pseudo-second-order model for Cd^{2+} and Pb^{2+} .Table 4. Pseudo-first-order and pseudo-second-order kinetics for the sorption of Cd^{2+} and Pb^{2+} .

Kinetics	Parameters	Cd^{2+}	Pb^{2+}
Pseudo-1st order	Q_{exp} (mg g^{-1})	2.400	5.400
	K_1 (min^{-1})	0.021	0.008
	Q_e (mg g^{-1})	1.817	1.233
	R^2	0.050	0.054

Table 4. *Cont.*

Kinetics	Parameters	Cd ²⁺	Pb ²⁺
Pseudo-2nd order	K ₂	0.254	0.143
	Q _e (mg g ^{−1})	2.310	5.219
	R ²	0.997	0.998

3.5. Application to Real Environmental Samples

The adsorptive removal process developed in this study was further evaluated using real environmental water samples, including effluent, influent, and Unisa tap water, to assess the performance of the AC@NiFe-LDH/PES nanocomposites in the presence of possible interfering species. The results are summarized in Table 5. Since the certified reference materials (CRM) for Cd²⁺ and Pb²⁺ were not available, the water samples were spiked with a multielement standard with a concentration level of 5 µg L^{−1}. A spike concentration of 5 µg L^{−1} was selected to represent elevated contamination levels typical of industrial wastewater, which often exceed regulatory discharge limits and therefore provide a stringent test of adsorbent performance. After adsorption, the AC@NiFe-LDH/PES beads were readily separated from the aqueous solution by simple physical removal (decantation), owing to their macroscopic bead structure, without the need for filtration or centrifugation. The obtained results demonstrated that the nanocomposites maintained high removal efficiencies for both metals even in the presence of co-existing ions and other matrix components. This indicates that the complex water matrices had no significant adverse impact on the adsorption capacity of the AC@NiFe-LDH/PES nanocomposites for Cd²⁺ and Pb²⁺.

Table 5. Adsorptive removal of Cd²⁺ and Pb²⁺ in real water samples (mean +/− SD).

Sample	Added (µg L ^{−1})	Cd ²⁺		Pb ²⁺	
		Found	%R	Found	%R
Unisa tap water	0	ND	-	ND	-
	5	5.08 ± 0.38	95.5	5.02 ± 0.55	92.7
Influent	0	ND	-	ND	-
	5	5.01 ± 0.39	93.4	5.02	96.6
Effluent	0	ND	-	0.003	-
	5	5.34 ± 0.45	94.6	5.29 ± 0.33	93.0

3.6. Proposed Mechanism

Figure 8 illustrates a proposed mechanism for the adsorption of Pb²⁺ and Cd²⁺ onto activated carbon/NiFe-LDH/Polyether sulfone composite beads. Metal ions are captured through several complementary pathways, including substitution within the NiFe-LDH layers, ion exchange with hydroxyl groups, surface complexation, and electrostatic attraction provided by the activated carbon. The Polyether sulfone matrix acts as a supporting framework, ensuring even distribution of LDH and carbon while maintaining the stability and efficiency of the beads. Based on the characterization and adsorption studies, the adsorption of Cd²⁺ and Pb²⁺ onto AC@NiFe-LDH/PES beads occurs through a combination of electrostatic attraction, ion exchange, and surface interactions. HR-TEM and SAED analyses confirm that the layered structure and crystallinity of NiFe-LDH are preserved within the composite, indicating that interlayer regions remain accessible for metal ion interaction. These interlayers facilitate ion exchange between metal ions in solution and hydroxyl groups associated with the LDH structure.

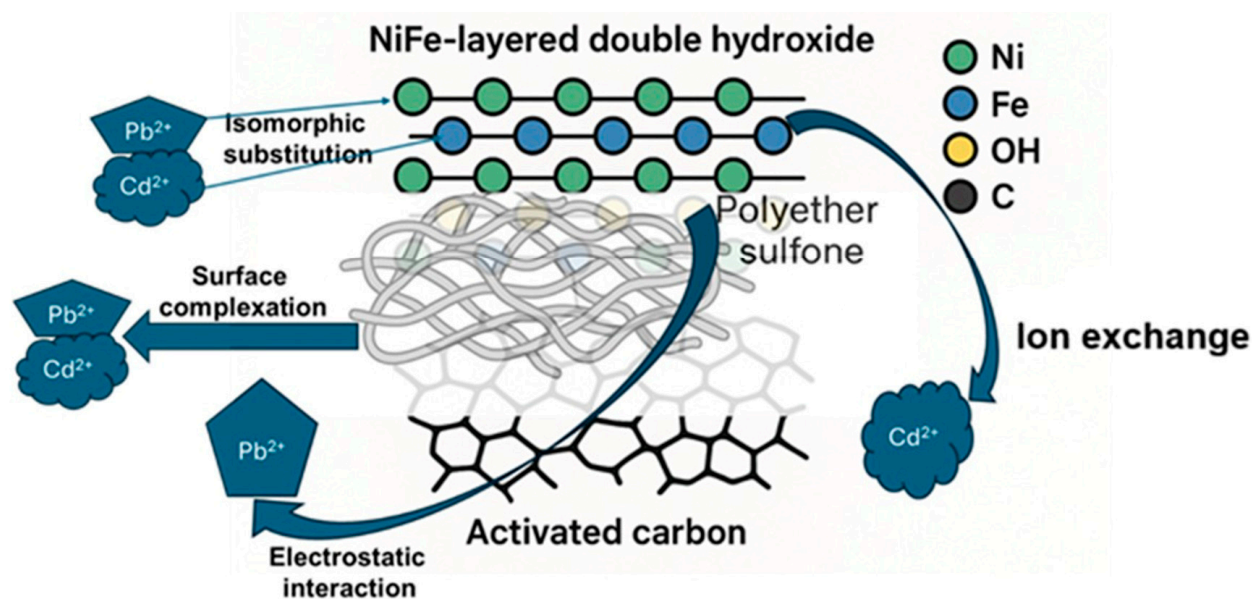


Figure 8. Proposed mechanism for the adsorption of Pb^{2+} and Cd^{2+} onto AC@NiFe-LDH/PES nanocomposite beads.

3.7. Reusability Study

Regeneration experiments were conducted over five consecutive adsorption–desorption cycles. Each adsorption step was performed at optimum conditions. Desorption was achieved using $0.1 \text{ mol L}^{-1} \text{ HNO}_3$ with 20 min contact time. After each cycle, the beads were thoroughly rinsed with deionized water and dried prior to reuse. Figure 9 shows the removal efficiency decreased by less than 8% from the first to the fifth cycle for both Cd^{2+} and Pb^{2+} , confirming good reusability. All experiments were conducted in triplicate ($n = 3$), and the relative standard deviation remained below 5%.

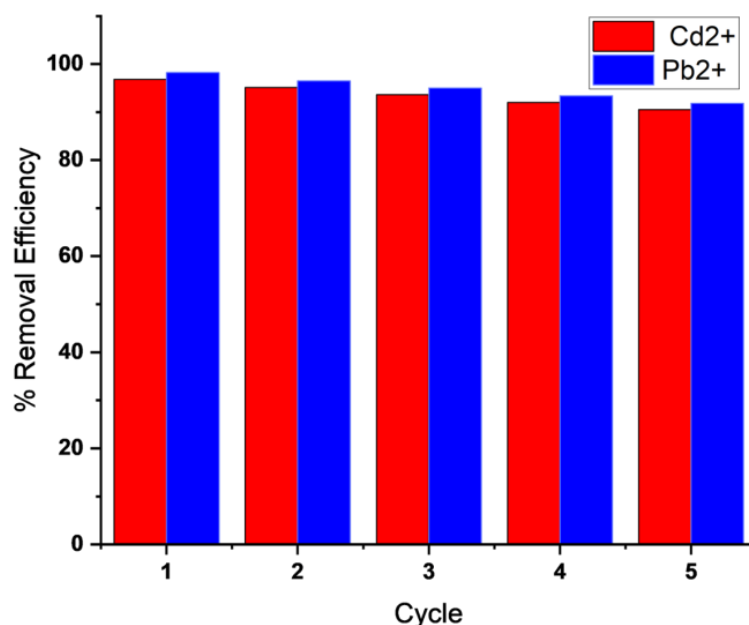


Figure 9. Reusability performance of AC@NiFe-LDH/PES nanocomposite beads for Pb^{2+} and Cd^{2+} removal over 5 cycles.

3.8. Comparison Study

Table 6 shows the comparison of the maximum adsorption capacities (Q_{max}) of AC@NiFe-LDH/PES beads with other reported adsorbents for Cd^{2+} and Pb^{2+} removal. The

results indicate that while some materials exhibit higher capacities, the nanocomposites developed in this study demonstrate competitive performance, particularly when considering their structural stability, ease of recovery, and reusability. Notably, their Pb^{2+} capacity is within the range reported for advanced carbon LDH and biochar nanocomposites [48], and their Cd^{2+} capacity compares favorably with other LDH-based adsorbents, confirming their suitability for practical wastewater treatment applications.

Table 6. Comparison of maximum adsorption capacities ($Q_{\max}$) of AC@NiFe-LDH/PES beads with various reported adsorbents for Cd^{2+} and Pb^{2+} removal from aqueous solutions.

Analyte	Adsorbent	$Q_{\max}$ (mg/g)	Reference
Cd^{2+}	$\text{ZrO}_2\text{-Fe}_3\text{O}$	114	[49]
Cd^{2+}	Zeolite NaX	~38.6	[50]
Cd^{2+}	KB/LDH biochar	~25.6	[51]
Cd^{2+}	CuMgAl-LDH/MMt	1.75	[52]
Pb^{2+}	OX-MWCNT	5.73	[48]
Pb^{2+}	Humic acids from sludge and lignite	27.6–147	[53]
Cd^{2+}	AC@NiFe-LDH/PES	3.02	This work
Pb^{2+}	AC@NiFe-LDH/PES	3.45	This work

4. Conclusions

In conclusion, the AC@NiFe-LDH/PES beads developed in this study represent an effective and sustainable adsorbent for the removal of cadmium (Cd^{2+}) and lead (Pb^{2+}) from aqueous solutions. The nanocomposites exhibited high adsorption capacities, with the process fitting well to the Langmuir isotherm model and following pseudo-second-order kinetics, confirming monolayer chemisorption as the dominant mechanism. The beads demonstrated excellent structural stability, and they can be reused over five adsorption–desorption cycles, retaining their efficiency with minimal loss in performance. Importantly, when applied to real environmental water samples (effluent, influent, and Unisa tap water), the AC@NiFe-LDH/PES nanocomposites maintained high removal efficiencies for both Cd^{2+} and Pb^{2+} , even in the presence of co-existing ions and complex matrix components. This highlights their strong selectivity and robustness under practical conditions. Overall, the superior adsorption performance, recyclability, and environmental compatibility of the AC@NiFe-LDH/PES beads underscore their potential as a promising solution for the remediation of heavy metal-contaminated waters, contributing to sustainable and eco-friendly wastewater treatment technologies. The present study focused on the adsorption performance and structural integrity of AC@NiFe-LDH/PES beads. Possible leaching of Ni^{2+} and Fe^{3+} ions from the LDH phase was not investigated and should be addressed in future studies to fully assess environmental safety.

Supplementary Materials: The following supporting information can be downloaded at <https://www.mdpi.com/article/10.3390/jcs10020068/s1>: Table S1: The overview of the adsorption isotherm equations of Langmuir, Freundlich, Temkin, and Dubinin–Radushkevich (D-R) used to describe the equilibrium adsorption behaviour.

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