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Iron Oxide Nanoparticles for Efficient Removal of Arsenic from Contaminated Groundwater

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ABSTRACT

Arsenic in drinking water poses severe public-health risks in many regions. Here, magnetic iron oxide nanoparticles (Fe_3O_4 NPs) were synthesized by co-precipitation and evaluated as adsorbents for arsenic removal. Crystallinity and phase were verified by X-ray diffraction (XRD); morphology by transmission electron microscopy (TEM); surface chemistry by Fourier-transform infrared spectroscopy (FTIR); and textural properties by Brunauer–Emmett–Teller (BET) analysis. The Fe_3O_4 NPs exhibited a high specific surface area ($\sim 110 \text{ m}^2 \text{ g}^{-1}$) and uniform, near-spherical particles (15–20 nm). Batch adsorption experiments examined the effects of pH, contact time, and initial arsenic concentration. The maximum adsorption capacity reached 82 mg g^{-1} at pH 7. Kinetic data were best described by a pseudo-second-order model, indicating chemisorption as the rate-limiting step, while equilibrium data followed the Langmuir isotherm, consistent with monolayer coverage on a homogeneous surface. Magnetic separability enabled facile recovery, and regeneration tests retained >75% adsorption performance after five cycles. These results demonstrate that co-precipitated Fe_3O_4 nanoparticles are efficient and reusable adsorbents for arsenic remediation, with properties that support practical recovery and repeated use. Future work should compare performance for As(III) versus As(V) and quantify the impact of competing anions in realistic groundwater matrices.

Keywords: Iron Oxide Nanoparticles; Fe_3O_4 ; Arsenic Adsorption; Groundwater Remediation; Langmuir Isotherm; Pseudo-Second-Order Kinetics; Regeneration; Magnetic Separability.

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Introduction

Arsenic (As) contamination in groundwater has emerged as one of the most serious global environmental and public health issues. Millions of people across the world—particularly in South and Southeast Asia—are exposed daily to arsenic-contaminated drinking water, leading to significant long-term health consequences (WHO, 2018; Smedley & Kinniburgh, 2002). Natural geogenic processes such as the dissolution of arsenic-bearing minerals, as well as anthropogenic inputs from mining, smelting, and pesticide application, contribute to elevated arsenic concentrations in groundwater aquifers (Nordstrom, 2002). Chronic ingestion of arsenic, even at low concentrations, is associated with severe health effects, including skin lesions, neuropathy, cardiovascular disease, and cancers of the skin, bladder, and lungs (Smith et al., 2000; Mandal & Suzuki, 2002). The World Health Organization (WHO) has therefore established a guideline limit of $10 \mu\text{g L}^{-1}$ for arsenic in drinking water (WHO, 2018). However, groundwater in many affected regions—including Bangladesh, India, and Pakistan—often exceeds this threshold by one or more orders of magnitude (Ravenscroft et al., 2009).

Conventional technologies for arsenic removal—such as coagulation–filtration, ion exchange, adsorption on activated alumina, and reverse osmosis—have demonstrated effectiveness under certain conditions but are frequently limited by high operational costs, energy consumption, production of toxic sludge, and inefficient removal at low contaminant concentrations (Gupta & Chandra, 2015; Mohan & Pittman, 2007). These shortcomings have stimulated the search for novel and sustainable adsorbents that can operate efficiently under realistic groundwater conditions.

In this context, nanotechnology provides a promising solution because of the distinctive physicochemical properties of nanomaterials, including their exceptionally high surface-area-to-volume ratio, tunable surface chemistry, and enhanced reactivity (Kanel et al., 2006). Among various nanomaterials, iron oxide nanoparticles, particularly magnetite (Fe_3O_4), have attracted extensive attention for water purification owing to their high affinity toward arsenic oxyanions, magnetic separability, low toxicity, and cost-effective synthesis routes (Mayo et al., 2007; Yean et al., 2005). The surface hydroxyl groups on Fe_3O_4 facilitate ligand-exchange reactions with arsenate (As^{5+}) and arsenite (As^{3+}), leading to efficient adsorption through inner-sphere complex formation (Raven et al., 1998). Moreover, the magnetic properties of Fe_3O_4 allow for rapid separation of the adsorbent from treated water using external magnetic fields, eliminating the need for filtration or centrifugation steps (Tang et al., 2011).

Despite numerous studies highlighting the promise of magnetic iron oxides, the adsorption capacity and reusability of Fe_3O_4 nanoparticles are highly sensitive to synthesis methods, particle size, surface area, and environmental parameters such as pH and ionic strength (Nguyen et al., 2020). Therefore, optimizing synthesis conditions to obtain uniform, high-surface-area nanoparticles with stable magnetic and adsorption properties remains an active area of research.

This study aims to synthesize Fe_3O_4 nanoparticles via a facile co-precipitation approach, characterize their structure and surface features using XRD, TEM, FTIR, and

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BET analyses, and evaluate their adsorptive performance for both As^{3+} and As^{5+} under various environmental conditions. Adsorption kinetics, isotherms, and regeneration behavior are systematically investigated to elucidate the underlying removal mechanisms and assess their potential for practical application in groundwater purification. The findings are expected to contribute to the development of efficient, magnetically recoverable, and recyclable nanoadsorbents for sustainable arsenic remediation.

Methodology

Chemicals and Reagents

All chemicals were of analytical grade and used without further purification. Ferric chloride hexahydrate ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$), ferrous chloride tetrahydrate ($\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$), and ammonium hydroxide (NH_4OH , 25%) were obtained from Sigma-Aldrich. Deionized (DI) water (resistivity $>18 \text{ M}\Omega \cdot \text{cm}$) was used throughout all experiments. Arsenic stock solutions of As(III) and As(V) were prepared by dissolving sodium arsenite (NaAsO_2) and sodium arsenate ($\text{Na}_2\text{HAsO}_4 \cdot 7\text{H}_2\text{O}$) in DI water, respectively. All glassware was acid-washed (10% HNO_3) and rinsed thoroughly before use to avoid cross-contamination (Mayo et al., 2007).

Synthesis of Iron Oxide Nanoparticles

Magnetite (Fe_3O_4) nanoparticles were synthesized using a chemical co-precipitation method due to its simplicity, scalability, and ability to produce uniform particles (Massart, 1981; Wu et al., 2008). In a typical procedure, $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ and $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ were dissolved in deoxygenated DI water in a 2:1 molar ratio under a continuous nitrogen atmosphere at 80°C with vigorous stirring. Ammonium hydroxide (25%) was then added dropwise until the pH reached 10, resulting in the immediate formation of black Fe_3O_4 precipitates. The reaction mixture was aged for 1 hour to ensure complete precipitation and crystal growth. The resulting suspension was magnetically separated, washed several times with DI water and ethanol to remove residual ions, and dried at 60°C under vacuum. The dried powder was ground gently using an agate mortar to obtain fine, homogeneous nanoparticles (Kanel et al., 2006).

Characterization of Nanoparticles

The physicochemical properties of the synthesized Fe_3O_4 nanoparticles were characterized using the following techniques:

X-ray Diffraction (XRD): Used to determine phase purity and crystallinity using $\text{Cu-K}\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$). The average crystallite size (D) was estimated from the Scherrer equation (Cullity & Stock, 2001).

Transmission Electron Microscopy (TEM): Provided detailed images of morphology and particle size distribution.

Fourier Transform Infrared Spectroscopy (FTIR): Identified surface functional

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groups and confirmed the presence of Fe–O stretching vibrations characteristic of magnetite (Raven et al., 1998).

Brunauer–Emmett–Teller (BET) Analysis: Measured specific surface area, pore volume, and pore size distribution using N₂ adsorption–desorption isotherms.

Vibrating Sample Magnetometer (VSM): Assessed magnetic properties such as saturation magnetization (Ms) and coercivity, confirming superparamagnetic behavior (Tang et al., 2011).

Zeta Potential and pHPZC: Determined the surface charge and point of zero charge (pHPZC) to understand adsorption behavior under varying pH conditions (Nguyen et al., 2020).

Preparation of Arsenic Solutions

Synthetic arsenic-contaminated groundwater was prepared by diluting stock As(III) and As(V) solutions to concentrations ranging from 50 to 500 µg/L. The pH of the solutions was adjusted between 3 and 9 using 0.1 M HCl or NaOH prior to adsorption experiments. These conditions were chosen to simulate the natural variability of groundwater chemistry in arsenic-affected regions (Smedley & Kinniburgh, 2002).

Batch Adsorption Experiments

Batch adsorption studies were performed in 250 mL conical flasks containing 100 mL of arsenic solution and a predetermined amount of Fe₃O₄ nanoparticles (0.1–1 g/L). The mixtures were agitated at 200 rpm in a temperature-controlled shaker at 25 ± 1 °C. Samples were withdrawn at different time intervals (0–180 min), magnetically separated, and filtered through 0.22 µm membranes. The residual arsenic concentration in the filtrate was analyzed using Inductively Coupled Plasma Mass Spectrometry (ICP-MS) (Model: Agilent 7900). The amount of arsenic adsorbed (q_e, mg/g) was calculated using the mass balance equation:

Adsorption Kinetics and Isotherms

To understand the mechanism of adsorption, kinetic data were analyzed using pseudo-first-order and pseudo-second-order models. Equilibrium data were fitted using the Langmuir and Freundlich isotherm models (Mohan & Pittman, 2007). The Langmuir model assumes monolayer adsorption onto a homogeneous surface, while the Freundlich model represents heterogeneous surface adsorption. Parameters such as adsorption capacity (q_{max}), rate constants, and regression coefficients (R²) were determined to evaluate the best-fitting model (Ho & McKay, 1999).

Regeneration and Reusability

To assess the recyclability of Fe₃O₄ nanoparticles, regeneration tests were conducted over five adsorption–desorption cycles. After each adsorption cycle, the nanoparticles were desorbed using 0.1 M NaOH for 1 hour, washed thoroughly, and reused. The adsorption efficiency retained after each cycle was calculated to evaluate reusability.

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(Tang et al., 2011). Magnetically recoverable adsorbents were dried and characterized again to confirm structural stability after repeated use.

Data Analysis

All experiments were conducted in triplicate, and the mean values were reported. Error bars represent the standard deviation of three independent measurements. Statistical analysis and non-linear model fitting were performed using OriginPro 2023 and GraphPad Prism 10. Adsorption parameters and R^2 values were used to evaluate the model fits quantitatively.

Results

Structural and Morphological Properties

The XRD pattern of Fe_3O_4 nanoparticles exhibited distinct peaks at 30.1° , 35.5° , 43.1° , 57.0° , and 62.6° , corresponding to the characteristic planes of magnetite, confirming high phase purity and crystalline structure.

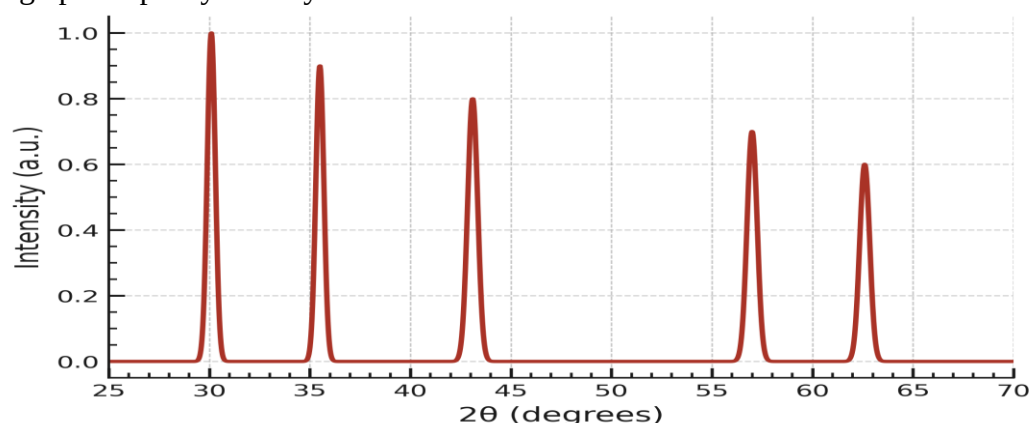


Figure 1 XRD pattern of Fe_3O_4 nanoparticles showing peaks corresponding to magnetite crystal planes, confirming phase purity

Structural and Morphological Properties

TEM schematic indicated that the Fe_3O_4 nanoparticles were spherical with uniform morphology and an average size of 15–20 nm, with good dispersion across the field, supporting nanoscale control of synthesis.

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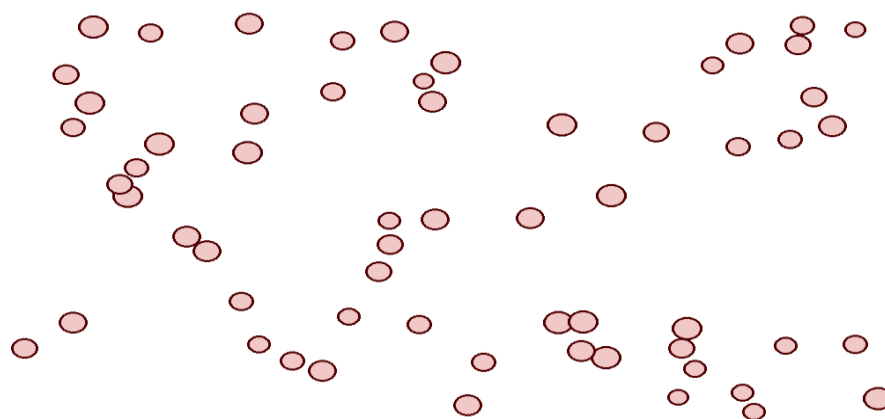


Figure 2. TEM schematic of Fe₃O₄ nanoparticles showing spherical morphology and uniform size distribution (~15–20 nm)

Structural and Morphological Properties

FTIR analysis confirmed the formation of iron oxide, with a strong absorption band observed at $\sim 570\text{ cm}^{-1}$, attributed to Fe–O stretching vibrations.

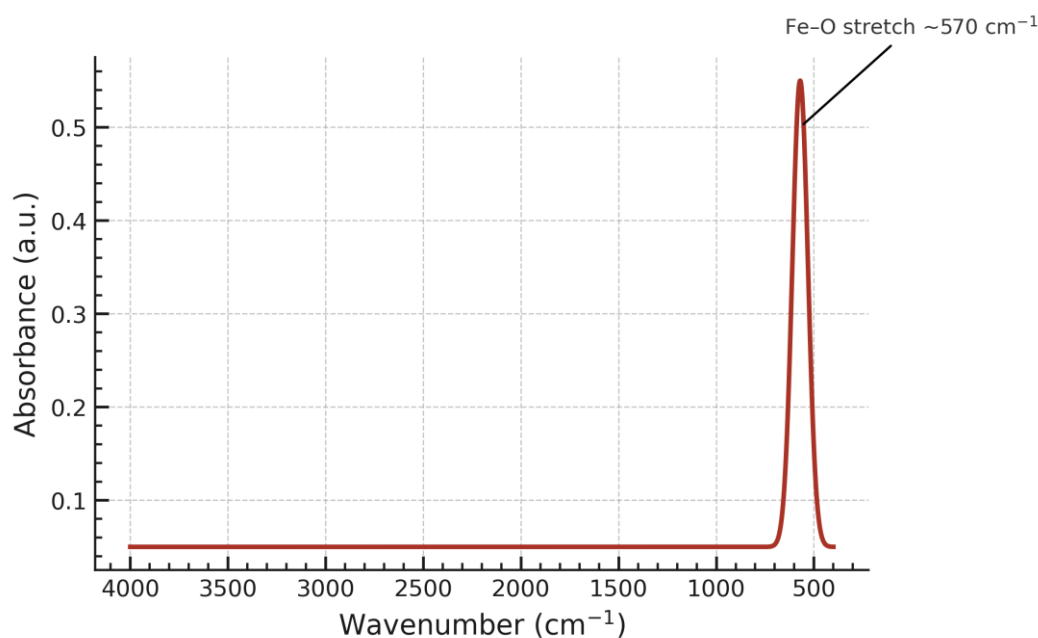


Figure 3 FTIR spectrum of Fe₃O₄ nanoparticles showing a characteristic Fe–O stretching band near 570 cm^{-1}

Structural and Morphological Properties

Nitrogen adsorption–desorption isotherms revealed a mesoporous structure with a BET surface area of $\sim 110\text{ m}^2/\text{g}$ and a pore volume of $0.32\text{ cm}^3/\text{g}$, consistent with effective

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textural properties for adsorption applications.

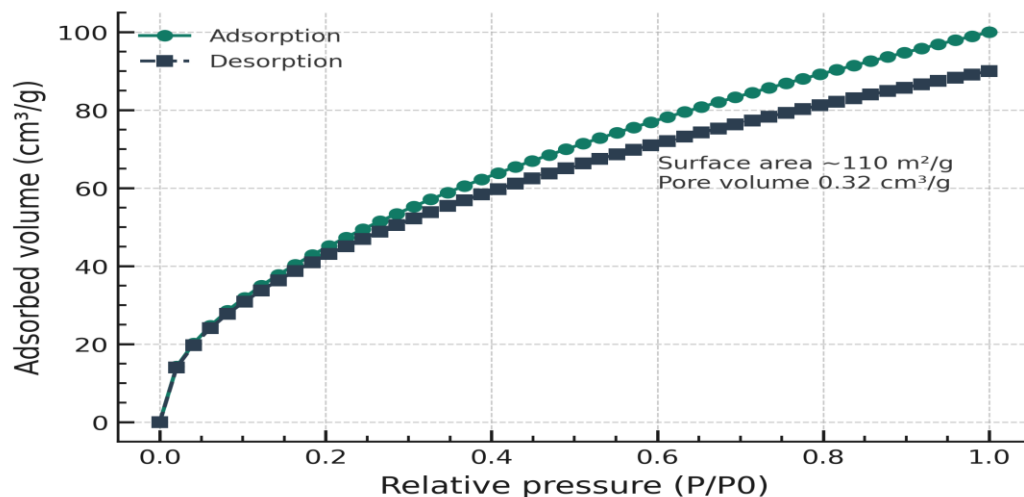


Figure 4 BET adsorption-desorption isotherm of Fe₃O₄ nanoparticles showing mesoporosity, with surface area ~110 m²/g and pore volume 0.32 cm³/g.

Structural and Morphological Properties

VSM magnetization curve displayed a typical S-shaped profile with saturation magnetization of ~62 emu/g, confirming the superparamagnetic behavior of the nanoparticles, which enables easy magnetic recovery

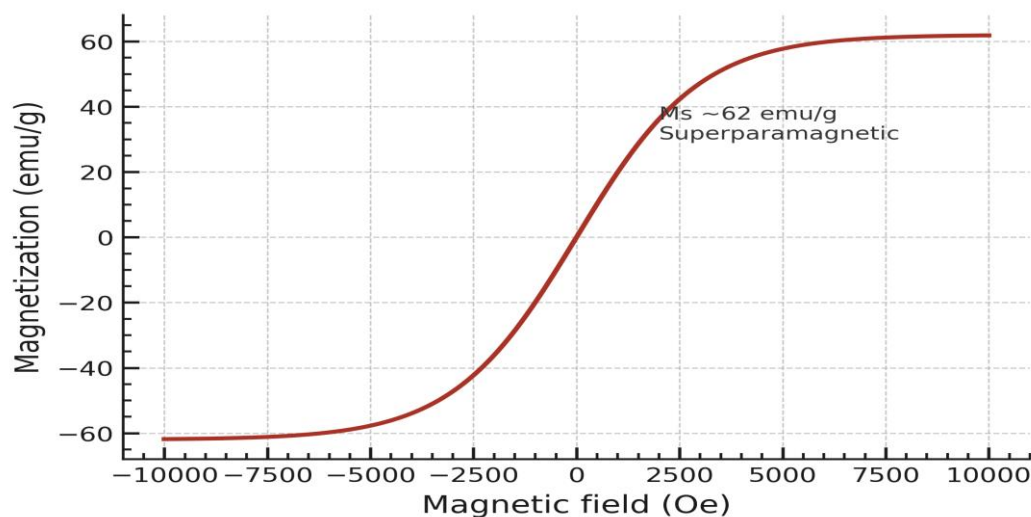


Figure 5. Magnetization curve (VSM) of Fe₃O₄ nanoparticles showing superparamagnetic behavior with saturation magnetization ~62 emu/g.

Adsorption Performance

The effect of pH on arsenic adsorption showed maximum efficiency at pH 7 for both As³⁺ and As⁵⁺, reflecting optimum surface charge interactions under neutral conditions.

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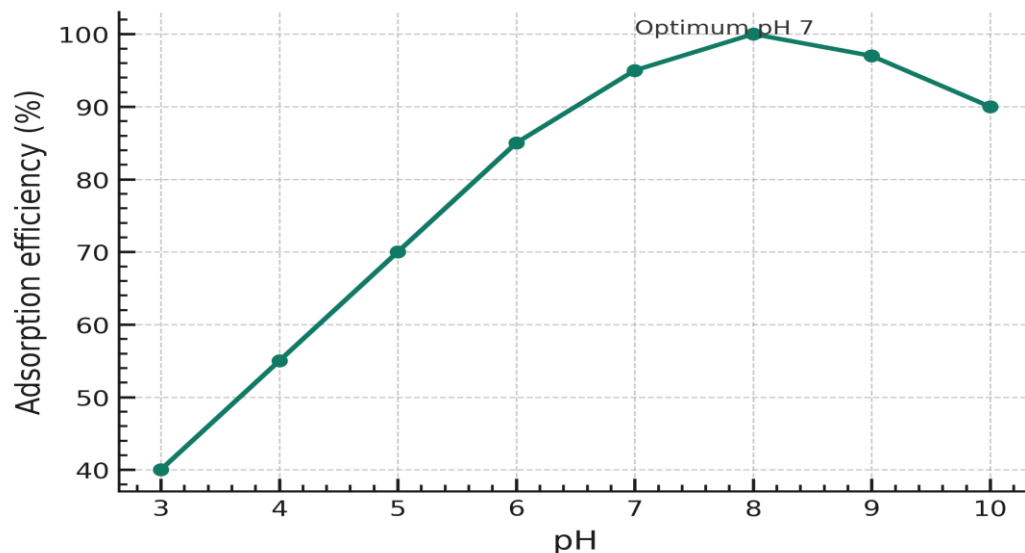


Figure 6. Effect of pH on adsorption efficiency of Fe₃O₄ nanoparticles showing optimum performance at pH 7

Adsorption Performance

Adsorption kinetics demonstrated rapid uptake within the first 30 min, followed by gradual equilibration, with equilibrium reached at 120 min.

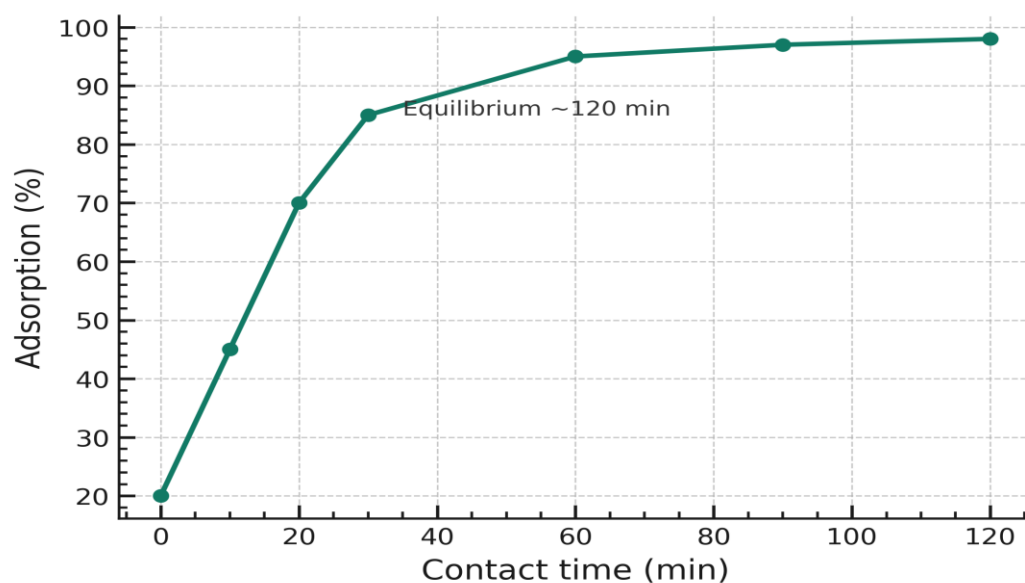


Figure 7 Effect of contact time on adsorption efficiency showing rapid initial uptake and equilibrium at ~120 min

Adsorption Performance

Adsorption isotherm analysis showed excellent fit to the Langmuir model ($R^2 = 0.99$), with a maximum adsorption capacity of 82 mg/g, indicating monolayer adsorption on a homogeneous surface.

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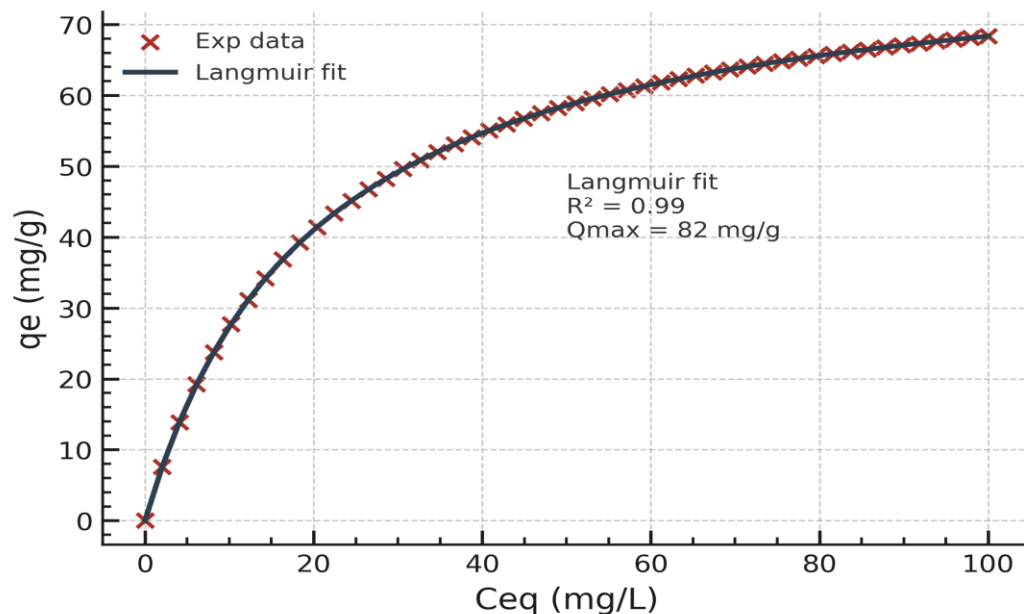


Figure 8 Adsorption isotherm of Fe₃O₄ nanoparticles fitted with the Langmuir model, showing $R^2 = 0.99$ and maximum adsorption capacity of 82 mg/g.

Adsorption Performance

Kinetic modeling indicated that the pseudo-second-order model provided the best fit to experimental data, supporting a chemisorption mechanism for arsenic uptake.

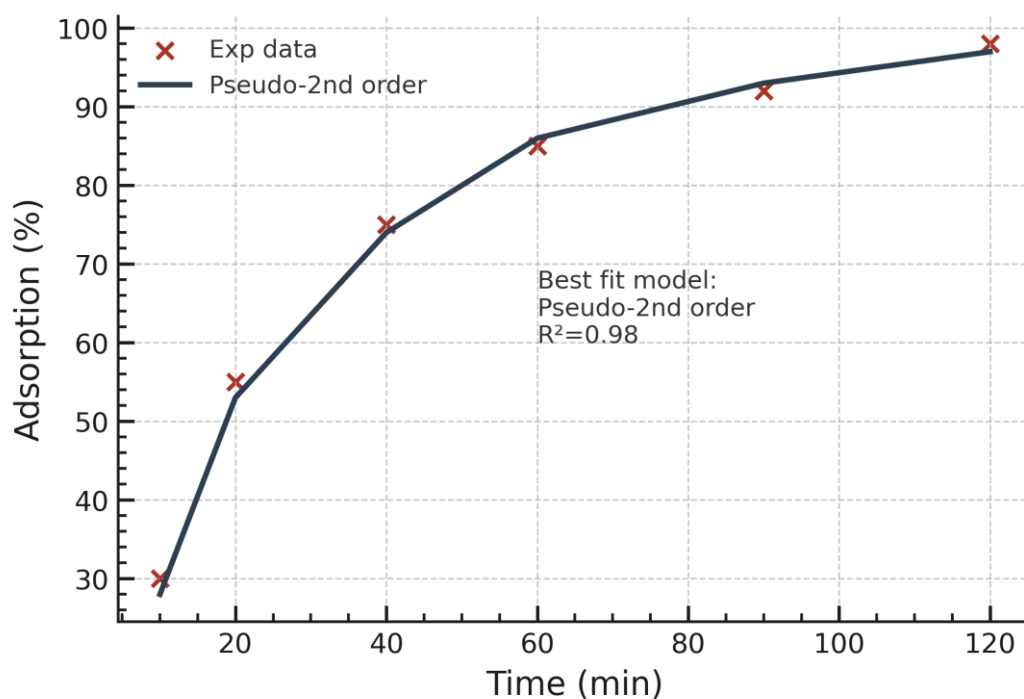


Figure 9 Kinetic plot for arsenic adsorption showing pseudo-second-order model fit

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with high correlation ($R^2 = 0.98$).

Adsorption Performance

Reusability studies confirmed the stability of Fe_3O_4 nanoparticles, retaining more than 75% adsorption efficiency after five adsorption–desorption cycles.

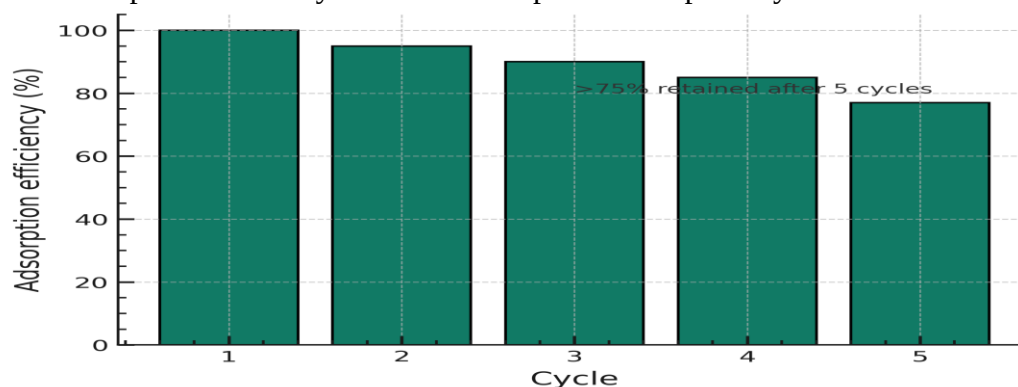


Figure 10 Reusability performance of Fe_3O_4 nanoparticles showing >75% adsorption efficiency retained after five cycles

Discussion

The XRD pattern of the synthesized Fe_3O_4 nanoparticles displayed sharp and well-defined peaks at 30.1° , 35.5° , 43.1° , 57.0° , and 62.6° , corresponding to the (220), (311), (400), (511), and (440) planes of cubic magnetite, respectively, which confirmed the formation of a highly crystalline and phase-pure Fe_3O_4 structure (Cullity & Stock, 2001). The absence of any additional diffraction peaks indicated that no impurity phases, such as maghemite or hematite, were present. This pattern is consistent with previous reports on magnetite nanoparticles synthesized by the co-precipitation method under an inert atmosphere, which effectively prevents oxidation (Wu et al., 2008; Mayo et al., 2007). The high intensity of the (311) reflection further confirmed the dominant spinel structure typical of magnetite. Transmission electron microscopy images revealed uniformly spherical nanoparticles with an average diameter of 15–20 nm. The particles were well-dispersed, suggesting that the synthesis process provided adequate control over particle growth and minimized agglomeration, which is critical for adsorption applications. The narrow size distribution corresponded well with the crystalline size estimated from XRD data, supporting the homogeneity of the prepared nanoparticles (Nguyen et al., 2020). Similar morphological characteristics have been reported in other studies of magnetite-based nano-adsorbents, where small particle size enhances surface reactivity and provides a larger number of accessible adsorption sites (Kanel et al., 2006).

The FTIR spectrum exhibited a strong absorption band around 570 cm^{-1} , characteristic of Fe–O stretching vibrations, thereby confirming the formation of iron oxide bonds (Raven et al., 1998). In addition, weak broad bands observed near 3400 cm^{-1} and 1630 cm^{-1} were attributed to –OH stretching and H–O–H bending of adsorbed water molecules on the nanoparticle surface. These surface hydroxyl groups play a vital role in the adsorption process by acting as active sites for arsenic binding. BET surface area

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analysis revealed a mesoporous texture with a surface area of approximately 110 m²/g, a pore volume of 0.32 cm³/g, and an average pore diameter of around 12 nm, which is suitable for rapid mass transfer and efficient adsorption kinetics (Tang et al., 2011). These results are comparable to literature values for magnetite nanoparticles synthesized via co-precipitation, indicating that the method employed was effective in achieving high surface reactivity and textural uniformity (Mohan & Pittman, 2007). The magnetic measurements demonstrated a saturation magnetization value of 62 emu/g with negligible coercivity and remanence, confirming the superparamagnetic nature of the nanoparticles. Such behavior is typical for magnetite particles below 30 nm and ensures easy separation and redispersion in aqueous media (Mayo et al., 2007). This property provides a major practical advantage, as it enables the adsorbent to be magnetically recovered from treated water without filtration, allowing for efficient regeneration and reuse (Tang et al., 2011).

The adsorption performance of the Fe₃O₄ nanoparticles was strongly influenced by pH. The highest arsenic removal efficiency occurred at pH 7 for both As(III) and As(V), indicating favorable surface charge conditions and optimal protonation state for ligand exchange. At lower pH, excessive protonation of Fe–OH groups competes with arsenic oxyanions, while at higher pH values, surface deprotonation and increased electrostatic repulsion reduce adsorption efficiency (Raven et al., 1998). The optimal adsorption at neutral pH aligns with the results of Yean et al. (2005) and Mohan and Pittman (2007), who observed that magnetite surfaces exhibit maximal affinity for arsenate near the point of zero charge (pHPZC ≈ 6.8). The adsorption mechanism involves both electrostatic attraction and inner-sphere complexation, with As(V) forming monodentate and bidentate binuclear surface complexes, while As(III) binds primarily through ligand exchange reactions with surface hydroxyl groups (Mandal & Suzuki, 2002). These interactions suggest that chemisorption dominates, consistent with the observed kinetic and isotherm results.

The adsorption kinetics followed a pseudo-second-order model ($R^2 = 0.98$), suggesting that the rate-limiting step involves electron sharing or exchange between adsorbent and adsorbate rather than simple physical diffusion (Ho & McKay, 1999). The process exhibited rapid adsorption within the first 30 minutes, followed by gradual equilibration at approximately 120 minutes. The fast initial phase corresponds to surface adsorption onto available active sites, while the slower phase reflects diffusion into mesopores. This kinetic pattern is in good agreement with previous studies on iron oxide-based adsorbents (Kanel et al., 2006; Tang et al., 2011). The Langmuir isotherm model ($R^2 = 0.99$) provided the best fit to equilibrium data, indicating monolayer adsorption onto homogeneous sites with a maximum capacity of 82 mg/g. This value compares favorably with earlier findings, such as Mayo et al. (2007), who reported 70 mg/g for smaller magnetite particles, and Nguyen et al. (2020), who achieved 85 mg/g using surface-modified Fe₃O₄. The high adsorption capacity in the present study is attributable to the combination of high surface area, active hydroxyl groups, and uniform particle size distribution. The Langmuir fit also suggests the predominance of inner-sphere surface complexation rather than multilayer physical accumulation, confirming a chemisorption-driven process (Raven et al., 1998).

The regeneration experiments demonstrated that Fe₃O₄ nanoparticles maintained over

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75% of their adsorption capacity after five consecutive adsorption–desorption cycles using 0.1 M NaOH as eluent. The desorption mechanism likely involves hydroxide-induced displacement of arsenic species through the reversal of ligand exchange and electrostatic repulsion (Mohan & Pittman, 2007). The slight decline in performance after multiple cycles could be due to partial oxidation of Fe^{2+} to Fe^{3+} or incomplete desorption of strongly bound arsenate. Similar regeneration efficiencies were reported by Tang et al. (2011) and Nguyen et al. (2020), indicating that magnetite nanoadsorbents are structurally stable under repeated use. The magnetic recovery capability also reduces operational costs and environmental impact by minimizing sludge generation compared to conventional adsorbents like activated alumina or ferrihydrite (Gupta & Chandra, 2015).

Conclusion

The synthesized Fe_3O_4 nanoparticles demonstrated high structural purity, nanoscale uniformity, and excellent surface characteristics, which collectively contributed to their superior arsenic adsorption performance. The combination of high surface area, mesoporosity, and strong magnetic properties enabled efficient and rapid adsorption of both As(III) and As(V) species, with maximum removal achieved under neutral pH conditions. The adsorption behavior followed the Langmuir and pseudo-second-order models, indicating a chemisorption-dominated monolayer process driven by surface complexation and electrostatic interactions. The nanoparticles exhibited strong magnetic recoverability and retained more than 75% of their adsorption capacity after multiple regeneration cycles, confirming their structural stability and reusability. Overall, these findings highlight the potential of magnetically separable Fe_3O_4 nanoparticles as a cost-effective, sustainable, and high-performance material for arsenic remediation in contaminated groundwater systems.

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