

Article

Synthesis and Characterization of a Novel $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ Ternary Composite for Efficient Pb (II) and Cd (II) Ion Adsorption from Aqueous Solutions

Mahmoud M. Youssif ^{1,2,*} , Mateusz M. Marzec ³  and Marek Wojnicki ^{1,*} ¹ Faculty of Non-Ferrous-Metals, AGH University of Krakow, al. Mickiewicza 30, 30-059 Krakow, Poland² Department of Chemistry, Faculty of Science, Tanta University, Tanta 31527, Egypt³ Academic Centre for Materials and Nanotechnology, AGH University of Krakow, al. Mickiewicza 30, 30-059 Krakow, Poland; marzecm@agh.edu.pl

* Correspondence: youssif@agh.edu.pl or mahmoud.youssif@science.tanta.edu.eg (M.M.Y.); marekw@agh.edu.pl (M.W.)

Abstract

Lead (Pb^{2+}) and cadmium (Cd^{2+}) are among the most hazardous heavy metal pollutants in wastewater owing to their high toxicity, environmental persistence, and detrimental impacts on human health and aquatic ecosystems. In this study, a novel ternary magnetic composite, $\text{SnFe}_2\text{O}_4/\text{activated carbon}/\text{polypyrrole}$ ($\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$), was effectively synthesized and tested as an effective adsorbent in the removal of Pb^{2+} and Cd^{2+} from aqueous water. The composite was prepared by depositing spinel SnFe_2O_4 nanoparticles on activated carbon, followed by in situ polymerization of polypyrrole to enhance surface functionality and adsorption affinity. The successful fabrication of the porous $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ hybrid composite was confirmed through FTIR, XRD, SEM-EDS, BET, XPS, and VSM characterization. The composite demonstrated a relatively high surface area ($352.3 \text{ m}^2/\text{g}$) and adequate magnetic responsiveness (12.33 emu/g), ensuring facile magnetic separation following wastewater treatment. Batch adsorption experiments showed great removal efficiency of 95.02 and 92.48% for Pb^{2+} and Cd^{2+} ions, respectively, at optimum conditions. The adsorption equilibrium data followed the Langmuir isotherm model with maximum adsorption capacities of 187.07 mg/g for Pb^{2+} and 96.45 mg/g for Cd^{2+} ions, which were attributed to monolayer adsorption on homogenous active sites. The kinetic and isothermal model indicated that the adsorption process was controlled by the combination of physical and chemical interactions. Thermodynamic parameters showed negative Gibbs free energy and enthalpy changes ($\Delta H^\circ = -49.74 \text{ kJ/mol}$ for Pb^{2+} and -38.82 kJ/mol for Cd^{2+} ions), confirming the spontaneous and exothermic nature of adsorption. Furthermore, the increasingly negative ΔG° values at lower temperatures indicated that the adsorption was thermodynamically more favorable under cooler conditions. According to the regeneration studies, the composite maintained a high removal efficiency after five consecutive cycles. In general, $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite has good potential as a stable, reusable, and high-performance adsorbent to treat heavy metal wastewater.



Academic Editor: Corby G. Anderson

Received: 29 May 2026

Revised: 17 June 2026

Accepted: 22 June 2026

Published: 25 June 2026

Copyright: © 2026 by the authors.

Licensee MDPI, Basel, Switzerland.

This article is an open access article distributed under the terms and conditions of the [Creative Commons Attribution \(CC BY\)](https://creativecommons.org/licenses/by/4.0/) license.

Keywords: tin ferrite; polypyrrole (PPy); magnetic composite; heavy metals; wastewater; adsorption

1. Introduction

Contamination of aquatic systems with heavy metals has become a major issue of concern in the world owing to persistence, bioaccumulation, and the extreme toxicity of

such wastes [1,2]. In contrast to organic contaminants, heavy metals are non-biodegradable and tend to accumulate in living organisms over time, causing ecological and health risks [3]. Lead (Pb) and cadmium (Cd) are particularly interesting among the toxic heavy metals due to their large-scale industrial application and the negative biological impact. Lead is a very dangerous element that is used in many industries, including mining, chlor-alkali, and the production of electronics [4]. Lead compounds may bioaccumulate in aquatic organisms even in trace levels and further biomagnify along the food chain, and this is extremely dangerous to human health [5]. The neurological disorders, renal dysfunction, and developmental defects have been linked to exposure to lead [6]. The other harmful heavy metal is cadmium, which finds a wide application in electroplating, alloy creation, batteries, and pigments [7,8]. Cadmium is a very dangerous heavy metal and has no known biological role; any exposure, even in low doses, can have severe impacts on health, such as kidney dysfunction, respiratory problems, heart disease, and even cancer [9,10]. According to the World Health Organization (WHO), the guideline value for lead (Pb) in drinking water is 0.01 mg/L, while for cadmium (Cd) it is 0.003 mg/L. Similarly, the Council of the European Union establishes parametric values of 0.01 mg/L for lead and 0.005 mg/L for cadmium. In contrast, the United States Environmental Protection Agency (US EPA) has set an action level of 0.015 mg/L for lead, with a maximum contaminant level goal (MCLG) of zero due to the absence of a safe exposure threshold. For cadmium, the US EPA has established a maximum contaminant level (MCL) and MCLG of 0.005 mg/L in drinking water [7,11]. Thus, the efficient elimination of Pb (II) and Cd (II) from polluted water is a key point of environmental and public health security. Various physicochemical methods like chemical precipitation, ion exchange, membrane filtration, electrochemical treatments, and photocatalysis have been devised for the removal of heavy metals. Nonetheless, these techniques are usually associated with disadvantages, including high operation, incomplete extraction, production of toxic sludge, and low selectivity in low concentrations [12–15]. Of all the existing techniques, adsorption has emerged as a promising technique due to low cost, simplicity in design, scalability, and extremely high efficiency in the removal of trace pollutants [16].

During the last several years, considerable attention has been devoted to the design of advanced adsorbents based on the combination of several functional materials. In contrast to many commonly studied ferrite nanoparticles, SnFe_2O_4 has emerged as a promising spinel ferrite owing to its unique electronic structure, high chemical stability, the presence of Sn-related surface defects and oxygen vacancies that provide additional active sites for metal-ion binding, and favorable magnetic properties that enable the easy recovery of spent adsorbents using an external magnetic field. These characteristics make SnFe_2O_4 particularly attractive for the adsorption of toxic heavy metals [17,18]. One of the most popular adsorbents is activated carbon (AC), due to its high specific surface area, developed porous structure, and adjustable surface chemistry that can be utilized to trap metal ions and organic pollutants [19,20]. However, the regeneration of AC and the comparatively low selectivity restrict its usage in contaminated wastewater of high concentration. Polypyrrole (PPy) is a conducting polymer that combines complementary functionality with respect to carrying heavy metals using electrostatic attractions, hydrogen bonds, and chelation due to its conjugated π -electrons, $-\text{NH}$, and $=\text{C}-\text{N}$ functional groups [21]. In addition, PPy is stable in the environment, simple to synthesize, and can be deposited as thin layers on porous supports to increase surface reactivity [22].

On these grounds, the current research presents a new SnFe_2O_4 /activated carbon/polypyrrole (SnFe_2O_4 /AC/PPy) ternary composite that can effectively remove Pb^{2+} and Cd^{2+} ions from polluted water. SnFe_2O_4 nanoparticles in this composite will offer good magnetic properties and active sites, AC will offer high surface area and porosity

to increase adsorption, and polypyrrole (PPy) provides abundant nitrogen-containing functional groups to increase the affinity of the metals and stability. The integration of these components is expected to increase adsorption capacity, selectivity, recycling, and separation feasibility in aqueous media relative to the individual components or binary composites. FTIR, XRD, SEM, BET, XPS, and VSM analyses were used in a systematic characterization of the structural, morphological, and magnetic properties of the composite. In addition to this, adsorption kinetics, thermodynamics, and regeneration performance were studied to determine its potential as a sustainable and efficient adsorbent in real-life wastewater treatment.

2. Experimental Procedure

2.1. Chemicals

In this study, analytical-grade reagents and chemicals were used, not purified in any way. The carbon support material of the activated type was purchased through Norit Nederland B.V. (Amersfoort, the Netherlands) and subsequently milled into a fine powder. Ferric chloride hexahydrate ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, $\geq 98\%$), ferrous chloride (FeCl_2 , 99.99%), anhydrous tin chloride (SnCl_2 , 99.99%), and sodium dodecyl sulfate (SDS) ($\text{CH}_3(\text{CH}_2)_{11}\text{OSO}_3\text{Na}$, 99%) were bought from Sigma-Aldrich. Pyrrole monomer ($\text{C}_4\text{H}_5\text{N}$, 98%) and the ammonium hydroxide solution (NH_4OH , 25 wt.%) were provided by Chempur (Piekary Śląskie, Poland). Sodium hydroxide in pellet form (NaOH , 99.99%) and hydrochloric acid (HCl , 37 wt.%) were obtained from Avantor Performance Materials (Gliwice, Poland). Moreover, Chemland Materials (Stargard, Poland) also supplied sulfuric acid (H_2SO_4 , 95 wt.%) and ethylene glycol ($\text{HOCH}_2\text{CH}_2\text{OH}$, 99.8%). The experimental work also involved high-performance liquid chromatography-grade ($\text{CH}_3\text{CH}_2\text{OH}$, 99.9%) ethanol as a solvent to guarantee the purity of the solvent. To prepare the standard solutions of heavy metal ions, lead chloride (PbCl_2 , 99.99%) and cadmium chloride (CdCl_2 , 99.99%) of analytical grade were purchased from Chempur (Piekary Śląskie, Poland) and then used to prepare stock solutions with a 1000 mg/L concentration.

2.2. Synthesizing $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ Composite

The ternary composite preparation is provided in Figure 1. First, tin ferrite (SnFe_2O_4) was prepared by a solvothermal method based on the procedure that was described in [23]. In particular, 0.314 g of SnCl_2 and 0.658 g of FeCl_2 were dissolved in 50 mL of ethylene glycol and mixed for 30 min to obtain a homogeneous solution. An amount of 4 mL of NH_4OH (1 M) and then NaOH solution (5 M) was slowly added to the homogeneous solution until the medium pH was approximately 12. The mixture was stirred for a further 30 min at 60 °C, and a Teflon-lined autoclave was used to maintain the mixture at 190 °C during the next 18 h. The black precipitate obtained was collected, washed numerous times with deionized water, and dried at 60 °C for 24 h. In order to prepare the binary $\text{SnFe}_2\text{O}_4/\text{AC}$ composite, the same solvothermal reaction was carried out in the presence of activated carbon (AC), which served as support and was also utilized as a high-surface-area matrix of the ferrite nanoparticles.

The ternary composite $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ was prepared by the oxidative polymerization of pyrrole in the presence of $\text{SnFe}_2\text{O}_4/\text{AC}$. 0.5 g of the prepared $\text{SnFe}_2\text{O}_4/\text{AC}$ composite was added to 100 mL of 0.001 M sodium dodecyl sulfate (SDS) solution, and this was dispersed by sonication over a period of 30 min to get a fine dispersion. During continuous mechanical stirring, the pyrrole monomer (0.7 mL) was added. The oxidizing agent, FeCl_3 (4.9 g dissolved in 100 mL of distilled water), was slowly added dropwise at (0–5 °C), while maintaining stirring for 8 h. The reaction process was done by phase separation as the top layer held the free polypyrrole (PPy), which was collected by decantation, and

the lower layer was denser composed of $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ coated with PPy, which was collected by magnet separation and washed thoroughly with a 1:1 ethanol–water mixture and dried under a vacuum at 60°C for 24 h to obtain a composite (Figure 1c) [24].

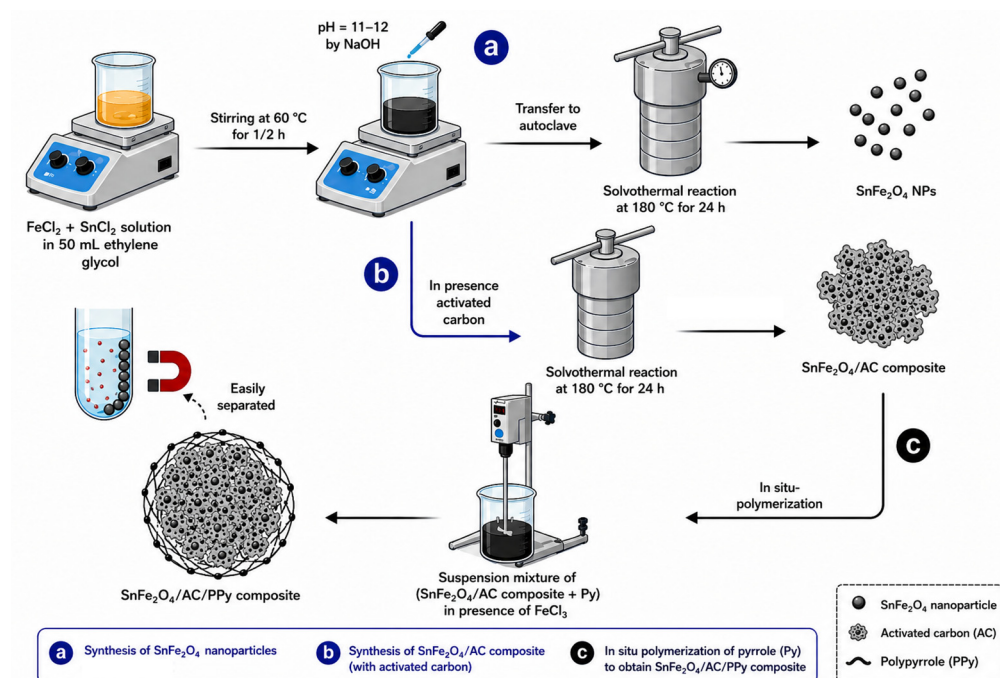


Figure 1. Synthesis steps for (a) SnFe_2O_4 NPs, (b) $\text{SnFe}_2\text{O}_4/\text{AC}$, and (c) $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ ternary nanocomposite.

2.3. Analyses

The phase composition and crystallographic characteristics of the synthesized materials were investigated through XRD analysis using a Rigaku MiniFlex II diffractometer (Tokyo, Japan), equipped with $\text{Cu K}\alpha$ radiation ($\lambda = 1.54059 \text{ \AA}$), 40 kV, 40 mA, and scanning at $2\theta = 10\text{--}70$ degrees with a step size of 0.02° and a scanning speed of $0.2^\circ/\text{min}$. Magnetic saturation, magnetization, and magnetic responsiveness were studied with the help of a vibrating sample magnetometer (VSM, LDJ 9600, LDJ Electronics Company, Troy, MI, USA). The Fourier transform infrared (FTIR) spectra were recorded using a Nicolet FTIR spectrometer (Thermo Scientific, Waltham, MA, USA) equipped with a DTGS detector. The spectra were collected in the wavenumber range of $4000\text{--}400 \text{ cm}^{-1}$ with a spectral resolution of 4 cm^{-1} and 32 accumulated scans. FTIR analysis was used to identify the surface functional groups and investigate the potential chemical interactions within the composite structure as well as between the adsorbent surface and metal ions. The scanning electron microscopy (SEM, JEOL JCM-6000 Plus, Tokyo, Japan) was used to study surface morphology, particle aggregation, and textural features. The elemental compositions were obtained by energy-dispersive X-ray spectroscopy (EDS, Waltham, MA, USA), which was performed at an accelerating voltage of 15.0 keV, and quantitative analysis was performed using the ZAF correction method. Chemical and band structure analyses were conducted using a multitechnique X-ray–UV–photoelectron spectroscopy and Low-Energy Inverse Photoemission Spectroscopy system (VersaProbeII, ULVAC-PHI, Inc., Chigasaki, Japan). The samples were irradiated with a focused monochromatic $\text{Al K}\alpha$ ($E = 1486.6 \text{ eV}$) X-ray beam with a diameter of $100 \mu\text{m}$, rastered over an area of $400 \times 400 \mu\text{m}^2$. The photoelectron take-off angle was 45° , and the pass energy in the analyzer was set to 117.50 eV (0.5 eV step) for survey scans and 46.95 eV (0.1 eV step) to obtain high-energy-resolution spectra for the C 1s, O 1s, N 1s, Sn 3d, Fe 2p, Pb 4f, and Cd 3d regions. A dual beam charge com-

pensation with 7 eV Ar⁺ ions and 1 eV electrons was used to maintain a constant sample surface potential regardless of the sample conductivity. All XPS spectra were charged with reference to the unfunctionalized, saturated carbon (C-C) C 1s peak at 284.8 eV. The operating pressure in the analytical chamber was less than 3×10^{-9} mbar. Deconvolution of spectra was carried out using PHI MultiPak software (v.9.9.3). The spectrum background was subtracted using the Shirley method. The behavior of the prepared materials with a wide pH range in terms of surface charge and their colloidal stability was measured by measurements using a Malvern ZetaSizer Nano ZS (Malvern Instruments Ltd., Malvern, UK). Quantitative measurement of residual concentrations of metal ions in aqueous solution at the start and end of adsorption experiments was done using a micro plasma atomic emission spectrometer (MP-AES, Agilent 4200, Santa Clara, CA, USA) to ensure that the performance of adsorption was adequately evaluated.

2.4. Adsorption Experiment

The batch experiments involving SnFe₂O₄/AC/PPy magnetic composite for the adsorption of Pb²⁺ and Cd²⁺ ions were carried out, and the average values were taken into account to analyze them. Initially, 50 mL aqueous solutions containing Pb²⁺ or Cd²⁺ at quantities (12.5, 25, 50, 75, 100, and 125 mg/L) were prepared, and the pH was set to desired values using 0.2 M NaOH and HCl. In every experiment, the adsorbent was added at a dosage of 25 mg for Pb²⁺ and 35 mg for Cd²⁺ solution. The mixing of the samples was done at 150 rpm in a water bath shaker maintained at 25 °C for 180 min to ensure the adsorption process. The solid phase was then magnetically isolated after treatment, and the remaining concentrations of metals were measured through micro-plasma atomic emission spectrometry (MP-AES, Agilent 4200). The equilibrium data of adsorption were discussed in terms of Langmuir, Freundlich, Temkin, and Dubinin–Radushkevich (D-R) isotherm models to characterize the binding properties and adsorption capacity. pH of the solution and adsorbent dosage were analyzed in a systematic manner with an initial concentration of 25 mg/L, which was taken as the representative level in the media. The pH of the solution was varied between 2 and 9, and the adsorbent dosage was varied between 5 and 45 mg to determine their effect on the uptake of Pb²⁺ and Cd²⁺ ions.

Under controlled conditions (initial pH of 6 and 7; adsorbent doses of 25 mg and 35 mg for Pb²⁺ and Cd²⁺, respectively; contact time of 180 min; and initial concentration of 12.5 mg/L), temperature-dependent studies were also conducted at a range of 25–45 °C. Based on these experiments, the thermodynamic parameters of the adsorption process were obtained.

To perform the kinetic analysis of the Pb²⁺ or Cd²⁺ adsorption, experiments were carried out at different contact times (5–180 min) under optimal conditions (pH 6 and 7, the dosage of adsorbent 25 and 35 mg for Pb²⁺ and Cd²⁺ ions, respectively, temperature 25 °C, initial concentration of metal 50 mg/L). Pseudo-first-order (PFO), pseudo-second-order (PSO), and intraparticle diffusion models were used to evaluate the sorption kinetics. It is worth noting that the adsorption efficacy and capacity of Pb²⁺ or Cd²⁺ at each time were calculated using the following Equations (1)–(3), respectively.

$$R\% = \frac{C_o - C_t}{C_o} \times 100\% \quad (1)$$

$$q_t = \frac{(C_o - C_t)}{m} \times V \quad (2)$$

$$q_e = \frac{(C_o - C_e)}{m} \times V \quad (3)$$

Here, C_0 and C_e represent the initial and equilibrium concentration of Pb^{2+} or Cd^{2+} in the solution (mg/L), respectively, and C_t indicates the concentration at a specific time. V is the volume of the solution (L), and m is the dosage of the adsorbent (g).

2.5. Reusability

The reusability of the magnetic composite was evaluated through successive adsorption–desorption cycles. After the adsorption experiment, an external magnet was used to readily separate the spent magnetic composite from the solution. The retained metal ions were desorbed by treating the recovered material with 0.1 M HCl under stirring at room temperature for 2 h for each cycle. The regenerated composite was then thoroughly washed with distilled water and dried. The dried material was subsequently reused under the same optimized adsorption conditions. A total of five adsorption–desorption cycles were performed to evaluate the reusability of the material.

3. Results and Discussion

3.1. Characterization of the Adsorbent

The FT-IR spectra of $SnFe_2O_4$, $SnFe_2O_4/AC$, and $SnFe_2O_4/AC/PPy$ composite are shown in Figure 2a, which were used for structural characterization and identification of the functional groups in the synthesized materials. The spectrum of $SnFe_2O_4$ showed a sharp absorption peak at 605 cm^{-1} , which is attributed to the characteristic stretching vibration mode of Fe–O in the prepared $SnFe_2O_4$ [25,26]. Moreover, the strong band at 3439 cm^{-1} might be assigned to the stretching vibrations of Fe–OH groups at the surface and to the physically adsorbed water. The Fe–O stretching vibrations of $SnFe_2O_4$ are associated with a prominent absorption peak in the spectrum of the $SnFe_2O_4/AC$ composite at 578 cm^{-1} . Furthermore, the broad band at 3435 cm^{-1} is probably caused by physically adsorbed water molecules and O–H stretching from surface hydroxyl groups. The peaks at 1629 , 1383 , and 1184 cm^{-1} are attributed to overlapping C=O stretching and C=C skeletal vibrations, C–H bending vibrations, and C–O stretching vibrations on the activated carbon surface, respectively [27]. These results, together with the Fe–O peak at 578 cm^{-1} , indicate that $SnFe_2O_4$ nanoparticles were successfully incorporated on the AC surface. The magnetic $SnFe_2O_4/AC/PPy$ composite showed peaks at 3439 cm^{-1} corresponding to the N–H and O–H bond stretching [28]. The strong peaks appearing at 1632 cm^{-1} were related to the C=O and C=C stretching vibrations in the AC and PPy rings, further confirming the presence of PPy and AC in the composite [29]. The obvious peak at 1548 cm^{-1} could be attributed to the C=C stretching vibration in the structure of the pyrrole ring [29]. Additionally, the peaks at 1453 and 1382 cm^{-1} correspond to the vibrations of C–N, and C–C in PPy ring [30]. The peak at around 1168 cm^{-1} corresponds to C–N bond stretching vibrations in the structure of the pyrrole ring [29,30]. A band at 1037 cm^{-1} was assigned to the in-plane bending vibration of the C–H and N–H bonds of the pyrrole ring [30,31]. The absorption bands at 875 and 776 cm^{-1} were attributed to C–C out-of-plane deformation vibrations and out-of-plane vibration of C–H or wagging vibration of pyrrole, respectively [29–31]. The Fe–O stretching peak in the composite shifted to 589 cm^{-1} , and the relative intensity of this peak receded as compared to pure $SnFe_2O_4$ NPs. These results confirmed that $SnFe_2O_4$ and AC have been successfully encapsulated inside the polymer matrix and composite.

Figure 2b shows the X-ray diffraction (XRD) patterns of $SnFe_2O_4$ nanoparticles, $SnFe_2O_4/AC$, and magnetic $SnFe_2O_4/AC/PPy$ composite. The XRD pattern of $SnFe_2O_4$ NPs exhibits sharp diffraction peaks at 18.1° , 30.05° , 35.40° , 36.72° , 43.10° , 53.37° , 57.02° , and 62.51° , which are correlated to the (111), (220), (311), (222), (400), (422), (511) and (440) crystal planes (JCPDS No. 73-1720) [32,33]. These reflections are characteristic of a spinel structure, confirming the successful formation of crystalline $SnFe_2O_4$ NPs. The

absence of impurity peaks indicates high phase purity of the synthesized nanoparticles. For SnFe₂O₄/AC, the primary diffraction peaks of SnFe₂O₄ are still very clear, which indicates that the spinel crystal structure is formed successfully even with the activated carbon. Additionally, a broad and weak diffraction band at $2\theta \approx 20\text{--}25^\circ$ is attributed to the amorphous structure of activated carbon [34]. The minor decrease in peak intensity of SnFe₂O₄ indicates the limitation of the crystal growth and enhanced dispersality of nanoparticles on the activated carbon surface, which is due to the strong interaction between the interfaces during the synthesis. In the SnFe₂O₄/AC/PPy composite, the primary diffraction peaks of SnFe₂O₄ are still visible in the SnFe₂O₄/AC/PPy composite, which confirms that the magnetic phase of the composite did not change its structure following the PPy polymerization [35,36]. The broad peak in the low-angle region ($\approx 20\text{--}25^\circ$) results from the overlap between the amorphous contributions of activated carbon and polypyrrole (PPy). The additional dampening of the SnFe₂O₄ peaks' intensities indicates the coating and partial encapsulation of SnFe₂O₄/AC by the PPy matrix. On the whole, the XRD findings indicate that in situ synthesis in the presence of activated carbon results in the well-dispersed SnFe₂O₄ nanoparticles anchored onto the AC, followed by the deposition of PPy, which maintains the magnetic spinel structure. This hierarchical structure is desirable in adsorption usage due to high surface area, versatile functional groups, and magnetic separability.

The average crystal sizes of all materials were determined using the Debye–Scherrer equation, which relates the broadening of diffraction peaks to the size of coherently diffracting domains. The equation is expressed as follows:

$$D = \frac{K \cdot \lambda}{\beta \cdot \cos\theta} \quad (4)$$

These symbols, D , K , λ , β , and θ , denote the average crystal size (nm), the dimensionless shape factor, which is typically equal to 0.9, X-ray source wavelength (Cu $K\alpha$ radiation, $\lambda = 0.154$ nm), FWHM (full width at half-maximum intensity) of the peak, and diffraction angle (in radians), respectively. It should be noted that crystallite size estimation using the Scherrer equation results in an approximate value, as peak broadening may also be influenced by instrumental broadening and lattice strain effects. Therefore, the calculated values should be regarded as apparent crystallite sizes rather than absolute crystallite dimensions. The average crystallite size of pristine SnFe₂O₄ nanoparticles, calculated using the Scherrer equation, was 19.31 nm, with a crystallinity of 80.53%. When SnFe₂O₄ nanoparticles were synthesized in situ in the presence of activated carbon, the crystallite size slightly decreased to 18.97 nm, while the crystallinity decreased to 43.45% for the SnFe₂O₄/AC composite. This behavior indicates that activated carbon participates during the nucleation and growth stages of SnFe₂O₄, acting as a heterogeneous nucleation substrate and spatial confinement matrix. As a result, the growth of SnFe₂O₄ crystallites is moderately restricted, leading to improved dispersion and suppressed crystallite coalescence without altering the spinel crystal structure. The reduction in crystallinity is attributed to the incorporation of the partially amorphous activated carbon phase, which contributes to the broadening and attenuation of the diffraction peaks. A more pronounced reduction in crystallite size to 16.46 nm and crystallinity to 38.88% was observed for the SnFe₂O₄/AC/PPy composite. This behavior is attributed to the subsequent in situ polymerization of polypyrrole, which forms a coating layer around the SnFe₂O₄/AC. The PPy matrix further limits crystallite growth through interfacial confinement and lattice strain, while its largely amorphous nature contributes to the further decrease in crystallinity, as evidenced by the additional broadening and attenuation of the XRD peaks. Nevertheless, the characteristic diffraction peaks of spinel SnFe₂O₄ remained clearly visible, confirming the preservation of the crys-

talline ferrite structure. The gradual decrease in both crystallite size and crystallinity is fully consistent with the XRD results and confirms the successful stepwise construction of the hierarchical $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite.

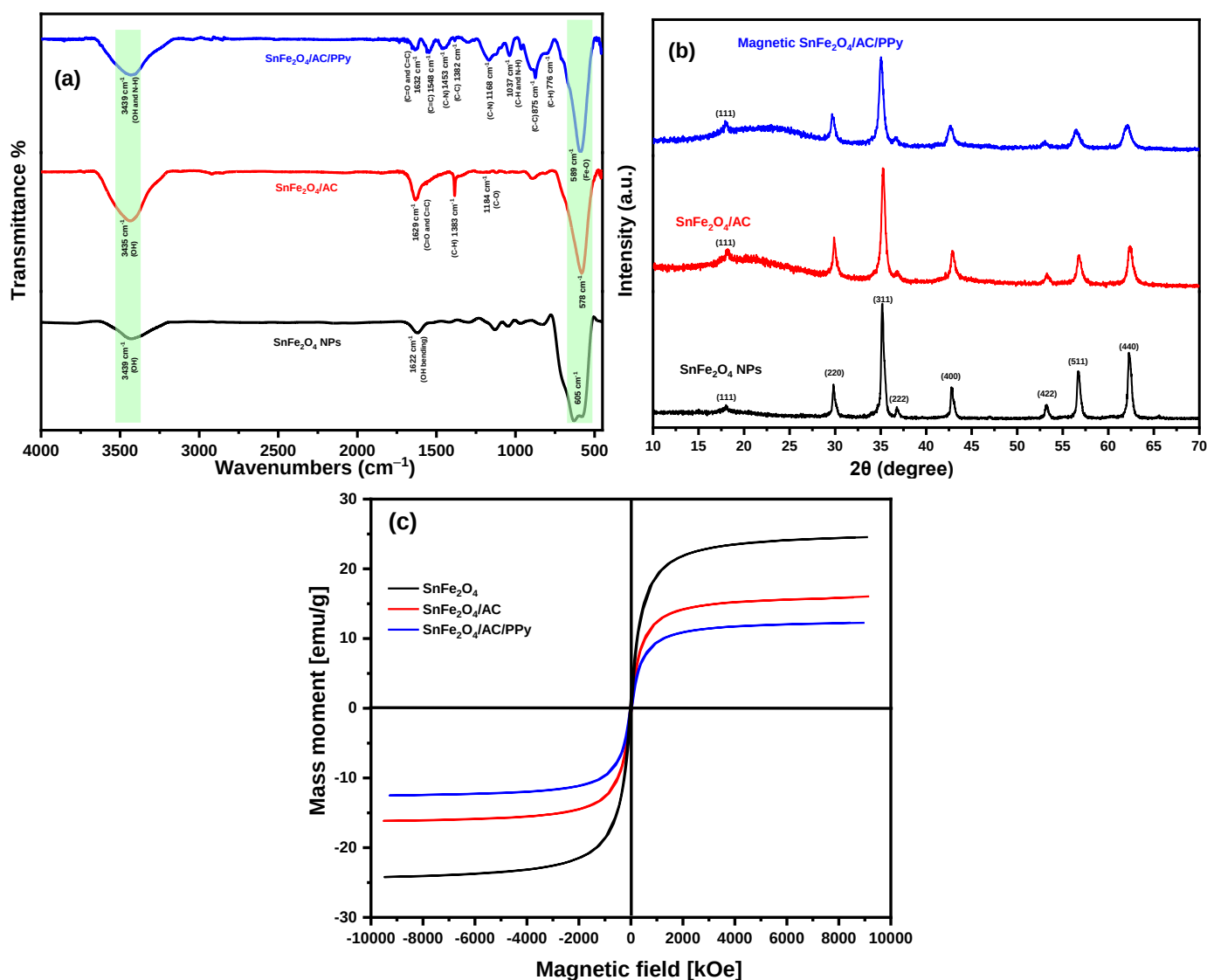


Figure 2. (a) FT-IR spectra, (b) XRD pattern, and (c) VSM spectra of SnFe_2O_4 , $\text{SnFe}_2\text{O}_4/\text{AC}$, and $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite.

The magnetic properties of SnFe_2O_4 , $\text{SnFe}_2\text{O}_4/\text{AC}$, and $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ were studied at room temperature using vibrating sample magnetometry (VSM), and the magnetization curves are shown in Figure 2c. All samples exhibit superparamagnetic behavior, as evidenced by the absence of obvious hysteresis. The saturation magnetization (M_s) decreases in the following order: SnFe_2O_4 (24.21 emu/g), $\text{SnFe}_2\text{O}_4/\text{AC}$ (16.15 emu/g), and $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ (12.33 emu/g). This decrease in M_s is mainly explained by the incorporation of non-magnetic components, such as AC and PPy, which cause a dilution effect and weaken the overall magnetic response. However, even with the reduction in saturation magnetization, the $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite retains sufficient magnetic responsiveness, and can be rapidly and efficiently separated from solutions using an external magnetic field after adsorption, highlighting its practical applicability.

SEM-EDS analyses were employed to study the morphological and elemental composition of SnFe_2O_4 nanoparticles (NPs), $\text{SnFe}_2\text{O}_4/\text{AC}$, and $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite.

The SEM image of pure SnFe_2O_4 NPs (Figure 3a) shows a near-spherical shape that tends to agglomerate, which is usually attributed to the magnetic dipole–dipole interaction and high surface energy of the nanosized particles. The particle size is approximately nanometer-scale, which is consistent with the crystalline properties observed in the XRD analysis. EDS confirms the presence of Sn, Fe, and O with atomic% 1:2:4, which confirms the successful formation of SnFe_2O_4 without detectable impurities. The corresponding EDS mass values are summarized in Table 1. For $\text{SnFe}_2\text{O}_4/\text{AC}$ (Figure 3c), the SEM image depicts that SnFe_2O_4 nanoparticles are evenly dispersed and anchored on the rough and porous surface of the activated carbon. Severe agglomeration of nanoparticles is effectively inhibited by the presence of AC, showing that in situ synthesis enhances good interfacial interactions between SnFe_2O_4 and AC. This porous framework of carbon offers many active sites, and the dispersion of nanoparticles is possible. The presence of Sn, Fe, O, and C in the EDS spectra proves the successful formation of the composite. The surface morphology of the $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite (Figure 3e) becomes more compact and irregular, indicating the creation of a coating PPy layer on the $\text{SnFe}_2\text{O}_4/\text{AC}$ structure. The nanoparticles and carbon surface are mainly covered by the PPy matrix, which contributes to a more connected and hierarchical morphology. The coating is expected to add more functional groups to it and enhance the performance of adsorption. The spectrum of EDS also confirms the existence of C, N, Sn, Fe, and O, which proves the efficient polymerization of PPy on the composite. All these SEM–EDS studies stated that a hierarchical $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite was successfully prepared in a stepwise manner with good surface morphology and elemental distribution, which are necessary for effective heavy metal adsorption. Figure 4a,c illustrates the SEM images of the $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite following adsorption of Pb^{2+} and Cd^{2+} , respectively. The SEM images demonstrate that there is an observable difference in the surface texture of the composite, which has become rougher and partially covered, and this is an indication that there is an effective interaction between metal ions and available adsorption sites. The distinct presence of Pb (Figure 4b) and Cd (Figure 4d) in the respective EDS spectra indicates successful uptake of heavy metal ions. These results prove that the porous structure, the PPy coating, and the dispersed magnetic nanoparticles all contribute to the increase in the adsorption capacity.

Table 1. Quantitative EDX analysis of the synthesized samples.

Sample	Fe (%)	O (%)	N (%)	C (%)	Sn (%)	Pb (%)	Cd (%)
SnFe_2O_4	49.42	28.85	--	--	21.72	--	--
$\text{SnFe}_2\text{O}_4/\text{AC}$	32.41	37.01	--	20.38	10.20	--	--
$\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$	27.73	22.40	3.33	40.66	5.88	--	--
$\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ after Pb^{2+} ions adsorption	16.50	23.42	3.66	50.83	1.57	4.02	--
$\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ after Cd^{2+} ions adsorption	23.93	40.38	5.87	23.94	4.52	--	1.35

The textural properties of SnFe_2O_4 , $\text{SnFe}_2\text{O}_4/\text{AC}$, and $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ were investigated by nitrogen adsorption–desorption measurements, and the specific surface area was determined using the Brunauer–Emmett–Teller (BET) method. The adsorption–desorption isotherms and pore size distribution curves are presented in Figure 5, while the calculated textural parameters are summarized in Table 2. As shown in Figure 5, the adsorption–desorption isotherms of $\text{SnFe}_2\text{O}_4/\text{AC}$ and $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composites can be classified as Type IV according to the IUPAC classification, which is characteristic of mesoporous materials [37,38]. The presence of a pronounced hysteresis loop indicates capillary condensation within the mesopores and confirms the mesoporous nature of the synthesized composites. This observation is consistent with the pore size distribution

curves, which reveal the predominance of mesopores throughout the composite structure. Pure SnFe_2O_4 exhibited a relatively low specific surface area of $31.9 \text{ m}^2/\text{g}$, indicating limited availability of surface-active sites. Upon incorporation of activated carbon (AC), a remarkable increase in textural properties was observed. The $\text{SnFe}_2\text{O}_4/\text{AC}$ composite showed a very high BET surface area of $1080.6 \text{ m}^2/\text{g}$, with a total pore volume of $0.433 \text{ cm}^3/\text{g}$ and an average pore diameter of 6.16 nm . This significant enhancement is attributed to the highly porous structure and large intrinsic surface area of activated carbon, which contributes abundant adsorption sites and facilitates mass transfer. After polymerization of polypyrrole on the $\text{SnFe}_2\text{O}_4/\text{AC}$ surface, the BET surface area decreased to $352.3 \text{ m}^2/\text{g}$, while the total pore volume decreased to $0.112 \text{ cm}^3/\text{g}$. In contrast, the average pore diameter increased to 8.27 nm . The reduction in surface area and pore volume is primarily attributed to the deposition of PPy within the pore structure and on the composite surface, resulting in the partial blockage or filling of micropores and mesopores. The increase in average pore diameter suggests that smaller pores were preferentially occupied by the polymer layer, leaving a greater proportion of larger pores accessible to nitrogen molecules during analysis. Despite the decrease in specific surface area following PPy incorporation, the $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite retained a well-developed mesoporous structure and a relatively high surface area suitable for adsorption applications. Moreover, the PPy coating introduced nitrogen-containing functional groups capable of enhancing interactions with metal ions through coordination and electrostatic attraction mechanisms. Therefore, the adsorption performance of the composite is governed not only by its favorable textural properties but also by the synergistic contribution of surface functional groups, which collectively enhance the accessibility and affinity of adsorption sites toward target metal ions.

X-ray photoelectron spectroscopy (XPS) was then utilized to analyze the surface composition and chemical states of the synthesized $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite. The presence of Fe, Sn, O, C, and N, as shown in Figure 6a, was evidenced by the survey spectrum that illustrates the successful incorporation of SnFe_2O_4 , activated carbon (AC), and polypyrrole (PPy) into the composite structure. The high-resolution Fe 2p spectrum (Figure 6b) was deconvoluted into major peaks. The peak at 710.3 eV can be attributed to the Fe^{3+} characteristic of the ferrite phase, and the peak at 715.2 eV is specific and corresponds to the $\text{Fe}^{3+}/\text{Sn } 3p_{3/2}$, confirming the interaction of iron and tin species. Moreover, the peak located at 708.5 eV could be attributed to Fe–Sn bonding; this confirms that Sn can easily incorporate into the ferrite framework and combine with iron species to produce SnFe_2O_4 phase. The high-resolution Sn 3d spectrum (Figure 6c) exhibits a main peak at 486.5 eV corresponding to Sn^{2+} and/or Sn^{4+} species. The coexistence of these oxidation states suggests the presence of mixed-valence tin, which is consistent with the spinel SnFe_2O_4 structure. The O 1s spectrum (Figure 6d) was resolved into three components. The peak at 530.1 eV is attributed to lattice oxygen associated with metal–oxygen bonds in the ferrite structure. The peaks at 531.4 and 532.7 eV are assigned to surface oxygen species related to $\text{O}=\text{C}$ groups and oxygen-deficient sites in metal oxides, as well as $\text{O}-\text{C}$ and $-\text{OH}$ functionalities originating from the carbonaceous and polymeric components of the composite. The C 1s spectrum (Figure 6e) reveals several characteristic carbon species associated with activated carbon and the PPy matrix. The peaks at 284.4 and 284.8 eV correspond to $\text{C}=\text{C}$ (sp^2) and $\text{C}-\text{C}$ (sp^3) bonds, respectively. The component at 286.0 eV is attributed to $\text{C}-\text{OH}$, $\text{C}-\text{NH}$, and/or $\text{C}-\text{O}-\text{C}$ groups, while the peak at 287.1 eV is assigned to $\text{C}=\text{O}$ and/or $\text{O}-\text{C}-\text{O}$ species. The signal at 288.1 eV corresponds to $\text{O}=\text{C}-\text{O}$ groups, whereas the feature at 289.2 eV is attributed to $\text{O}-(\text{C}=\text{O})-\text{O}$ species and/or shake-up satellite peaks. The shake-up excitation originates from $\pi-\pi^*$ transitions of sp^2 -hybridized carbon and aromatic structures, providing further evidence for the presence of conjugated

carbon domains within the composite. These results confirm the successful incorporation of activated carbon and polypyrrole into the $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite. In addition, the N 1s spectrum shown in (Figure 6f) can be deconvoluted into two separated peaks at 399.8 eV and 401.0 eV, corresponding to C-NH and C-NH⁺, respectively, confirming that PPy is incorporated into the synthesized composite. The synthesis of the $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite is strongly supported by XPS results. The presence of Fe, Sn, O, C, and N, along with typical chemical states and binding energies from the high-resolution spectra, has confirmed that a ternary composite has been successfully constructed comprising SnFe_2O_4 nanoparticles, activated carbon, and polypyrrole in an integrated form.

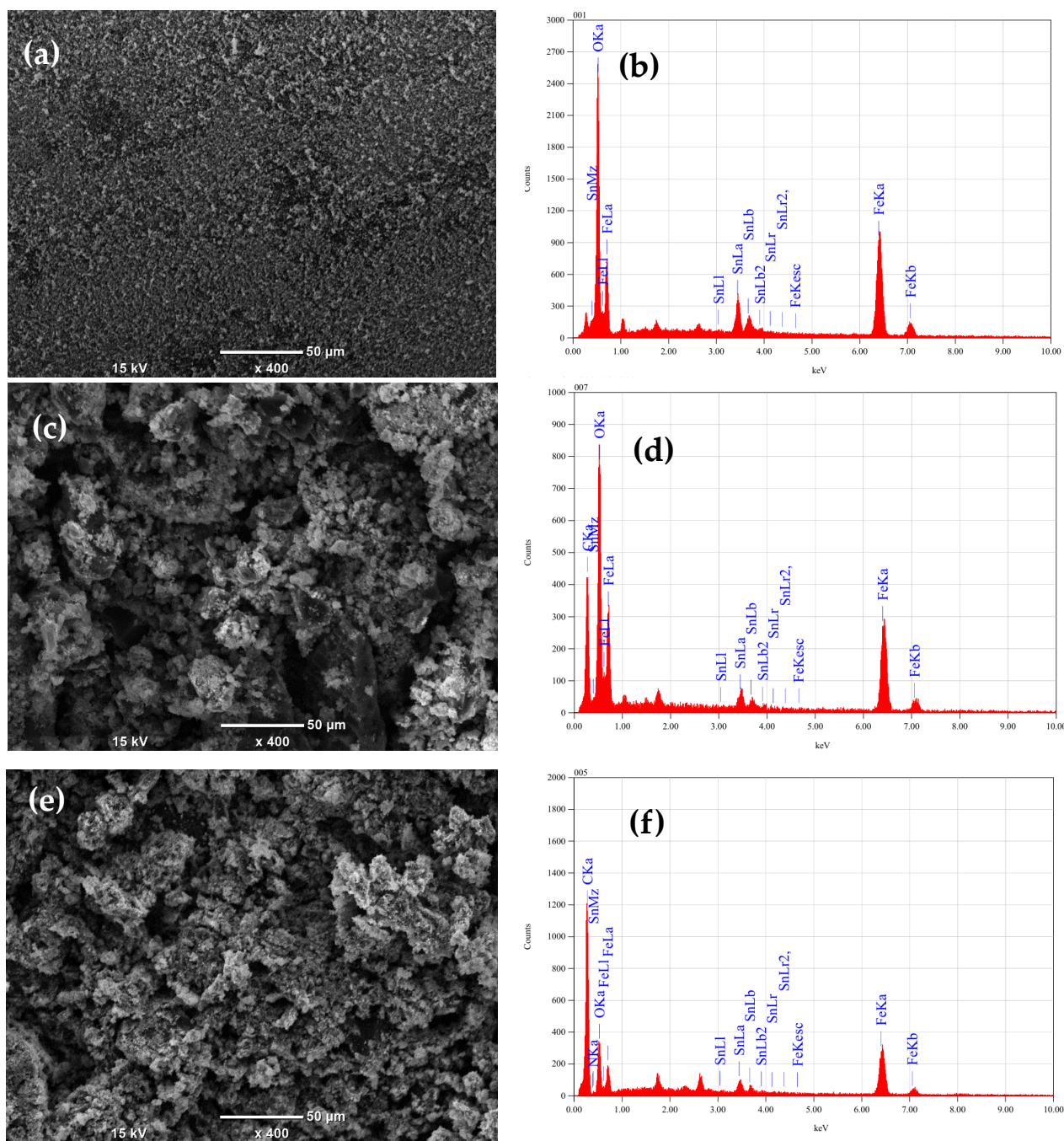


Figure 3. SEM–EDS images for (a,b) SnFe_2O_4 NPs, (c,d) $\text{SnFe}_2\text{O}_4/\text{AC}$, and (e,f) $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite.

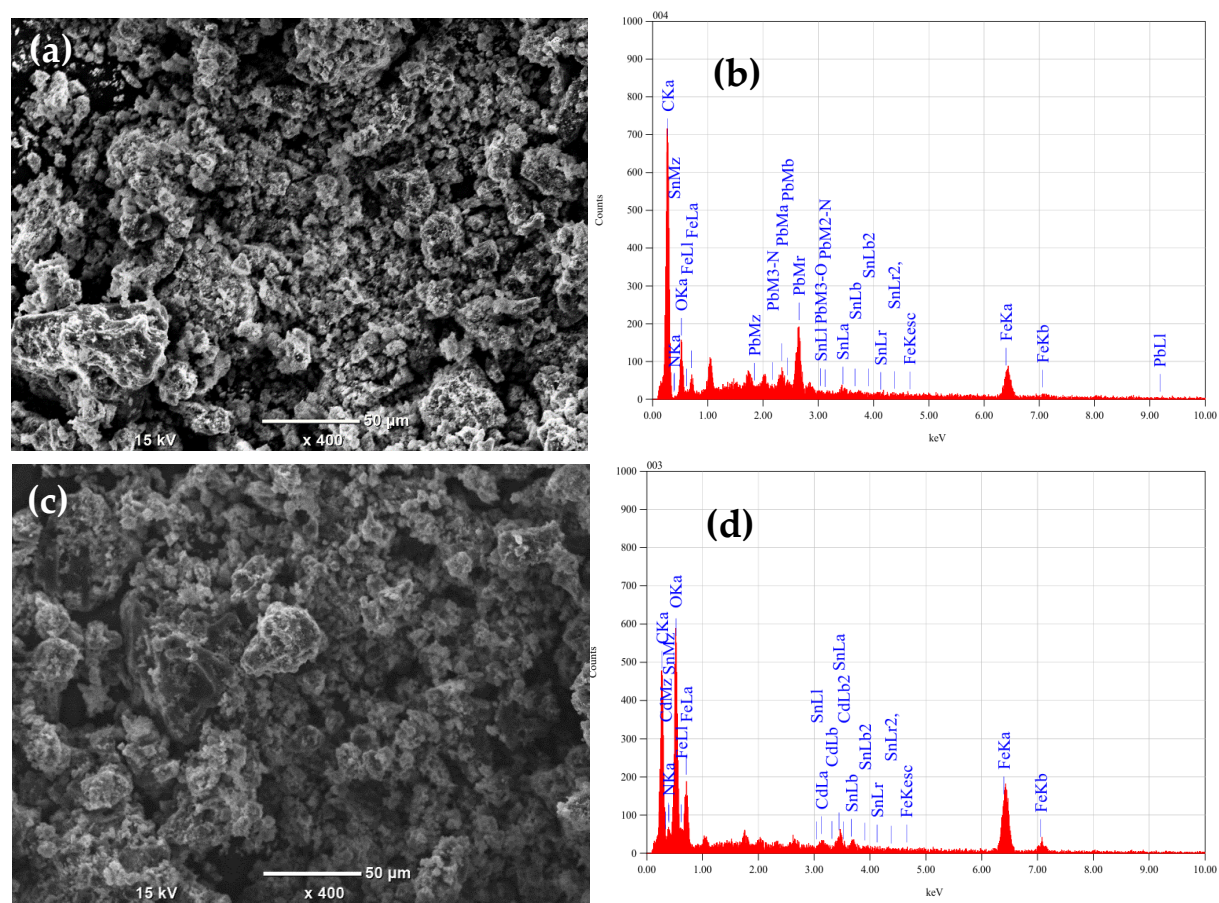


Figure 4. SEM-EDS images for SnFe₂O₄/AC/PPy composite after (a,b) Pb²⁺ and (c,d) Cd²⁺ adsorption, respectively.

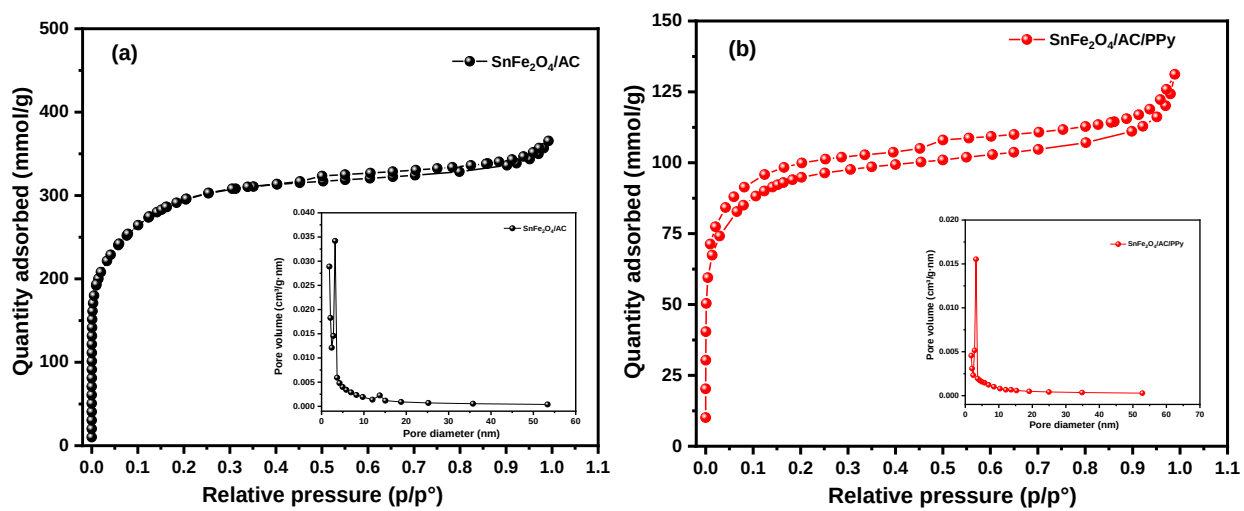


Figure 5. N₂ adsorption–desorption isotherm plots and pore size distribution curves of (a) SnFe₂O₄/AC, and (b) SnFe₂O₄/AC/PPy.

Table 2. Textural parameters of SnFe₂O₄, SnFe₂O₄/AC, and SnFe₂O₄/AC/PPy.

Sample	BET Surface Area (m ² /g)	Total Pore Volume (cm ³ /g)	Pore Diameter (nm)
SnFe ₂ O ₄	31.9	--	--
SnFe ₂ O ₄ /AC	1080.6	0.433	6.16
SnFe ₂ O ₄ /AC/PPy	352.3	0.112	8.27

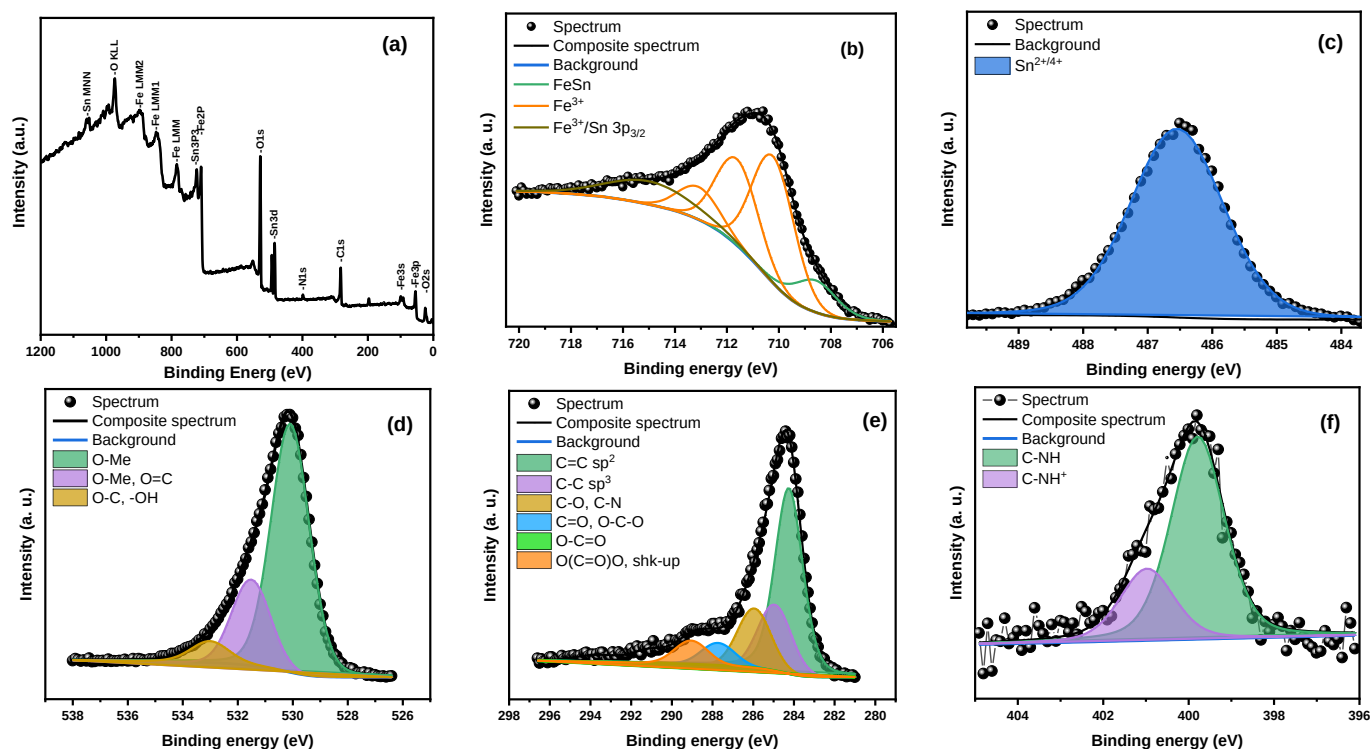


Figure 6. XPS spectra of the $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite: (a) survey spectrum and high-resolution spectra of (b) Fe 2p, (c) Sn 3d, (d) O 1s, (e) C 1s, and (f) N 1s.

3.2. Effect of Adsorbent Quantity and pH

Adsorption performance in aqueous systems is strongly influenced by several operational parameters, including contact time, adsorbent dosage, temperature, solution characteristics, and the initial concentration of contaminants. These factors play a crucial role in determining the adsorption efficiency and overall removal capacity of the adsorbent. Among the investigated operational parameters, adsorbent dosage has a crucial impact on determining the adsorption efficiency of metal ions. Changes in the quantity of adsorbent alter the number of accessible active sites, therefore affecting both the contaminant removal efficiency and the cost-effectiveness of the adsorption process. Figure 7a represents the effect of $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ dosage on the removal efficiency of Pb^{2+} and Cd^{2+} ions in the dosage range of 5–45 mg under the same experimental conditions (pH for Pb^{2+} and Cd^{2+} solution = 6 and 7, respectively, metal-ion concentration = 25 mg/L at temperature 25 °C). The two metal ions show a clear increase with an increase in the adsorbent dose from 5 mg to 45 mg. At lower dosages, the few active sites available restrict the uptake of metal ions, and hence, the removal efficiency is low. As the adsorbent dosage increases, the surface area and the density of functional binding sites are exposed; therefore, this greatly improves the interaction between metal ions and the adsorbent, increasing adsorption. Consequently, the efficiencies of Pb^{2+} and Cd^{2+} removal increase rapidly, approaching near-full removal at higher doses. Nevertheless, an additional increase in the removal rate of Pb^{2+} and Cd^{2+} is minimal at dosages above 25 mg and 35 mg, respectively, suggesting that the majority of the active sites are already occupied and the system approaches almost maximum saturation [39]. This indicates that the further addition of adsorbent does not enhance performance in a proportional way and can result in aggregation of the particles or overlapping of the sites, thus diminishing the adsorption efficiency in terms of per unit mass. Hence, the emphasized dosages are the optimal conditions that can achieve a balance of high material utilization and removal efficiency.

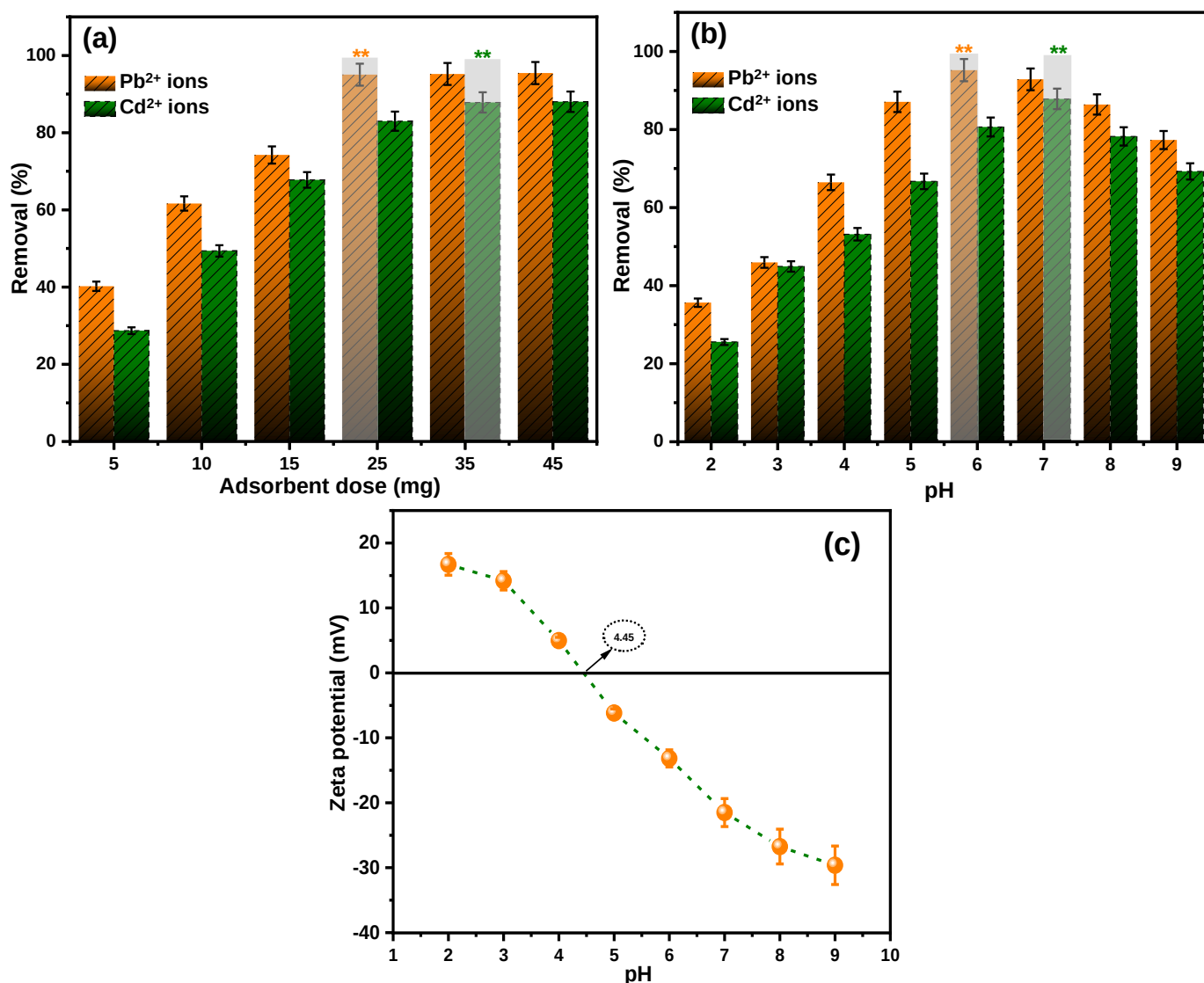


Figure 7. Effect of adsorbent dosage (a) and pH (b) on the adsorption efficiency of Pb²⁺ and Cd²⁺ ions, and the influence of pH on the surface charge on the adsorbent (c).

The solution pH is the strongest parameter since it not only regulates the speciation of the metal ions but also controls the surface characteristics of the adsorbent and determines the protonation–deprotonation behavior of the surface functional groups. Consequently, pH has a direct influence on electrostatic interaction and surface complexation processes that lead to metal uptake. The pH influence on the adsorption of Pb²⁺ and Cd²⁺ ions onto the SnFe₂O₄/AC/PPy composite was also assessed systematically at adsorbent mass = 35 mg, metal-ion concentration = 25 mg/L, as in Figure 7b. A high level of acidic conditions significantly decreased removal efficiency but gradually improved as the pH increased. Optimal media were observed at pH 6 in the case of Pb²⁺ and pH 7 in the case of Cd²⁺ ions to achieve high removal efficiency of 95.21% and 87.84%, respectively, and the adsorption efficiency began to decrease after these optimal pH values. This pattern indicates the presence of an optimal pH range that promotes efficient adsorption, highlighting the strong influence of solution chemistry on the adsorption performance.

The reason behind the observed behavior is governed by both the point of zero charge (pH_{pzc}) of the composite and the aqueous speciation of Pb²⁺ and Cd²⁺ ions. pH_{pzc} of SnFe₂O₄/AC/PPy was determined at 4.45, as seen in Figure 7c, and this means that the adsorbent surface is positively charged at lower pH_{pzc} and negatively charged at higher pH_{pzc}.

Electrostatic repulsion of the positively charged surface with the divalent metal cations occurs at pH values below pH_{pzc} , which inhibits adsorption. In addition, the large quantity of protons present in acidic media also competes with metal ions over available sites of adsorption, which greatly inhibits the interaction of metals on the surface [40]. Over the pH_{pzc} , the deprotonation of the functional groups on its surface forms negatively charged functional groups that increase electrostatic interaction with Pb^{2+} and Cd^{2+} ions [41]. The simultaneous decrease in the concentration of protons relieves competitive adsorption, and more of the metal ions can have access to active binding sites. Pb^{2+} and Cd^{2+} are mostly in the divalent cationic state at pH 6 and 7–8, respectively, so adsorption is preferred in this pH range. At elevated pH values, insoluble metal hydroxides are formed, like $\text{Pb}(\text{OH})_2$ and $\text{Cd}(\text{OH})_2$, and apparent adsorption efficiency declines as chemical precipitation becomes a powerful mechanism in removal [42]. Therefore, adsorption experiments conducted at high pH were not used for isothermal or kinetic modeling to avoid interference from precipitation effects. All adsorption parameters reported in this study were derived from experiments performed at pH = 6 and 7, where Pb^{2+} and Cd^{2+} remained predominantly soluble, and adsorption was the dominant removal mechanism. Furthermore, Pb^{2+} exhibited a higher removal efficiency than Cd^{2+} , indicating a greater binding affinity toward the active sites of the $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite. This preferential uptake can be associated with variations in the physicochemical characteristics of the metal ions, such as ionic radius, hydration energy, and coordination behavior with the surface functional groups of the adsorbent, which influence the strength of adsorbate–adsorbent interactions [43,44].

3.3. Adsorption Time and Kinetic Study

Contact time is an important physical parameter in adsorption studies, due to its direct effect on contamination uptake by the adsorbent. This is very useful as it tells about the time that metal ions take to interact with active adsorption sites and reach equilibrium conditions. Maximum adsorption capacity can only be achieved when an adequate amount of contact time is permitted. On the other hand, short contact times will not allow complete adsorption to take place and limit metal-ion removal. In order to analyze the equilibrium behavior of the system, batch adsorption experiments were conducted under controlled environmental conditions. Figure 8a,b shows the time-dependent behavior of the removal of Pb^{2+} and Cd^{2+} ions from aqueous solution by the $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite over contact periods ranging from 5 to 180 min. The adsorption experiments were conducted at an initial metal concentration of 50 mg/L for both metal ions, and at a constant temperature of 25 °C with optimized pH 6 for Pb^{2+} and 7 for Cd^{2+} . The adsorbent dosages employed in the kinetic experiments were 25 mg for Pb^{2+} and 35 mg for Cd^{2+} . A sharp increase in adsorption efficiency was observed during the early stage of the adsorption process, particularly within the first 15 min for Pb^{2+} and the first 25 min for Cd^{2+} . This behavior indicates the rapid occupation of readily accessible active sites on the adsorbent surface. The high initial adsorption rate can be attributed to the abundance of vacant adsorption sites and the strong driving force arising from the concentration gradient between the aqueous phase and the adsorbent surface [45,46]. As the duration of interaction increased, the rate of adsorption gradually slowed, and the equilibrium was established at 110 min in the case of Pb^{2+} and 130 min in the case of Cd^{2+} . Beyond these periods, the extension of contact time did not give any significant enhancement to the removal of metals, implying that the active sites of the adsorption had been saturated. Additionally, the obtained results indicate that Pb^{2+} possesses a significantly higher adsorption affinity toward the $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite surface than Cd^{2+} , as evidenced by its higher removal efficiency and faster attainment of adsorption equilibrium. This enhanced adsorption performance suggests a stronger binding affinity of Pb^{2+} for the active sites and surface functional groups of

the composite, resulting in more stable metal–adsorbent interactions and facilitating its preferential adsorption over Cd^{2+} ions.

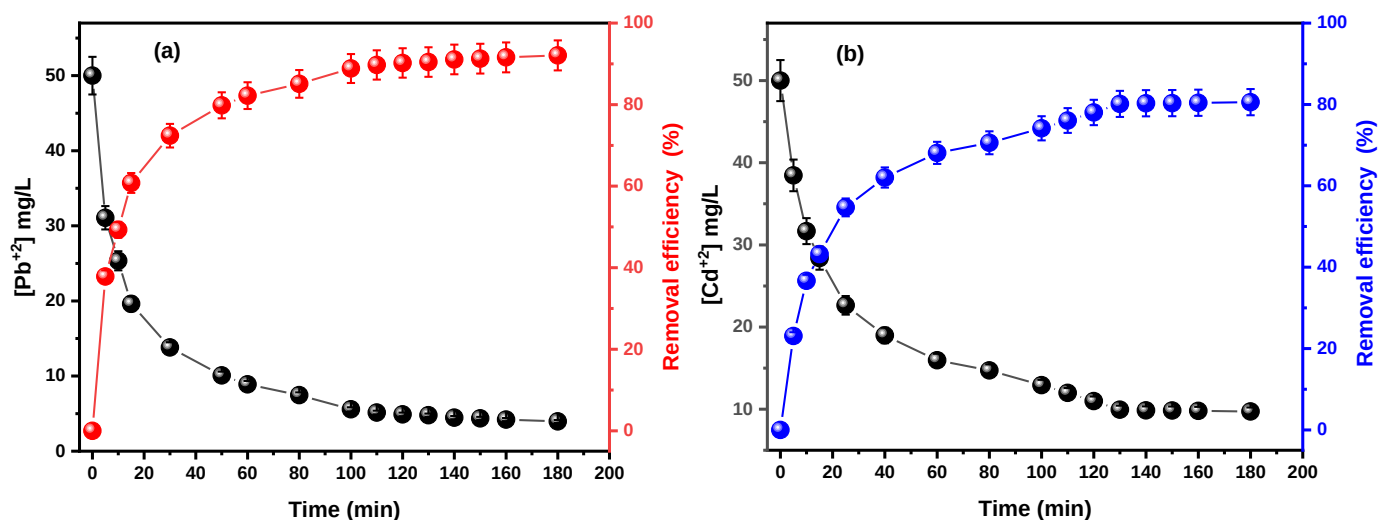


Figure 8. The concentration changes and removal efficiency for (a) Pb^{2+} and (b) Cd^{2+} ions during adsorption times.

In a bid to understand the adsorption process better, the kinetic data of both Pb^{2+} and Cd^{2+} removal by the $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite were assessed using pseudo-first order (PFO) and pseudo-second order (PSO) and intraparticle diffusion kinetic models in nonlinear equations (Equations (5)–(7)) [47–49].

PFO:	$q_t = q_e \left(1 - e^{-k_1 t}\right)$	(5)
PSO:	$q_t = \frac{k_2 q_e^2 t}{1 + k_2 q_e t}$	(6)
Intra-particle diffusion:	$q_t = K_p t^{0.5} + C$	(7)

In these equations, t is the contact time (min); k_1 (min^{-1}), k_2 ($\text{g}/\text{mg}\cdot\text{min}$), and k_p ($\text{mg}/\text{g}/\text{min}$) are the rate constants of the PFO, PSO, and intraparticle diffusion models, respectively; q_t (mg/g) is the quantity of metal adsorbed with time t (min); and q_e (mg/g) is the calculated equilibrium adsorption capacity.

The results of the fitting, as shown in Figure 9a–c, are collected in Table 3, which indicates that the PSO is the best model to describe the experimental data, as evidenced by its higher correlation coefficients (R^2) compared to the other models. The high degree of agreement implies that the adsorption of Pb^{2+} and Cd^{2+} on the $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite is dominated by chemisorption processes, which entail the exchange or sharing of electrons between the metal ions and active sites in the composite surface [50,51]. The calculated k_2 of Pb^{2+} adsorption was higher than that of Cd^{2+} adsorption, indicating the faster Pb^{2+} adsorption onto the surface of $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite. Further information regarding the diffusion behavior was acquired using the intraparticle diffusion plots in Figure 9c. The multilinear nature of the plots and their deviation from the origin indicate that the adsorption process occurs in a series of successive steps instead of occurring under one diffusion mechanism [52]. The initial linear region is associated with a fast external mass transfer or boundary-layer diffusion whereby the metal ions are rapidly carried off the solution into the adsorbent surface, as indicated by relatively large k_{p1} values. The second stage is a slower intraparticle diffusion process of the ions into the pores of the composite, which becomes the rate-controlling step. Lastly, the third area is linked with the

slow diffusion of the ions to deeper microporous active sites, which leads to adsorption equilibrium [53–55].

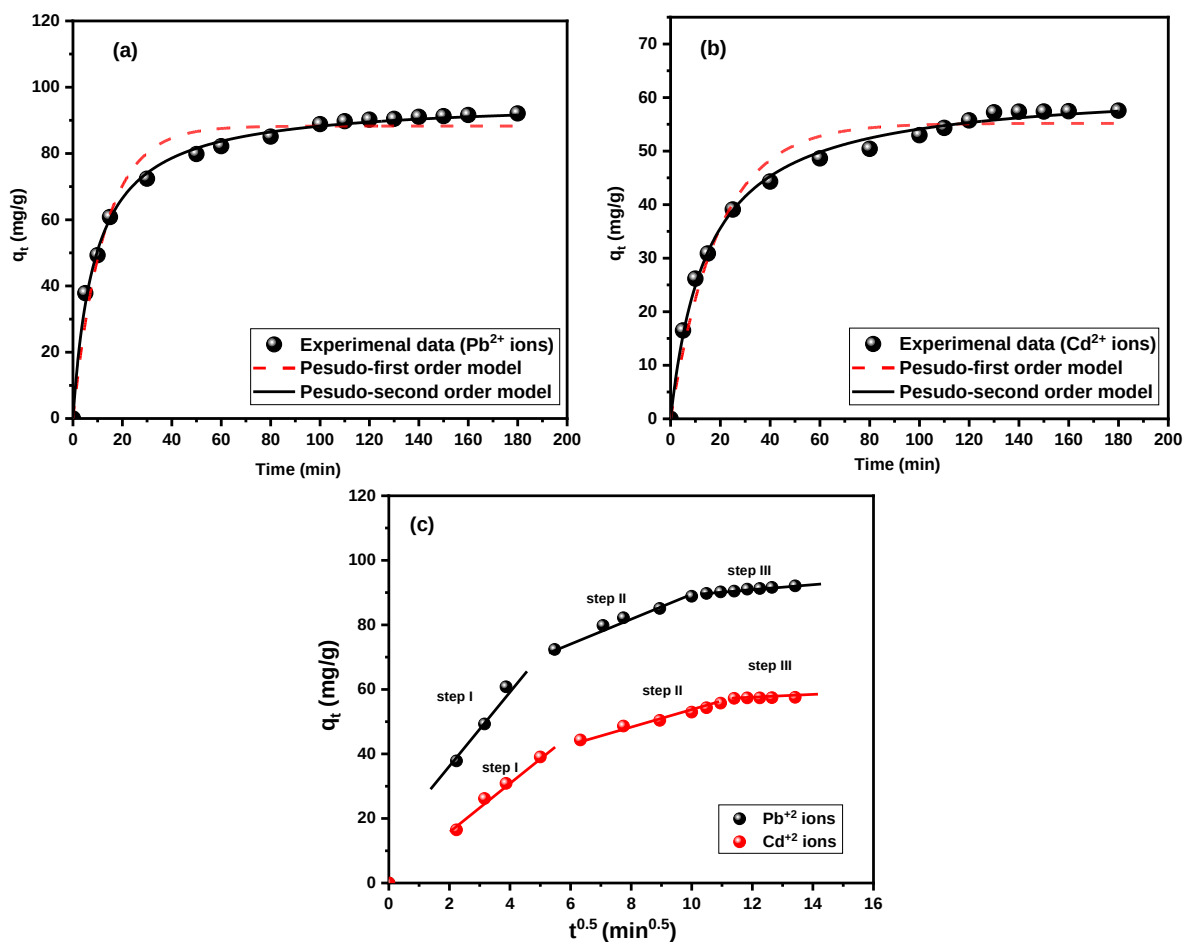


Figure 9. (a,b) Nonlinear regression of adsorption kinetic models, and (c) linear regression of the intraparticle diffusion model for Pb^{2+} and Cd^{2+} using $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ ternary composite.

Table 3. Kinetic parameters for the adsorption of Pb^{2+} and Cd^{2+} ions using $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ ternary composite.

Model	Parameter	Pb^{2+} Ion	Cd^{2+} Ion
PFO	$q_{e, \text{ experimental}} \text{ (mg/g)}$	92.08	57.54
	$q_{e, \text{ calculated}} \text{ (mg/g)}$	88.28	62.20
	$K_1 \text{ (1/min)}$	0.079	0.052
	Adjusted R^2	0.970	0.976
	R^2	0.973	0.973
PSO	$q_{e, \text{ experimental}} \text{ (mg/g)}$	92.08	57.54
	$q_{e, \text{ calculated}} \text{ (mg/g)}$	96.13	55.18
	$K_2 \times 10^{-4} \text{ (g/mg min)}$	11.60	10.08
	Adjusted R^2	0.998	0.997
	R^2	0.997	0.996
IPD step I	$K_{p1} \text{ (mg/g/min)}$	13.93	8.04
	Adjusted R^2	0.994	0.988
	R^2	0.988	0.983
IPD step II	$K_{p2} \text{ (mg/g/min)}$	3.53	2.35
	Adjusted R^2	0.981	0.991
	R^2	0.975	0.989

3.4. Metal-Ion Concentration and Isotherm Study

The initial concentration of metal ions in solution plays a significant role in determining the adsorption performance of an adsorbent. Changes in the initial concentration affect the driving force for mass transfer between the aqueous phase and the adsorbent surface, thereby influencing the adsorption process. The effect of initial metal-ion concentration on the adsorption performance of the $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite was studied within the (12.5, 25, 50, 75, 100, and 125 mg/L) concentration range under the same conditions of the experiment (pH for Pb^{2+} and Cd^{2+} solution was 6 and 7, adsorbent dosage for Pb^{2+} and Cd^{2+} was 25 and 35 mg, respectively). Figure 10a,b show that increasing the concentration of Pb^{2+} and Cd^{2+} affected both the removal efficacy and the adsorption capacity. At the lower initial concentrations, the percentage of metal ions removed was high because of the fact that active sites are available in large numbers as compared to the number of ions available in the solution. Nevertheless, the removal efficiency decreased with an increasing metal-ion concentration, which indicates progressive saturation of adsorption binding sites on the composite surface [56,57]. Moreover, the high concentrations may further increase the repulsion of the electrostatic force between incoming metal ions and existing adsorbed ones, making uptake even more difficult [58]. Although the removal percentage decreased, adsorption capacity increased significantly from 24.01 to 169.56 mg/g of Pb^{2+} and from 16.51 to 87.29 mg/g of Cd^{2+} with increasing the metal concentration from 12.5 to 125 mg/L. The observed behavior can be explained by the stronger concentration gradient between the aqueous phase and the adsorbent surface, which occurs when there are more metal ions. The increasing gradient strengthens the driving force for mass transfer, which helps ions diffuse more effectively towards active sites until the system reaches saturation [58].

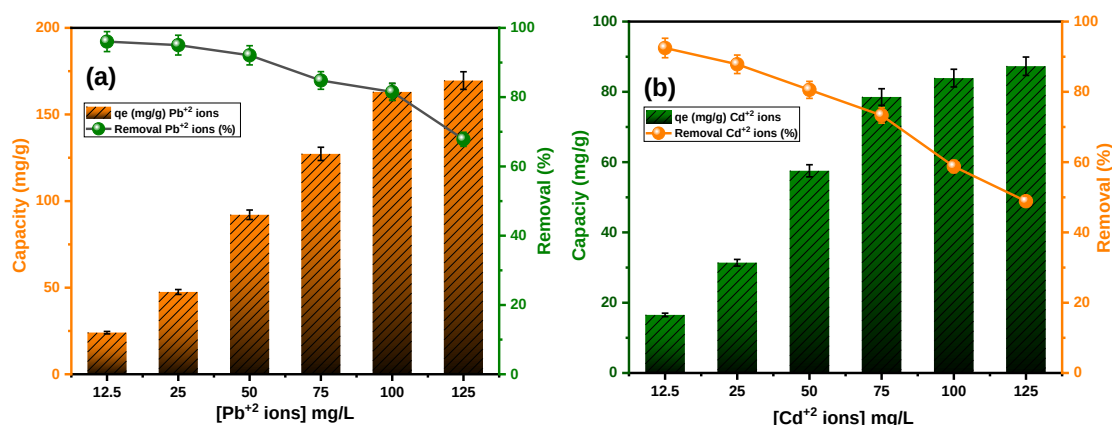


Figure 10. Effect of metal-ion concentration on the removal percentage and capacity for (a) Pb^{2+} and (b) Cd^{2+} ions.

In order to interpret the interactions between Pb^{2+} or Cd^{2+} ions and $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ adsorbent, experimental equilibrium data were analyzed with the various adsorption isotherm models, including the Langmuir, Freundlich, Temkin, and Dubinin–Radushkevich (D-R) models, as described by Equations (8)–(11) [59–61].

Langmuir:	$q_e = \frac{q_m K_L C_e}{(1 + K_L C_e)}$	(8)
Freundlich:	$q_e = K_F C_e^{1/n}$	(9)
Temkin	$q_e = \frac{RT}{b} \ln(K_T C_e)$	(10)
Dubinin–Radushkevich (D-R):	$q_e = q_m \exp[-\beta(RT \ln(1 + \frac{1}{C_e}))^2],$ $E = \frac{1}{(2\beta)^{0.5}}$	(11)

In these models, C_e denotes the equilibrium concentration of metal ions remaining in solution (mg/L), while q_m represents the maximum adsorption capacity predicted by the Langmuir and Dubinin–Radushkevich (D–R) isotherms (mg/g). The K_L parameter corresponds to the Langmuir adsorption constant (L/mg), and K_F is the Freundlich adsorption constant (mg/g). K_T refers to the Temkin equilibrium binding constant, whereas (b) is associated with the adsorption energy or potential. (R) represents the universal gas constant (J/mol·K), and (T) is the absolute temperature (K). β denotes the D–R isotherm activity coefficient, expressed in mol^2/J^2 .

The data of adsorption equilibrium of Pb^{2+} and Cd^{2+} ions on $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite was analyzed by the nonlinear terms of the selected isotherm models, as shown in Figure 11a,b. The respective isotherm coefficients and fitting parameters are summarized in Table 4. The model fittings show that the Langmuir isotherm offers the most remarkable explanation of experimental data for both metal ions. This is justified by greater correlation coefficients (R^2) using the Langmuir model compared to the other models, and the correlation coefficients are close to unity, meaning that there is a perfect match between the theoretical assumptions of monolayer adsorption and experimental equilibrium results. The strong agreement between the experimental equilibrium data and the Langmuir isotherm model suggests that Pb^{2+} and Cd^{2+} adsorption on the $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite takes place on a homogeneous surface containing energetically identical active sites, which supports a monolayer adsorption mechanism. This behavior further implies that the adsorption process involves physicochemical interactions [62,63]. In addition, the Langmuir model presupposes a limited number of free adsorbing sites, which means that the ultimate adsorption capacity of a material is determined by the occupancy of the sites on the adsorbing surface [64]. The values of the dimensionless separation factor (R_L) are in the range of 0–1, which proves that the adsorption of both metal ions on the $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ surface is favorable within the studied conditions. The adsorption capacity was studied systematically because this value is an important parameter for determining both the performance of the adsorbent and its economic feasibility. The highest adsorption capacities for Pb^{2+} and Cd^{2+} reached 187.07 and 96.45 mg/g, respectively, using the $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite. To better evaluate the adsorption ability of $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$, the obtained maximum adsorption capacities were compared with those of previously published adsorbents, as summarized in Table 5 [65–73]. The comparison indicates that the magnetic $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite exhibits higher adsorption capacities than many reported adsorbents. This superior performance can be attributed to the synergistic effect of activated carbon, which provides high porosity and surface area, SnFe_2O_4 nanoparticles that contribute active adsorption sites and magnetic recoverability, and polypyrrole, which introduces nitrogen-containing functional groups that enhance metal-ion binding. These results confirm that the developed composite is a promising candidate for efficient heavy-metal removal from water.

The Freundlich isotherm parameters provide valuable information regarding the adsorption behavior of the $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite. The obtained $1/n$ values for both Pb^{2+} and Cd^{2+} were within the range of 0–1, indicating favorable adsorption and strong interactions between the adsorbent surface and the metal ions [74,75]. Furthermore, the Freundlich constant (K_F), which reflects the adsorption capacity and affinity of the adsorbent, was found to be 52.31 mg/g for Pb^{2+} and 25.66 mg/g for Cd^{2+} . The higher K_F value observed for Pb^{2+} suggests a stronger affinity of the composite toward lead ions compared with cadmium ions [75]. Overall, the relatively high K_F values confirm the effectiveness of the $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite as a promising adsorbent for the removal of heavy metal ions from aqueous solutions, owing to its abundant active sites and strong binding capability.

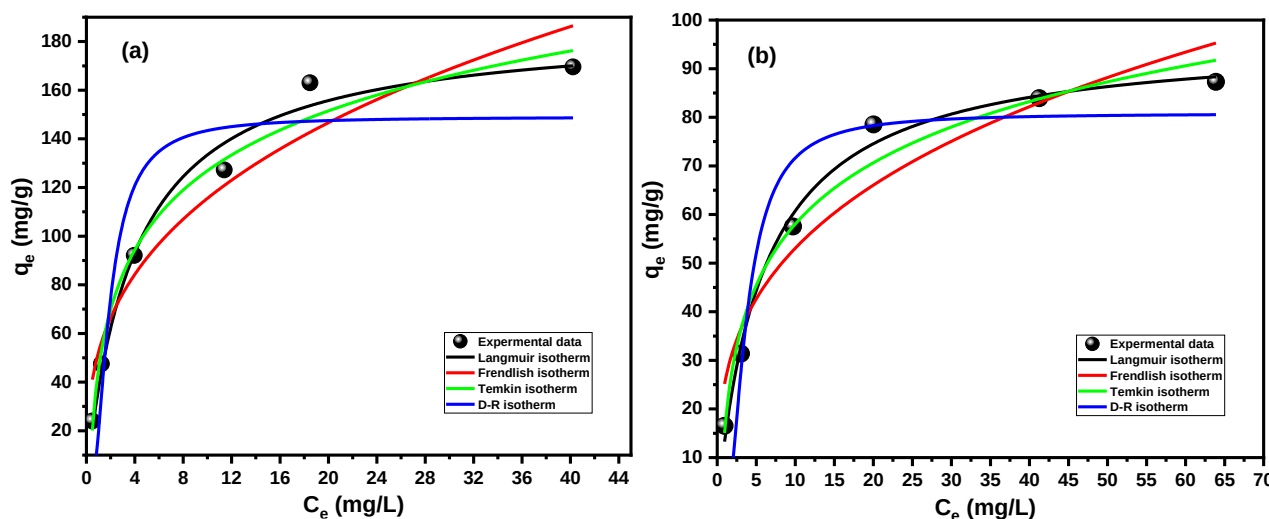


Figure 11. Nonlinear isotherm models for the adsorption of (a) Pb^{2+} and (b) Cd^{2+} ions by $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ ternary composite.

Table 4. Selected isotherm parameters for the adsorption of Pb^{2+} and Cd^{2+} ions using $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ ternary composite.

Model	Parameter	Pb^{2+}	Cd^{2+}
Langmuir	K_L (L/mg)	0.248	0.170
	$q_{\max}$ (mg/g)	187.07	96.45
	Adjusted R^2	0.987	0.992
	R^2	0.983	0.989
Freundlich	$1/n$	0.343	0.315
	K_F (mg/g)	52.31	25.66
	Adjusted R^2	0.934	0.921
	R^2	0.918	0.901
Temkin	B_T (J/mol)	35.50	18.16
	K (L/mg)	1.105	1.143
	Adjusted R^2	0.982	0.975
	R^2	0.978	0.968
Dubinin–Radushkevich (D-R)	q_s (mg/g)	148.99	80.78
	E (KJ/mol)	1.089	0.614
	β (mol^2/J^2) $\times 10^{-7}$	4.21	13.24
	Adjusted R^2	0.861	0.879
	R^2	0.827	0.848

The Dubinin–Radushkevich (D–R) isotherm model was employed to evaluate the adsorption energy associated with the uptake of Pb^{2+} and Cd^{2+} ions. The calculated mean free adsorption energy (E) values were 1.089 and 0.614 kJ/mol for Pb^{2+} and Cd^{2+} , respectively. Since both values are considerably lower than 8 kJ/mol, the adsorption process is predominantly governed by physical interactions, such as weak van der Waals forces and electrostatic attractions, rather than chemical bond formation [76,77]. These findings suggest that physisorption plays a significant role in the removal of both metal ions by the $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite. Nevertheless, when considered together with the kinetic and equilibrium isotherm analyses, the overall adsorption mechanism can be characterized as a physicochemical process involving the combined contribution of physical adsorption and surface interactions at the active adsorption sites [78,79].

Table 5. Comparative adsorption capacities of SnFe₂O₄/AC/PPy composite and various adsorbents for Cd²⁺ and Pb²⁺ removal from wastewater.

Adsorbents	Adsorption Capacity (mg/g)		Reference
	Pb ²⁺	Cd ²⁺	
MnFe ₂ O ₄ nanocomposite (BC/FM)	154.94	127.83	[65]
Magnetic biochar composite (MBC)	142.00	125.00	[66]
Fe ₃ O ₄ -CS composite	23.75	12.38	[67]
Fe ₃ O ₄ @SiO ₂ -NH ₂ /GO nanocomposite	13.46	18.58	[68]
Hardwood composite (HWC)	207.90	119.60	[69]
Activated carbon	9.30	9.26	[70]
PPy/Fe ₃ O ₄ composite	11.35	8.92	[71]
r-GO/PPy/SiO ₂ composite	19.30	--	[72]
Fe ₃ O ₄ @SiO ₂ @PPy-PANI magnetic nanocomposite	98.00	84.00	[73]
SnFe ₂ O ₄ /AC/PPy composite	187.07	96.45	This work

3.5. Adsorption Thermodynamics

The temperature dependence of Pb²⁺ and Cd²⁺ ions adsorption behavior on the SnFe₂O₄/AC/PPy composite was systematically studied at a temperature range of 25 to 45 °C under the same conditions (concentration of both metal ions: 12.5 mg/L, pH for Pb²⁺ and Cd²⁺ solution = 6 and 7, respectively, adsorbent mass for Pb²⁺ and Cd²⁺: 25 and 35 mg, respectively). Figure 12a,b indicate that the adsorption capacity and the removal efficiency reduced gradually as the temperature increased. In particular, the adsorption capacity of Pb²⁺ decreased from 24.01 to 22.02 mg/g, while that of Cd²⁺ declined from 16.51 to 14.67 mg/g as the temperature increased from 25 to 45 °C. On the same note, the percentage of elimination decreased from 96.05% to 88.07% for Pb²⁺ and from 92.48% to 82.17% for Cd²⁺, corresponding to reductions of approximately 7.98% and 10.31%, respectively. Interestingly, the greater reduction observed for Cd²⁺ compared to Pb²⁺ suggests that Cd²⁺ adsorption is more sensitive to temperature variations. This behavior may be attributed to the relatively weaker binding affinity of Cd²⁺ toward the active adsorption sites of the SnFe₂O₄/AC/PPy composite, whereas Pb²⁺ tends to form more stable surface complexes with oxygen- and nitrogen-containing functional groups. As a result, increasing temperature promotes the desorption of Cd²⁺ ions more readily than Pb²⁺ ions, leading to a larger decrease in adsorption capacity and removal efficiency. These findings suggest that low temperatures are more suitable for effective metal-ion uptake. The decrease in adsorption performance as observed at high temperatures implies that the adsorption of Pb²⁺ and Cd²⁺ onto the SnFe₂O₄/AC/PPy composite is an exothermic reaction. This behavior may be caused by a number of factors, such as the weakening of van der Waals forces between metal ions and surface binding sites, enhanced propensity of desorption and release of ions to the solution, and potential changes in structure or morphology of the adsorbent that decrease the stability of active sites [80–82]. Moreover, increasing temperatures improves the mobility of metal ions and decreases the thickness of the boundary layer around the adsorbent particles, which allows ions to escape from the surface and lowers the adsorption efficiency [83].

In order to further understand the thermodynamic character of the adsorption process, the standard thermodynamic parameters were calculated based on the use of Equations (12) and (13). In the equations, R stands for the universal gas constant (8.314 J/mol·K), T for the absolute temperature (K), K_d for the distribution coefficient which is determined by using the formula $K_d = (q_e/C_e)$, where q_e (mg/g) represents the quantity of adsorbed metal ions at equilibrium, and C_e (mg/L) is the corresponding equilibrium concentration of metal ions in solution. The slope and the interception of the

Van't Hoff plot (Figure 12c) were used to determine the values of the Gibbs free energy change (ΔG°), enthalpy change (ΔH°), and entropy change (ΔS°); the obtained values are summarized in Table 6.

$$\ln K_d = \frac{\Delta S^\circ}{R} - \frac{\Delta H^\circ}{RT} \quad (12)$$

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ \quad (13)$$

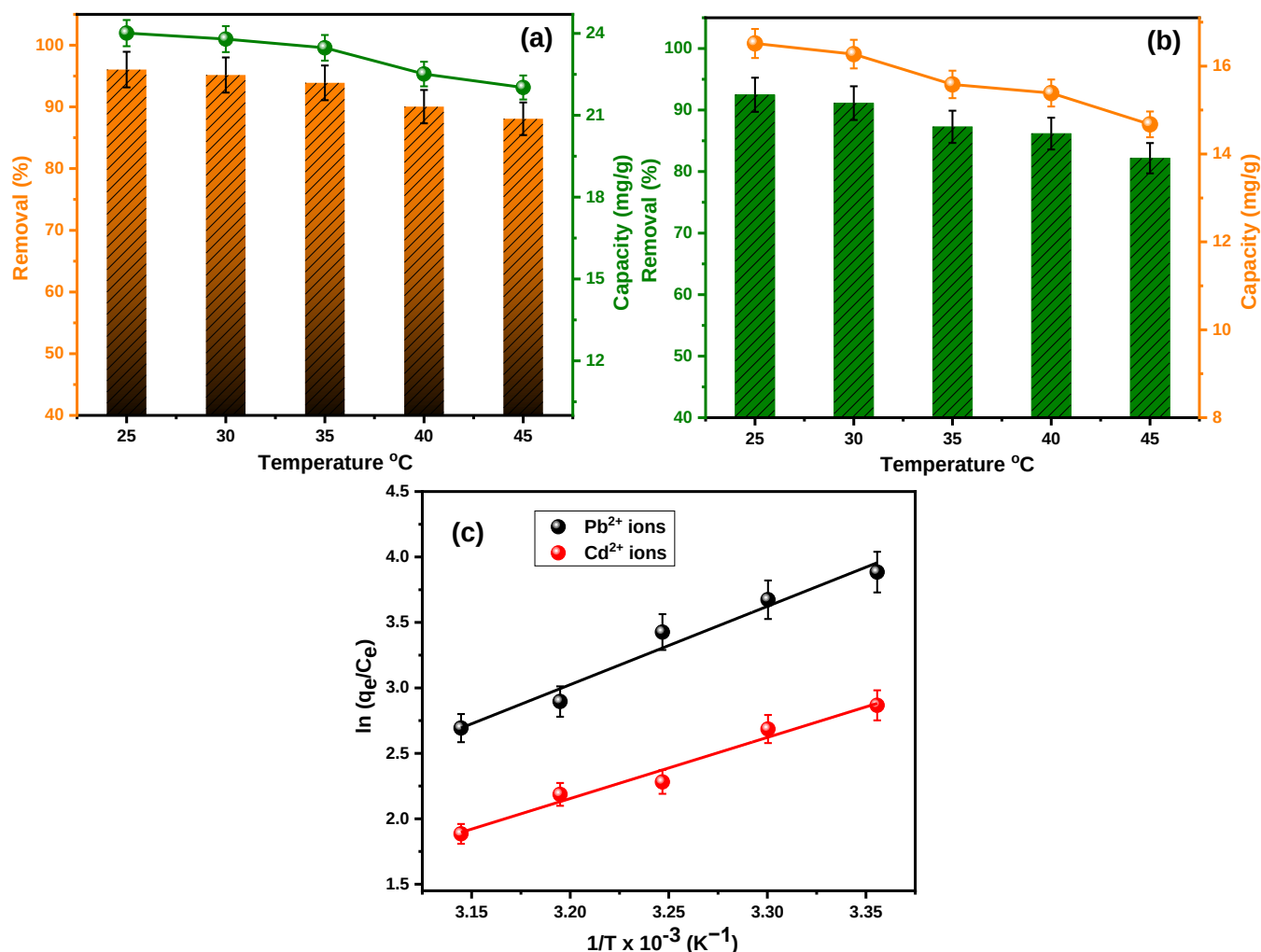


Figure 12. Effect of temperature on adsorption efficiency and capacity of (a) Pb²⁺ and (b) Cd²⁺ ions, and (c) linear relationship of $\ln(q_e/C_e)$ vs. $1/T$.

Table 6. Thermodynamic parameters for Pb²⁺ and Cd²⁺ ion adsorption by SnFe₂O₄/AC/PPy composite.

Metal Ions	Temperature (°C)	ΔG° (kJ/mol)	ΔH° (kJ/mol)	ΔS° (J/mol · K)
Pb ²⁺	25	−9.81	−49.74	−134.03
	30	−9.13		
	35	−8.46		
	40	−7.79		
	45	−7.12		
Cd ²⁺	25	−7.33	−38.82	−106.31
	30	−6.61		
	35	−6.07		
	40	−5.54		
	45	−5.01		

The negative values of ΔS° indicate that the degree of randomness at the solid-solution interface is reduced during the adsorption of the ions, indicating a more ordered arrangement of Pb^{2+} and Cd^{2+} ions on the adsorbent surface [84]. The enthalpy values (ΔH°) were found to be -49.74 kJ/mol in the case of Pb^{2+} and -38.82 kJ/mol in the case of Cd^{2+} , which confirmed that the adsorption process was exothermic [85]. In addition, ΔH° values ranged from 25 to 80 kJ/mol, suggesting that physicochemical involvement in the adsorption [86–88], which is consistent with the kinetic analysis and Dubinin–Radushkevich isotherm results. Additionally, the ΔG° values of both metal ions are negative, which also indicates that the adsorption is spontaneous, and their values decrease with a rise in temperature, without exceeding 20 kJ/mol, indicating that the adsorption process is more favorable at low temperatures [89].

3.6. Effect of Interfering Ions, Regeneration Study

The presence of competing ions in the aqueous medium has a significant impact on the adsorption capacity of adsorbent materials for metal ions. In real-life applications, industrial effluents consist of more than one kind of ion that may compete with target ions for adsorption sites. The adsorption efficiency of adsorbent materials in the presence of these ions should be tested. In that context, the interference of common cations (Na^+ , K^+ , Mg^{2+} , and Ca^{2+}) and anions (Cl^- , NO_3^- , and SO_4^{2-}) to adsorb lead and cadmium ions at a concentration of 12.5 mg/L by the $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite adsorbent was explored. In the process, the interfering ions and either lead or cadmium ions were separately added to an aqueous medium, and then the adsorbent materials were added to the medium along with stirring for 180 min at 25 °C. At the end of the process, the concentration of lead and cadmium ions left in the medium was analyzed. As presented in Figure 13a,b, the addition of monovalent cations such as Na^+ and K^+ displayed very slight effects on the adsorption performance of Pb^{2+} and Cd^{2+} . The weak interference can be attributed to the lower charge density and stronger hydration that resulted in low competition with heavy metal ions for the active sites of the composite. In contrast, Mg^{2+} and Ca^{2+} divalent cations inhibited the adsorption process to a greater extent due to their higher charge and thus strong electrostatic attraction on the adsorbent surface. This suggests that $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ tends to preferentially interact with metal ions possessing higher valence [90,91]. Among the divalent ions, Ca^{2+} resulted in a more substantial decrease in the efficiency of adsorption compared to Mg^{2+} , which can be attributed to its lower hydrated ionic radius. In general, adsorbed ions with smaller hydrates will have a better opportunity to move towards the binding points of adsorption [92]. Additionally, Mg^{2+} exhibits higher hydration enthalpies (-1830 kJ/mol) than Ca^{2+} (-1505 kJ/mol), indicating higher binding affinities of Mg^{2+} to water molecules, thus reducing the competitive abilities of these metal ions towards the binding sites than Pb^{2+} and Cd^{2+} [93]. Moreover, it can be seen that Ca^{2+} has a more pronounced inhibitory effect on Cd^{2+} than on Pb^{2+} . This can be explained by differences in the value of the hydration radius between these ions. It is reported that Ca^{2+} has a smaller hydration radius than Cd^{2+} but a larger value than Pb^{2+} [94]. Among ions of identical charge, as the value of the hydration radius reduces, ion exchange and competitive adsorption mechanisms are more likely. Hence, the competition of Ca^{2+} ions with Cd^{2+} for placement on the active sites of the adsorbent is greater compared to Pb^{2+} .

Regarding the effects of anions, Figure 13a,b reveal that Cl^- and NO_3^- had no interference effects on the adsorption of Pb^{2+} and Cd^{2+} , whereas SO_4^{2-} decreased the adsorption capacity significantly for both metal ions. These effects may be related to the affinity of the sulfate ion to form inner- and outer-sphere complexes with the surface functional groups of the adsorbent material [95]. On the other hand, the negative charge on the surface of the composite material causes electrostatic repulsion to the anions, thus indirectly contributing

to the adsorption of the positively charged Pb^{2+} and Cd^{2+} ions [96]. Nevertheless, the affinity of the SO_4^{2-} ion significantly decreases the adsorption capacity by reducing the number of available adsorption sites on the adsorbent material surfaces. Therefore, in the presence of competing ions, the composite material $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ showed a high capability for the adsorption process. This verifies that the composite material, $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$, possesses a high potential for the effective removal of Pb^{2+} and Cd^{2+} ions from a complex system of wastewater containing other ions.

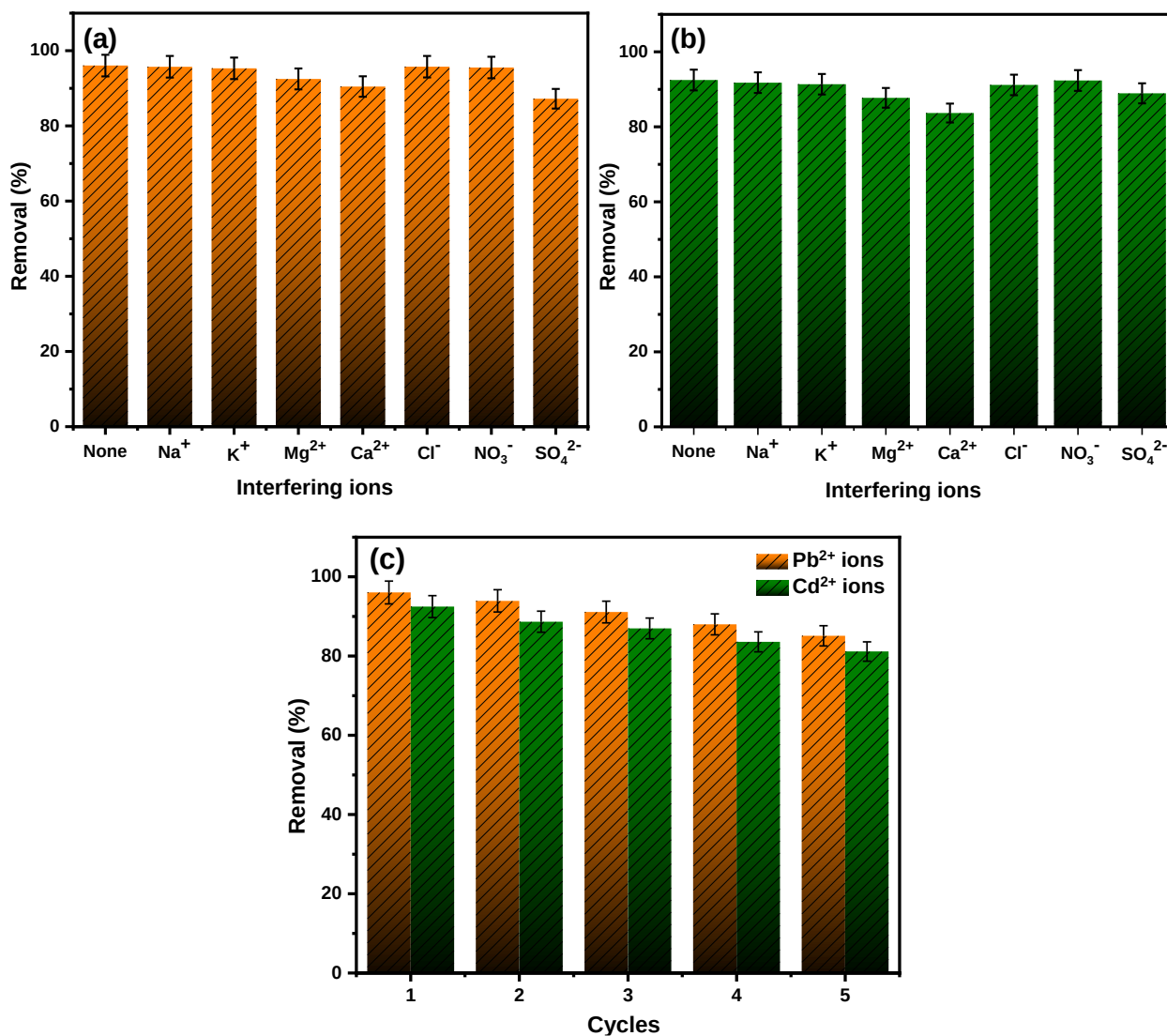


Figure 13. The impact of interference cations and anions on the adsorption of (a) Pb^{2+} , (b) Cd^{2+} ions, and (c) the reusability study of $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ ternary composite in the removal of Pb^{2+} and Cd^{2+} ions.

The reusability and regeneration of an adsorbent are among the most important considerations for evaluating its practical convenience and economic feasibility in wastewater treatment [97]. In this context, the reusability of the $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite was systematically checked under controlled experimental conditions (25 °C, initial metal ion concentration 12.5 mg/L for both metal ions, pH 6 for Pb^{2+} and pH 7 for Cd^{2+} , contact time of 180 min, and adsorbent dosages of 25 mg for Pb^{2+} and 35 mg for Cd^{2+}). Repeated adsorption–desorption experiments were performed with 0.1 M HCl as the regenerant. As shown in Figure 13c, the removal efficiencies of Pb^{2+} and Cd^{2+} gradually decreased over five consecutive adsorption–desorption cycles, from 96.05% to 85.11% and from 92.48%

to 81.15%, respectively. The overall reduction in removal efficiency after five cycles was approximately 10.94% for Pb^{2+} and 11.33% for Cd^{2+} . This decrease can be attributed mainly to the incomplete desorption of metal ions and the gradual coverage of active sites rather than permanent deactivation of the adsorbent [98]. Nevertheless, the composite retained high removal efficiencies after repeated use, demonstrating its good regeneration capability and stability. Notably, the magnetic nature of the $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite enabled its rapid and efficient separation from the aqueous phase using an external magnetic field, eliminating the need for filtration or centrifugation and facilitating easy recovery of the adsorbent. This magnetic recoverability minimizes material loss during adsorption–desorption cycles, simplifies the regeneration process, and enhances the practical applicability and economic feasibility of the composite for water treatment applications. To further investigate the cause of the slight performance decline, post-regeneration characterization was conducted after five adsorption–desorption cycles. XRD analysis revealed that the characteristic diffraction peaks of the composite remained essentially unchanged, with no significant shifts in peak positions or evidence of structural deterioration. These results confirm that the crystalline structure of the composite was preserved throughout the regeneration process. Therefore, the observed decrease in adsorption efficiency is primarily associated with residual adsorbed ions and partial occupation of active adsorption sites rather than structural degradation of the adsorbent. Furthermore, satisfactory regeneration performance suggests that adsorption is predominantly governed by reversible surface interactions, including surface complexation and electrostatic attraction, while irreversible pore-filling effects play a secondary role. These findings demonstrate the excellent structural stability, magnetic recoverability, and reusability of the $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite for repeated heavy-metal removal applications.

3.7. The Proposed Adsorption Mechanism

Studying the mechanism of heavy metal adsorption provides essential information about the interactions between the $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite and $\text{Pb}^{2+}/\text{Cd}^{2+}$ ions. It shows the role of parameters and influencing factors in the removal process. In general, various mechanisms such as physical adsorption, chemical adsorption, electrostatic interactions, hydrogen bonds, redox interactions, complexation, ion exchange, chemical precipitation, and sorption by pores are involved in the adsorption of heavy metals [99].

To gain deeper insight into the specific adsorption pathways, comprehensive post-adsorption characterizations were performed for $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite. The surface morphology of $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ was examined using SEM–EDS after Pb^{2+} and Cd^{2+} uptake, as displayed in Figure 4. The SEM images demonstrate that there is an observable difference in surface texture of the composite, which has become rougher and partially covered, and this is an indication that there is an effective interaction between metal ions and available adsorption sites Figure 4a,c. The clear presence of Pb (4.02 wt.%) and Cd (1.35 wt.%) in the respective EDS spectra indicates the successful uptake of heavy metal ions, as shown in Figure 4b,d. These findings highlight the cooperative role of the porous architecture, PPy coating, and magnetic SnFe_2O_4 nanoparticles in enhancing overall adsorption capacity. Meanwhile, the XRD patterns of the $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite before and after Pb^{2+} and Cd^{2+} adsorption (Figure 14a) exhibit the characteristic diffraction peaks without the appearance of new crystalline phases. Although slight changes in diffraction peak intensities were observed after adsorption, no significant shifts in peak positions occurred, indicating that the crystal structure of the composite remained largely unchanged during the adsorption process. The observed intensity variations may be attributed to the adsorption of Pb^{2+} and Cd^{2+} ions on the composite surface and their interaction with the active adsorption sites. The preservation of the characteristic diffraction peaks demon-

Further evidence from FTIR spectroscopy confirms the active participation of diverse surface functional groups (Figure 14b). Specifically, the broad band corresponding to O–H and N–H stretching, originally centered at 3439 cm^{-1} , undergoes a shift to 3429 cm^{-1} for Pb^{2+} and 3447 cm^{-1} for Cd^{2+} , accompanied by a marked decrease in intensity. This behavior unequivocally demonstrates the involvement of hydroxyl and amine moieties in metal binding via surface complexation and electrostatic attraction. Additionally, the band at 1632 cm^{-1} , attributed to overlapping C=O and C=C stretching vibrations from activated carbon and polypyrrole, shifted after adsorption. These changes suggest that Pb^{2+} and Cd^{2+} ions interact with oxygen-containing functional groups on the composite surface. The observed variation in the C=C-related vibration may also indicate possible interactions between the metal ions and the π -electron-rich structure of activated carbon and polypyrrole. Characteristic polypyrrole bands at 1548 , 1453 , and 1382 cm^{-1} (attributed to C=C and C–N vibrations of the pyrrole ring) exhibit both shifts and intensity attenuation, confirming that nitrogen sites actively participate through electrostatic interactions and metal–ligand coordination. The bands corresponding to C–N and H–N stretching at 1168 and 1037 cm^{-1} also show a shift to lower frequencies after adsorption, thus confirming the formation of inner-sphere surface complexes. In addition, variations in the aromatic and pyrrole ring out-of-plane deformation bands at 875 and 776 cm^{-1} after adsorption suggest that the aromatic structures of activated carbon and polypyrrole may participate in the adsorption process. These changes are consistent with the possible contribution of cation– π interactions between $\text{Pb}^{2+}/\text{Cd}^{2+}$ ions and π -electron-rich regions of the composite. The slight shift in wavenumber and decrease in intensity of the Fe–O stretching band at 589 cm^{-1} after adsorption confirm weak interactions with ferrite surfaces while maintaining structural characteristics of SnFe_2O_4 . Therefore, the presence of functional groups (–OH/–NH–, O–H, C–N, C=O, –COOH, C=C, and Fe–O) in the composite was an important factor in $\text{Pb}^{2+}/\text{Cd}^{2+}$ ion adsorption on $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite materials. Collectively, the FTIR spectral analysis confirms that Pb^{2+} and Cd^{2+} adsorption proceeds via synergy among surface complexation, electrostatic attraction, and cation– π bonding.

In terms of electrostatic contributions, the adsorption process is significantly influenced by solution pH. The composite exhibits a point of zero charge (pH_{pzc}) of 4.45. At pH values exceeding this threshold, the adsorbent surface becomes negatively charged, thereby promoting electrostatic attraction toward positively charged Pb^{2+} and Cd^{2+} species. The optimal removal conditions are observed at pH 6 for Pb^{2+} and pH 7 for Cd^{2+} . According to the Hard and Soft Acids and Bases (HSAB) theory, the –OH and –NH groups on the composite behave as hard bases, showing favorable interactions with borderline (Pb^{2+}) and soft (Cd^{2+}) acid cations. Physical adsorption may also contribute to the overall adsorption process through pore filling within the porous structure of the composite. However, considering the significant decrease in BET surface area and pore volume, coupled with the increase in average pore diameter after PPy coating (Table 2), pore filling is likely a secondary adsorption pathway. The increase in average pore diameter suggests that smaller pores were preferentially covered or partially filled by the polymer layer, leaving relatively larger pores accessible. Therefore, the enhanced adsorption performance is primarily attributed to surface complexation and electrostatic interactions associated with the functional groups introduced by PPy, whereas pore filling serves as a complementary adsorption and secondary adsorption mechanism for Pb^{2+} and Cd^{2+} uptake. Kinetic analysis reveals that the adsorption data conform to the pseudo-second-order (PSO) model, indicating that chemisorption steps such as hydrogen bonding and complex formation control the overall uptake rate. Furthermore, the Dubinin–Radushkevich (D–R) isotherm yields an adsorption energy ($E_{\text{D-R}}$) for Pb^{2+} ions were lower than 8 kJ/mol , suggesting a physical adsorption

mechanism. Altogether, the dominant mechanisms include coordination/complexation, electrostatic attraction, pore filling, and cation- π coordination.

To further expound the detailed mechanism of $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ in water purification, the elemental composition and chemical state of $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ before and after $\text{Pb}^{2+}/\text{Cd}^{2+}$ adsorption were illustrated via XPS analysis (Figure 14c–e). The survey spectrum exhibits well-resolved peaks at 285, 400, 531, 486, and 710 eV, corresponding to C 1s, N 1s, O 1s, Sn 3d, and Fe 2p, thereby corroborating the successful incorporation of activated carbon, polypyrrole, and SnFe_2O_4 into the composite architecture. Upon Pb^{2+} and Cd^{2+} uptake, slight shifts in the binding energies of C, N, and O are detected, implying direct electronic perturbations arising from metal–surface coordination. Notably, the high-resolution Pb 4f spectrum exhibits two characteristic peaks at 138.4 and 143.7 eV, corresponding to Pb 4f_{7/2} and Pb 4f_{5/2}, respectively. These peaks are consistent with the interaction between Pb^{2+} ions and oxygen- and nitrogen-containing functional groups on the composite surface [100]. Similarly, the high-resolution Cd 3d spectrum displays two characteristic peaks at 405.8 and 412.5 eV, assigned to Cd 3d_{5/2} and Cd 3d_{3/2}, respectively. The presence of these peaks suggests coordination interactions between Cd^{2+} and the O- and N-containing functional groups of the composite [101,102]. Collectively, these XPS findings offer compelling evidence that Pb^{2+} and Cd^{2+} ions are chemically immobilized onto the composite surface via specific binding with oxygen and nitrogen-rich functional groups. Therefore, the obtained results suggest that the adsorptive removal of Pb^{2+} and Cd^{2+} by the $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite is primarily governed by complexation with nitrogen- and oxygen-containing functional groups, electrostatic attraction, and pore filling, while cation- π interactions may also contribute to the overall adsorption process (Figure 15).

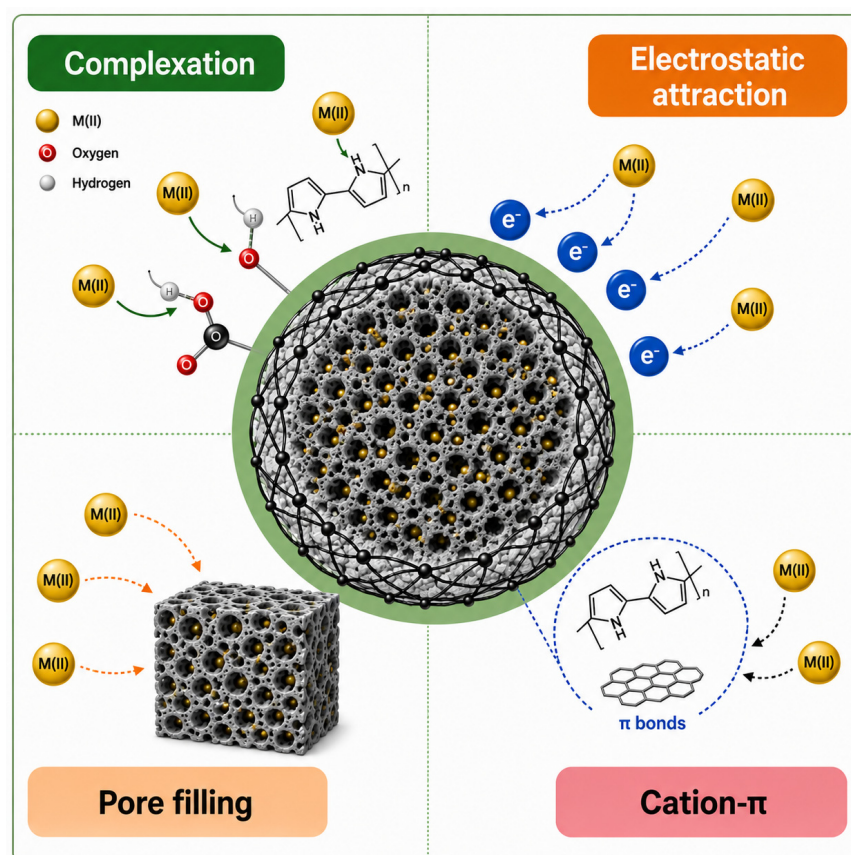


Figure 15. The adsorption mechanism of Pb^{2+} and Cd^{2+} ions by $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite.

4. Conclusions

A novel $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ magnetic composite effectively removes Pb^{2+} and Cd^{2+} ions from aqueous solutions. Various characterization techniques, such as FTIR, XRD, SEM, BET, XPS, and VSM, verified the successful formation of a porous hybrid structure with a certain surface area of $352.3 \text{ m}^2/\text{g}$ and a sufficient amount of magnetic saturation to perform a rapid magnetic separation. The paper examined its adsorption properties using kinetic, isotherm, and thermodynamic analysis. The effects of various operating parameters, such as solution pH, contact time, initial metal concentration, adsorbent dose, and temperature, were studied to find out the optimum conditions for sorption. The maximum removal efficiencies were obtained as 95.02% for Pb^{2+} and 92.48% for Cd^{2+} ions. Kinetic experiments showed that the pseudo-second-order (PSO) model fitted the experimental data, indicating that the adsorption was governed by surface interactions between the metal ions and the active sites of the composite. The Langmuir isotherm model, which is based on monolayer adsorption onto a homogeneous surface, described equilibrium data with maximum adsorption capacities of 187.07 mg/g for Pb^{2+} and 96.45 mg/g for Cd^{2+} . Dubinin–Radushkevich (D–R) model analysis of the data revealed that the mean adsorption energies were less than 8 kJ/mol ; thus, the uptake process was favored by physical interactions with chemical contribution. The thermodynamic parameters indicated that the adsorption of both metal ions was spontaneous and exothermic, as indicated by negative values of Gibbs free energy and enthalpy. Furthermore, regeneration and reusability investigations revealed that the $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ composite maintained a high removal efficiency after five sequential adsorption–desorption cycles, indicating its structural stability and recyclability. Overall, the results show that the synthesized $\text{SnFe}_2\text{O}_4/\text{AC}/\text{PPy}$ magnetic composite is a highly efficient, reusable, and promising adsorbent for removing heavy metals from contaminated water systems.

Author Contributions: Conceptualization, M.M.Y.; methodology, M.M.Y. and M.W.; validation, M.W.; formal analysis, M.M.Y. and M.M.M.; investigation, M.M.Y. and M.W.; data curation, M.M.Y. and M.M.M.; writing—original draft preparation, M.M.Y.; writing—review and editing, M.W.; supervision, M.W. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by the Excellence Initiative–Research University (IDUB) program and a scientific research subsidy of AGH University of Krakow, Project number 18004.

Data Availability Statement: The original contributions presented in this study are included in the article. Further inquiries can be directed to the corresponding authors.

Acknowledgments: BET analysis and porosity studies using adsorption methods were conducted by Kamil Kornaas at the Departmental Laboratory of Microstructural Studies, KCiMO, WiMiC, AGH University of Krakow.

Conflicts of Interest: The authors declare no conflicts of interest.

References

1. Sharma, M.; Kant, R.; Sharma, A.K.; Sharma, A.K. Exploring the impact of heavy metals toxicity in the aquatic ecosystem. *Int. J. Energy Water Resour.* **2025**, *9*, 267–280. [\[CrossRef\]](#)
2. Kaur, S.; Roy, A. Bioremediation of heavy metals from wastewater using nanomaterials. *Environ. Dev. Sustain.* **2021**, *23*, 9617–9640. [\[CrossRef\]](#)
3. Mani, D.; Kumar, C. Biotechnological advances in bioremediation of heavy metals contaminated ecosystems: An overview with special reference to phytoremediation. *Int. J. Environ. Sci. Technol.* **2014**, *11*, 843–872. [\[CrossRef\]](#)
4. Younas, H.; Nazir, A.; Bareen, F.e. From sources to solutions: Integrated approaches for Cd, Hg, and Pb remediation—A comprehensive review. *Plant Soil* **2025**, *510*, 1–47. [\[CrossRef\]](#)
5. Ali, S.; Naseer, S.; Rehman, M.; Wei, Z. Recent trends and sources of lead toxicity: A review of state-of-the-art nano-remediation strategies. *J. Nanopart. Res.* **2024**, *26*, 168. [\[CrossRef\]](#)

6. Ramírez Ortega, D.; González Esquivel, D.F.; Blanco Ayala, T.; Pineda, B.; Gómez Manzo, S.; Marcial Quino, J.; Mora, P.C.; de la Cruz, V.P. Cognitive Impairment Induced by Lead Exposure during Lifespan: Mechanisms of Lead Neurotoxicity. *Toxics* **2021**, *9*, 23. [[CrossRef](#)] [[PubMed](#)]
7. Billah, R.E.K.; Islam, M.A.; Khan, M.A.; Nazal, M.K.; Bahsis, L.; Achak, M.; Mahmoud, A.E.D.; Aziz, A.; Jeon, B.-H. Adsorption of lead(II) onto magnetic chitosan@calcium phosphate rock biocomposite. *Mater. Chem. Phys.* **2025**, *332*, 130249. [[CrossRef](#)]
8. Obaid, A.O.; Albishri, A.; Sallam, S.; Alharbi, M.M.; Alkhamis, K.M.; Alaysuy, O.; Alatawi, N.M.; El-Metwaly, N.M. Tailored glutamine modified nanozeolite-Y/xanthan gum composite based on rice straw for enhanced adsorption of cadmium ions from aqueous media. *Process Saf. Environ. Prot.* **2025**, *196*, 106965. [[CrossRef](#)]
9. Gusain, D.; Sahani, S.; Sharma, Y.C.; Han, S.S. Adsorption of cadmium ion from water by using non-toxic iron oxide adsorbent; experimental and statistical modelling analysis. *Int. J. Environ. Sci. Technol.* **2025**, *22*, 1417–1432. [[CrossRef](#)]
10. Mubarak, A.S.; Salih, S.S.; Kadhom, M.; Ghosh, T.K. Competitive and non-competitive adsorption of Cd(II) and Pb(II) from aqueous solution using Zr-BADS metal organic frameworks. *Sustain. Chem. Environ.* **2025**, *9*, 100231. [[CrossRef](#)]
11. U.S. Environmental Protection Agency (EPA). *Edition of the Drinking Water Standards and Health Advisories*; EPA: Washington, DC, USA, 2012.
12. Bakhtiari, S.; Salari, M.; Shahrashoub, M.; Zeidabadinejad, A.; Sharma, G.; Sillanpää, M. A Comprehensive Review on Green and Eco-Friendly Nano-Adsorbents for the Removal of Heavy Metal Ions: Synthesis, Adsorption Mechanisms, and Applications. *Curr. Pollut. Rep.* **2024**, *10*, 1–39. [[CrossRef](#)]
13. Li, S.; Li, X.; Li, S.; Xu, P.; Liu, Z.; Yu, S. In-situ preparation of lignin/Fe₃O₄ magnetic spheres as bifunctional material for the efficient removal of metal ions and methylene blue. *Int. J. Biol. Macromol.* **2024**, *259*, 128971. [[CrossRef](#)] [[PubMed](#)]
14. Cameu, G.M.; Almeida, L.; Oliveira, A.P.; Igansi, A.; Jaeschke, D.P.; Silveira, N., Jr.; Paes, R.; Dias, D.; Pinto, L.A.d.A.; Cadaval, T.R.S., Jr. Enhanced Adsorption of Pb(II) and Cd(II) by Activated Carbon Derived from Peach Stones for Efficient Water Decontamination. *Processes* **2025**, *13*, 3064. [[CrossRef](#)]
15. Rostami, M.S.; Khodaei, M.M. Recent advances in chitosan-based nanocomposites for adsorption and removal of heavy metal ions. *Int. J. Biol. Macromol.* **2024**, *270*, 132386. [[CrossRef](#)] [[PubMed](#)]
16. Li, S.; Wang, L.; Lu, P.; Li, Y.; Li, Y.; Wang, Y.; Yao, L.; Qiu, D. Nanoconfined polyethyleneimine in mesoporous MCM-41 silica for heavy metal ions removal. *Sep. Purif. Technol.* **2025**, *353*, 128421. [[CrossRef](#)]
17. Sedaghati-Jamalabad, G.; Bagheri-Mohagheghi, M.M. A study on the structural and optical properties of the SnFe₂O₄ spinel compound as anode electrode in Li ion-battery: The optical and dielectric parameters via synthesis methods. *Opt. Quantum Electron.* **2024**, *56*, 965. [[CrossRef](#)]
18. Jamalabad, G.S.; Mohagheghi, M.M.B. Effect of GO and rGO on the photocatalytic and adsorption properties of SnFe₂O₄ for crystal violet dye removal. *Eur. Phys. J. Plus* **2025**, *140*, 1099. [[CrossRef](#)]
19. Pet, I.; Sanad, M.N.; Farouz, M.; ElFaham, M.M.; El-Hussein, A.; El-sadek, M.S.A.; Althobiti, R.A.; Ioanid, A. Review: Recent Developments in the Implementation of Activated Carbon as Heavy Metal Removal Management. *Water Conserv. Sci. Eng.* **2024**, *9*, 62. [[CrossRef](#)]
20. Aljeboree, A.M.; Alshirifi, A.N.; Alkaim, A.F. Kinetics and equilibrium study for the adsorption of textile dyes on coconut shell activated carbon. *Arab. J. Chem.* **2017**, *10*, S3381–S3393. [[CrossRef](#)]
21. Mahmood, J.; Arsalani, N.; Naghash-Hamed, S.; Hanif, Z.; Geckeler, K.E. Preparation and characterization of hybrid polypyrrole nanoparticles as a conducting polymer with controllable size. *Sci. Rep.* **2024**, *14*, 11653. [[CrossRef](#)] [[PubMed](#)]
22. Tawfeek, A.M.; Jabbour, K.; Abid, A.G.; Nisa, M.U.; Manzoor, S.; Shabbir, B.; Rehman, M.Y.U.; Munawar, T.; Sillanpää, M.; Ashiq, M.N. Sm₂O₃ supported on conducting polymer (polypyrrole) as a highly potent electrocatalyst for water splitting. *J. Sol-Gel Sci. Technol.* **2025**, *115*, 292–305. [[CrossRef](#)]
23. Jia, Y.; Kim, D.-H.; Lee, T.; Kang, S.; Lee, B.W.; Rhee, S.J.; Liu, C. One-pot solvothermal synthesis of magnetic SnFe₂O₄ nanoparticles and their performance in the photocatalytic degradation of chlortetracycline with visible light radiation. *RSC Adv.* **2016**, *6*, 76542–76550. [[CrossRef](#)]
24. Scindia, S.S.; Kamble, R.B.; Kher, J.A. Nickel ferrite/polypyrrole core-shell composite as an efficient electrode material for high-performance supercapacitor. *AIP Adv.* **2019**, *9*, 055218. [[CrossRef](#)]
25. Park, D.E.; Chae, H.S.; Choi, H.J.; Maity, A. Magnetite–polypyrrole core–shell structured microspheres and their dual stimuli-response under electric and magnetic fields. *J. Mater. Chem. C* **2015**, *3*, 3150–3158. [[CrossRef](#)]
26. Zhu, M.; Diao, G. Synthesis of Porous Fe₃O₄ Nanospheres and Its Application for the Catalytic Degradation of Xylenol Orange. *J. Phys. Chem. C* **2011**, *115*, 18923–18934. [[CrossRef](#)]
27. Youssif, M.M.; Kornaus, K.; Wojnicki, M. Magnetic Activated Carbon Functionalized with Polyaniline for Efficient Pb (II) Adsorption from Aqueous Solutions. *Coatings* **2026**, *16*, 259. [[CrossRef](#)]
28. Fahim, A.M.; Hameed, T.A. Greener synthesis of carboxymethyl cellulose connected covalent organic framework/CuFe₂O₄ as a magnetic nanocomposite with structural, optical, magnetic, and computational insights. *Polym. Bull.* **2025**, *82*, 13007–13032. [[CrossRef](#)]

29. Ramírez-Ortega, A.A.; Medina-Llamas, M.; da Silva, R.J.; García-Elías, J.; de Lira-Gómez, P.; Medina-Llamas, J.C.; Chávez-Guajardo, A.E. Synthesis of a maghemite-polypyrrole nanocomposite for the removal of congo red dye from aqueous solutions. *Environ. Nanotechnol. Monit. Manag.* **2021**, *16*, 100597. [\[CrossRef\]](#)
30. Zhang, B.; Wang, J.; Su, X.; Duan, H.; Cai, H.; Wang, J.; Yang, S.; Huo, S. Enhanced microwave absorption properties of epoxy composites containing graphene decorated with core-shell Fe_3O_4 @polypyrrole nanoparticles. *J. Mater. Sci. Mater. Electron.* **2017**, *28*, 12122–12131. [\[CrossRef\]](#)
31. Zhang, M.; Yu, Z.; Yu, H. Adsorption of Eosin Y, methyl orange and brilliant green from aqueous solution using ferroferric oxide/polypyrrole magnetic composite. *Polym. Bull.* **2020**, *77*, 1049–1066. [\[CrossRef\]](#)
32. Shokri, A.; Shayesteh, S.F.; Boustani, K. The role of Co ion substitution in SnFe_2O_4 spinel ferrite nanoparticles: Study of structural, vibrational, magnetic and optical properties. *Ceram. Int.* **2018**, *44*, 22092–22101. [\[CrossRef\]](#)
33. Jia, Y.; Rhee, S.J.; Liu, C. Synthesis of magnetic recoverable g- C_3N_4 /SnFe $_2$ O $_4$ composite with enhanced visible light photocatalytic property. *Mater. Lett.* **2017**, *188*, 338–342. [\[CrossRef\]](#)
34. Sun, Y.-H.; Guan, D.-C.; Wei, J.-L.; Zhang, G.-L.; Nan, J.-M.; Cai, Y.-P. The lithium ions storage property of a novel polyhedral SnFe_2O_4 and Core-shell composite SnFe_2O_4 @carbon or carbon@ SnFe_2O_4 for lithium ion batteries. *Appl. Surf. Sci.* **2020**, *532*, 147396. [\[CrossRef\]](#)
35. Li, B.; Weng, X.; Wu, G.; Zhang, Y.; Lv, X.; Gu, G. Synthesis of Fe_3O_4 /polypyrrole/polyaniline nanocomposites by in-situ method and their electromagnetic absorbing properties. *J. Saudi Chem. Soc.* **2017**, *21*, 466–472. [\[CrossRef\]](#)
36. Tian, X.; Xu, X.; Bo, G.; Su, X.; Yan, J.; Yan, Y. A 3D flower-like Fe_3O_4 @PPy composite with core-shell heterostructure as a lightweight and efficient microwave absorbent. *J. Alloys Compd.* **2022**, *923*, 166416. [\[CrossRef\]](#)
37. Ali, N.S.; Harharah, H.N.; Salih, I.K.; Cata Saady, N.M.; Zendejboudi, S.; Albayati, T.M. Applying MCM-48 mesoporous material, equilibrium, isotherm, and mechanism for the effective adsorption of 4-nitroaniline from wastewater. *Sci. Rep.* **2023**, *13*, 9837. [\[CrossRef\]](#) [\[PubMed\]](#)
38. Rasee, A.I.; Awual, E.; Rehan, A.I.; Hossain, M.S.; Waliullah, R.M.; Kubra, K.T.; Sheikh, C.; Salman, S.; Hasan, N.; Hasan, M.; et al. Efficient separation, adsorption, and recovery of Samarium(III) ions using novel ligand-based composite adsorbent. *Surf. Interfaces* **2023**, *41*, 103276. [\[CrossRef\]](#)
39. Ivanchenko, A.; Soroka, O.; Yelatontsev, D.; Panasenko, V. Heavy metal ions removal from an aqueous solution using an adsorbent derived from walnut shell: Equilibrium, kinetic and thermodynamic studies. *Desalin. Water Treat.* **2025**, *321*, 101048. [\[CrossRef\]](#)
40. Shi, T.; Xie, Z.; Zhu, Z.; Shi, W.; Liu, Y.; Liu, M. Highly efficient and selective adsorption of heavy metal ions by hydrazide-modified sodium alginate. *Carbohydr. Polym.* **2022**, *276*, 118797. [\[CrossRef\]](#) [\[PubMed\]](#)
41. Ge, Q.; Liu, H. Tunable amine-functionalized silsesquioxane-based hybrid networks for efficient removal of heavy metal ions and selective adsorption of anionic dyes. *Chem. Eng. J.* **2022**, *428*, 131370. [\[CrossRef\]](#)
42. Li, X.; Cui, Y.; Du, W.; Cui, W.; Huo, L.; Liu, H. Adsorption Kinetics and Mechanism of Pb(II) and Cd(II) Adsorption in Water through Oxidized Multiwalled Carbon Nanotubes. *Appl. Sci.* **2024**, *14*, 1745. [\[CrossRef\]](#)
43. Park, J.-H.; Ok, Y.S.; Kim, S.-H.; Cho, J.-S.; Heo, J.-S.; Delaune, R.D.; Seo, D.-C. Competitive adsorption of heavy metals onto sesame straw biochar in aqueous solutions. *Chemosphere* **2016**, *142*, 77–83. [\[CrossRef\]](#) [\[PubMed\]](#)
44. Abollino, O.; Aceto, M.; Malandrino, M.; Sarzanini, C.; Mentasti, E. Adsorption of heavy metals on Na-montmorillonite. Effect of pH and organic substances. *Water Res.* **2003**, *37*, 1619–1627. [\[CrossRef\]](#) [\[PubMed\]](#)
45. Huang, X.; Wei, D.; Zhang, X.; Fan, D.; Sun, X.; Du, B.; Wei, Q. Synthesis of amino-functionalized magnetic aerobic granular sludge-biochar for Pb(II) removal: Adsorption performance and mechanism studies. *Sci. Total Environ.* **2019**, *685*, 681–689. [\[CrossRef\]](#) [\[PubMed\]](#)
46. Guo, Z.; Yang, R.; Yang, F.; Sun, L.; Li, Y.; Xu, J. Fabrication of polyethylenimine functionalized magnetic cellulose nanofibers for the sorption of Ni(II), Cu(II) and Cd(II) in single-component and multi-component systems. *Int. J. Biol. Macromol.* **2021**, *184*, 68–78. [\[CrossRef\]](#) [\[PubMed\]](#)
47. An, W.; Liu, Y.; Chen, H.; Sun, X.; Wang, Q.; Hu, X.; Di, J. Adsorption properties of Pb(II) and Cd(II) in acid mine drainage by oyster shell loaded lignite composite in different morphologies. *Sci. Rep.* **2014**, *14*, 11627. [\[CrossRef\]](#) [\[PubMed\]](#)
48. Zhang, M. Lead and cadmium decontamination from water media by CNT/Starch/ Fe_3O_4 , as reclaimable magnetic nanocomposite. *Biomass Convers. Biorefinery* **2025**, *15*, 8487–8500. [\[CrossRef\]](#)
49. Algarni, T.S.; Al-Mohaimeed, A.M.; Al-Odayni, A.-B.; Abduh, N.A.Y. Activated Carbon/ ZnFe_2O_4 Nanocomposite Adsorbent for Efficient Removal of Crystal Violet Cationic Dye from Aqueous Solutions. *Nanomaterials* **2022**, *12*, 3224. [\[CrossRef\]](#) [\[PubMed\]](#)
50. Vasconcelos, H.L.; Fávere, V.T.; Gonçalves, N.S.; Laranjeira, M.C.M. Chitosan modified with Reactive Blue 2 dye on adsorption equilibrium of Cu(II) and Ni(II) ions. *React. Funct. Polym.* **2007**, *67*, 1052–1060. [\[CrossRef\]](#)
51. Zhou, C.; Zhu, H.; Wang, Q.; Wang, J.; Cheng, J.; Guo, Y.; Zhou, X.; Bai, R. Adsorption of mercury (II) with an Fe_3O_4 magnetic polypyrrole-graphene oxide nanocomposite. *RSC Adv.* **2017**, *7*, 18466–18479. [\[CrossRef\]](#)

52. Wang, Y.; Li, J.; Xu, L.; Wu, D.; Li, Q.; Ai, Y.; Liu, W.; Li, D.; Zhou, Y.; Zhang, B.; et al. EDTA functionalized Mg/Al hydroxides modified biochar for Pb(II) and Cd(II) removal: Adsorption performance and mechanism. *Sep. Purif. Technol.* **2024**, *335*, 126199. [CrossRef]
53. Youssif, M.M.; El-Attar, H.G.; Małeck, S.; Włoch, G.; Czapkiewicz, M.; Kornaus, K.; Wojnicki, M. Mercury Ion Selective Adsorption from Aqueous Solution Using Amino-Functionalized Magnetic Fe₂O₃/SiO₂ Nanocomposite. *Materials* **2024**, *17*, 4254. [CrossRef] [PubMed]
54. Alizadeh, M.; Peighambaroust, S.J.; Foroutan, R. Efficacious adsorption of divalent nickel ions over sodium alginate-g-poly(acrylamide)/hydrolyzed *Luffa cylindrica*-CoFe₂O₄ bionanocomposite hydrogel. *Int. J. Biol. Macromol.* **2024**, *254*, 127750. [CrossRef] [PubMed]
55. Li, H.; Yuan, Z.; Ding, S.; Yuan, J. Adsorption of lead ions by magnetic carbon: Comparison of magnetic carbon properties and modification methods. *J. Environ. Chem. Eng.* **2023**, *11*, 110136. [CrossRef]
56. Nwosu-Obieogu, K.; Okolo, B.I. Biosorption of chromium (VI) from textile waste water using *luffa cylindrica* activated carbon. *Environ. Qual. Manag.* **2020**, *29*, 23–31. [CrossRef]
57. Youssif, M.M.; El-Attar, H.G.; Hessel, V.; Wojnicki, M. Recent Developments in the Adsorption of Heavy Metal Ions from Aqueous Solutions Using Various Nanomaterials. *Materials* **2024**, *17*, 5141. [CrossRef] [PubMed]
58. Alizadeh, M.; Peighambaroust, S.J.; Foroutan, R.; Azimi, H.; Ramavandi, B. Surface magnetization of hydrolyzed *Luffa Cylindrica* biowaste with cobalt ferrite nanoparticles for facile Ni²⁺ removal from wastewater. *Environ. Res.* **2022**, *212*, 113242. [CrossRef] [PubMed]
59. Cai, T.; Du, H.; Liu, X.; Tie, B.; Zeng, Z. Insights into the removal of Cd and Pb from aqueous solutions by NaOH–EtOH-modified biochar. *Environ. Technol. Innov.* **2021**, *24*, 102031. [CrossRef]
60. Chen, K.; He, J.; Li, Y.; Cai, X.; Zhang, K.; Liu, T.; Hu, Y.; Lin, D.; Kong, L.; Liu, J. Removal of cadmium and lead ions from water by sulfonated magnetic nanoparticle adsorbents. *J. Colloid Interface Sci.* **2017**, *494*, 307–316. [CrossRef] [PubMed]
61. Maceiras, R.; Feijoo, J.; Perez-Rial, L.; Alfonsin, V.; Falcon, P. Study of natural zeolites for hydrogen purification: CO₂ adsorption capacity and kinetic mechanism. *Fuel* **2024**, *376*, 132732. [CrossRef]
62. Liao, J.; He, X.; Zhang, Y.; Zhang, L.; He, Z. The construction of magnetic hydroxyapatite-functionalized pig manure-derived biochar for the efficient uranium separation. *Chem. Eng. J.* **2023**, *457*, 141367. [CrossRef]
63. Sun, C.; Chen, T.; Huang, Q.; Wang, J.; Lu, S.; Yan, J. Enhanced adsorption for Pb(II) and Cd(II) of magnetic rice husk biochar by KMnO₄ modification. *Environ. Sci. Pollut. Res.* **2019**, *26*, 8902–8913. [CrossRef] [PubMed]
64. Fouad, M.; El-Subruiti, G.M.; Hasanein, A.; Eltaweil, A. Selective adsorption of Pb (II) on green Sn-BDC-MOF/GO composite: Optimization, DFT studies and mechanism. *RSC Adv.* **2025**, *15*, 33976–33990. [CrossRef] [PubMed]
65. Zhang, L.; Guo, J.; Huang, X.; Wang, W.; Sun, P.; Li, Y.; Han, J. Functionalized biochar-supported magnetic MnFe₂O₄ nanocomposite for the removal of Pb (II) and Cd (II). *RSC Adv.* **2019**, *9*, 365–376. [CrossRef] [PubMed]
66. Singh, V.; Pant, N.; Sharma, R.K.; Padalia, D.; Rawat, P.S.; Goswami, R.; Singh, P.; Kumar, A.; Bhandari, P.; Tabish, A.; et al. Adsorption Studies of Pb(II) and Cd(II) Heavy Metal Ions from Aqueous Solutions Using a Magnetic Biochar Composite Material. *Separations* **2023**, *10*, 389. [CrossRef]
67. Şahin, M.; Atasoy, M.; Arslan, Y.; Yildiz, D. Removal of Ni(II), Cu(II), Pb(II), and Cd(II) from Aqueous Phases by Silver Nanoparticles and Magnetic Nanoparticles/Nanocomposites. *ACS Omega* **2023**, *8*, 34834–34843. [CrossRef] [PubMed]
68. Donga, C.; Mishra, S.B.; Abd-El-Aziz, A.S.; Ndlovu, L.N.; Mishra, A.K.; Kuvarega, A.T. (3-Aminopropyl) Triethoxysilane (APTES) Functionalized Magnetic Nanosilica Graphene Oxide (MGO) Nanocomposite for the Comparative Adsorption of the Heavy Metal [Pb(II), Cd(II) and Ni(II)] Ions from Aqueous Solution. *J. Inorg. Organomet. Polym. Mater.* **2022**, *32*, 2235–2248. [CrossRef]
69. El-Wakeel, S.T.; Fathy, N.A.; Tawfik, M.E. Porous carbons prepared from a novel hard wood composite waste for effective adsorption of Pb (II) and Cd (II) ions. *RSC Adv.* **2023**, *13*, 34935–34946. [CrossRef] [PubMed]
70. Kavand, M.; Eslami, P.; Razeh, L. The adsorption of cadmium and lead ions from the synthesis wastewater with the activated carbon: Optimization of the single and binary systems. *J. Water Process Eng.* **2020**, *34*, 101151. [CrossRef]
71. Joshi, N.C.; Kaur, K.; Kumar, N.; Bhandari, N.S.; Thakur, A. Synthesis and adsorption applications of PPY/Fe₃O₄ nanocomposite based material. *Nano-Struct. Nano-Objects* **2021**, *25*, 100669. [CrossRef]
72. Joshi, N.C. Highly efficient removal of Pb²⁺ and Cu²⁺ ions using r-GO/PPY/SiO₂ based nanosorbent. *Int. J. Environ. Anal. Chem.* **2024**, *104*, 3280–3294. [CrossRef]
73. Asgharinezhad, A.A.; Esmaeilpour, M.; Siavoshani, A.Y. Extraction and preconcentration of Ni (II), Pb (II), and Cd (II) ions using a nanocomposite of the type Fe₃O₄@SiO₂@polypyrrole-polyaniline. *RSC Adv.* **2022**, *12*, 19108–19114. [CrossRef] [PubMed]
74. Ali, H.; Ismail, A.M. Fabrication of Magnetic Fe₃O₄/Polypyrrole/Carbon Black Nanocomposite for Effective Uptake of Congo Red and Methylene Blue Dye: Adsorption Investigation and Mechanism. *J. Polym. Environ.* **2023**, *31*, 976–998. [CrossRef]
75. Youssif, M.M.; Wojnicki, M. Efficacious Removal of Cd²⁺ and Pb²⁺ Ions from Wastewater Using a Novel Fe₃O₄/SiO₂/PANI-SDBS Nanocomposite. *Materials* **2025**, *18*, 2083. [CrossRef] [PubMed]

76. Heybet, E.N.; Ugraskan, V.; Isik, B.; Yazici, O. Adsorption of methylene blue dye on sodium alginate/polypyrrole nanotube composites. *Int. J. Biol. Macromol.* **2021**, *193*, 88–99. [[CrossRef](#)] [[PubMed](#)]
77. Tümssek, F.; Avcı, Ö. Investigation of Kinetics and Isotherm Models for the Acid Orange 95 Adsorption from Aqueous Solution onto Natural Minerals. *J. Chem. Eng. Data* **2013**, *58*, 551–559. [[CrossRef](#)]
78. Puccia, V.; Avena, M.J. On the use of the Dubinin-Radushkevich equation to distinguish between physical and chemical adsorption at the solid-water interface. *Colloid Interface Sci. Commun.* **2021**, *41*, 100376. [[CrossRef](#)]
79. Alkhalidi, H.; Alharthi, S.; Alharthi, S.; AlGhamdi, H.A.; AlZahrani, Y.M.; Mahmoud, S.A.; Amin, L.G.; Al-Shaalan, N.H.; Boraie, W.E.; Attia, M.S.; et al. Sustainable polymeric adsorbents for adsorption-based water remediation and pathogen deactivation: A review. *RSC Adv.* **2024**, *14*, 33143–33190. [[CrossRef](#)] [[PubMed](#)]
80. Dubey, R.; Bajpai, J.; Bajpai, A.K. Chitosan-alginate nanoparticles (CANPs) as potential nanosorbent for removal of Hg (II) ions. *Environ. Nanotechnol. Monit. Manag.* **2016**, *6*, 32–44. [[CrossRef](#)]
81. Kagalkar, A.; Dharaskar, S.; Chaudhari, N.; Vakharia, V.; Karri, R.R. Enhanced metal ion adsorption using ZnO-MXene nanocomposites with machine learning-based performance prediction. *Sci. Rep.* **2025**, *15*, 15563. [[CrossRef](#)] [[PubMed](#)]
82. Soliman, N.K.; Moustafa, A.F. Industrial solid waste for heavy metals adsorption features and challenges; A review. *J. Mater. Res. Technol.* **2020**, *9*, 10235–10253. [[CrossRef](#)]
83. Raji, F.; Saraeian, A.; Pakizeh, M.; Attarzadeh, F. Removal of Pb (II) from aqueous solution by mesoporous silica MCM-41 modified by ZnCl₂: Kinetics, thermodynamics, and isotherms. *RSC Adv.* **2015**, *5*, 37066–37077. [[CrossRef](#)]
84. Li, J.; Wei, Y.; Zou, L.; Luo, Y.; Liu, Y.; Li, S. Study on the adsorption mechanism of heavy metal ions by Thiosulfate-Intercalated ZnAl-LDH. *Inorg. Chem. Commun.* **2025**, *179*, 114771. [[CrossRef](#)]
85. Sheikh, S.; Naghizadeh-Dehno, O.; Mirkhalafi, S.; Ghashang, M. Magnetic heterogenization of supramolecular carbohydrates for the efficient adsorption of heavy metals removal from wastewater. *Inorg. Chem. Commun.* **2024**, *168*, 112835. [[CrossRef](#)]
86. Obayomi, K.S.; Muhorakeye, A.; Bernstein, R.; Gross, A. Bimetallic oxide nanocomposite hydrochar for phosphate capture from aqueous environments. *Sep. Purif. Technol.* **2025**, *376*, 134142. [[CrossRef](#)]
87. Shao, Y.; Wang, X.; Kang, Y.; Shu, Y.; Sun, Q.; Li, L. Application of Mn/MCM-41 as an adsorbent to remove methyl blue from aqueous solution. *J. Colloid Interface Sci.* **2014**, *429*, 25–33. [[CrossRef](#)] [[PubMed](#)]
88. Bhatt, M.; Thakkar, H.; Thakore, S. Development of β -cyclodextrin based ester-linked COF via green solvothermal approach for enhanced adsorption of heavy metals from contaminated water. *J. Environ. Chem. Eng.* **2025**, *13*, 118665. [[CrossRef](#)]
89. Lafi, R.; Montasser, I.; Hafiane, A. Adsorption of congo red dye from aqueous solutions by prepared activated carbon with oxygen-containing functional groups and its regeneration. *Adsorpt. Sci. Technol.* **2019**, *37*, 160–181. [[CrossRef](#)]
90. Qi, X.-Y.; Xie, H.-F.; Li, X.; Muhammad, Y.; Chen, C.; Wang, H.; Qi, C.-S.; Li, W.; Hong, Y. Fabrication of an anionic layered metal-organic framework for the efficient removal of Sr²⁺. *J. Mol. Struct.* **2024**, *1299*, 137220. [[CrossRef](#)]
91. Lin, C.; Dong, B.; Xu, Z. Competitive adsorption of heavy metals onto xanthate-modified sludge hydrochar and its solidification as secondary minerals. *Chemosphere* **2024**, *356*, 141878. [[CrossRef](#)] [[PubMed](#)]
92. Yin, Z.; Wang, G.; Wang, L.; Ren, P.; Sun, J.; Zhao, H.; Ji, P. An effective way to utilize solid waste resources: The application of modified fly ash in removing Cu²⁺ and Pb²⁺ in wastewater. *Sep. Purif. Technol.* **2024**, *350*, 127948. [[CrossRef](#)]
93. Yang, W.; Cheng, M.; Han, Y.; Luo, X.; Li, C.; Tang, W.; Yue, T.; Li, Z. Heavy metal ions' poisoning behavior-inspired etched UiO-66/CTS aerogel for Pb(II) and Cd(II) removal from aqueous and apple juice. *J. Hazard. Mater.* **2021**, *401*, 123318. [[CrossRef](#)] [[PubMed](#)]
94. He, S.; Zhang, F.; Cheng, S.; Wang, W. Synthesis of Sodium Acrylate and Acrylamide Copolymer/GO Hydrogels and Their Effective Adsorption for Pb²⁺ and Cd²⁺. *ACS Sustain. Chem. Eng.* **2016**, *4*, 3948–3959. [[CrossRef](#)]
95. Lin, X.; Wei, G.; Liang, X.; Liu, J.; Ma, L.; Zhu, J. The Competitive Adsorption of Chromate and Sulfate on Ni-Substituted Magnetite Surfaces: An ATR-FTIR Study. *Minerals* **2021**, *11*, 88. [[CrossRef](#)]
96. Mahmud, M.; Hossain, M.S.; Mobarak, M.B.; Quddus, M.S.; Bashar, M.S.; Akhtar, U.S.; Jahan, S.A.; Islam, D.; Ahmed, S. Engineering GO@Zn-Hap@CA porous heterostructure for ultra-fast and ultra-high adsorption efficacy: Investigation towards the remediation of chromium and lead. *Environ. Sci. Adv.* **2022**, *1*, 827–848. [[CrossRef](#)]
97. Li, J.; Lin, G.; Liang, H.; Wang, S.; Hu, T.; Li, S.; Zhang, L. Recent advances in the synthesis of MOF-based composites for heavy-metal ion adsorption. *Coord. Chem. Rev.* **2025**, *545*, 217010. [[CrossRef](#)]
98. Shao, Z.; Di, K.; Ding, L.; You, F.; Fan, C.; Wang, K. Amino-enriched Zn-MOFs with self-reduction for energy-free simultaneous removal and electrochemical detection of heavy metal ions in the aquatic environment. *Anal. Chim. Acta* **2025**, *1333*, 343408. [[CrossRef](#)] [[PubMed](#)]
99. Raji, Z.; Karim, A.; Karam, A.; Khalloufi, S. Adsorption of Heavy Metals: Mechanisms, Kinetics, and Applications of Various Adsorbents in Wastewater Remediation—A Review. *Waste* **2023**, *1*, 775–805. [[CrossRef](#)]
100. Bo, S.; Luo, J.; An, Q.; Xiao, Z.; Wang, H.; Cai, W.; Zhai, S.; Li, Z. Efficiently selective adsorption of Pb(II) with functionalized alginate-based adsorbent in batch/column systems: Mechanism and application simulation. *J. Clean. Prod.* **2020**, *250*, 119585. [[CrossRef](#)]

101. Xia, H.; Zhang, Y.; Chen, Q.; Liu, R.; Wang, H. Unraveling adsorption characteristics and removal mechanism of novel Zn/Fe-bimetal-loaded and starch-coated corn cobs biochar for Pb(II) and Cd(II) in wastewater. *J. Mol. Liq.* **2023**, *391*, 123375. [[CrossRef](#)]
102. Han, Z.; Dong, Q.; Chen, G.; Dong, H.; Zhou, R. Interface Cd-N bond bridge accelerating charge separation to the enhanced visible light driven hydrogen production from water splitting on polyaniline@Cd-Zn-S photocatalyst. *Sep. Purif. Technol.* **2022**, *300*, 121839. [[CrossRef](#)]

Disclaimer/Publisher's Note: The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.