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Optimization of heavy metal (As, Cd, and Hg) adsorption performance of green-synthesized TiO₂–Fe₃O₄ nanocomposite using aloe vera extract via taguchi method

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Abstract: This study presents the green synthesis of a TiO₂–Fe₃O₄ nanocomposite using *Aloe vera* extract as a natural reducing and stabilizing agent for the simultaneous removal of As(III), Cd(II), and Hg(II) ions from aqueous environments. The biomediated sol–gel synthesis produced a nanocomposite with a crystallite size of ~14 nm (XRD) and an agglomerated, quasi-spherical morphology (SEM) driven by surface biomolecules. Adsorption performance was systematically evaluated using a Taguchi L₁₆ orthogonal array to optimize pH, adsorbent dosage, initial concentration, and contact time. Results revealed pH as the most significant factor. Under optimal conditions (pH 6, 0.05% dosage, 60 min), the composite demonstrated an exceptional maximum adsorption capacity of 160.9 mg/g for Cd(II), following the selectivity order Cd(II) > Hg(II) > As(III). FTIR analysis provided direct evidence that this high capacity is governed by chemisorption via inner-sphere surface complexation with biogenic oxygen-bearing functional groups. Furthermore, desorption studies using 0.5 M HCl demonstrated excellent practical viability, with the magnetic adsorbent retaining 84.5% of its initial Cd(II) removal efficiency after five consecutive regeneration cycles. By integrating eco-friendly synthesis with statistical optimization and robust reusability, this work offers a highly efficient and scalable approach for multi-metal wastewater remediation.

Keywords: Aloe vera extract; green synthesis; TiO₂–Fe₃O₄ nanocomposite; heavy metal adsorption; Taguchi optimization; wastewater treatment.

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1 Introduction

Water pollution with toxic heavy metals such as arsenic (As), cadmium (Cd), and mercury (Hg) has become one of the most critical environmental challenges worldwide. According to the World Health Organization (WHO) guidelines, the permissible limits for As, Cd, and Hg in drinking water and authorized wastewater discharges are strictly regulated (typically 0.01 mg/L, 0.003 mg/L, and 0.006 mg/L, respectively) due to their severe toxicity. (World Health Organization, 2022). These metals are non-biodegradable and tend to accumulate in living organisms, leading to severe health hazards even at trace concentrations. Conventional removal methods—such as chemical precipitation, ion exchange, and membrane technologies—often face limitations including secondary pollution, high operational cost, and complexity (Taher et al. 2024; Kumar et al. 2024; Ismail et al. 2024; Fu and Wang 2011). To ensure a sustainable environment, advanced wastewater treatment technologies, particularly nanomaterial-based techniques, are

increasingly applied. (Yaqoob et al. 2020). Among alternative techniques, adsorption emerges as a preferred method due to its operational simplicity, flexibility, and cost-effectiveness.

Magnetic nanocomposites, especially those combining TiO₂ and Fe₃O₄, offer dual advantages: strong adsorptive surfaces and facile magnetic separation from aqueous media. TiO₂ contributes chemical stability and high surface reactivity, while Fe₃O₄ imparts magnetic recoverability (Rezgui et al. 2022). Specifically, TiO₂ is highly distinguished by its large specific surface area, non-toxicity, and abundant surface hydroxyl groups, which serve as highly active binding sites for heavy metal complexation (Hua et al. 2012). Recent literature confirms that integrating TiO₂ into magnetic frameworks not only enhances the overall adsorption capacity but also provides excellent chemical stability over a broad pH range (Tan et al. 2015). Several studies report on Fe₃O₄@TiO₂ (core–shell) composites applied in pollutant

removal. For instance, $\text{Fe}_3\text{O}_4@\text{TiO}_2$ showed higher removal efficiencies compared to $\text{Fe}_3\text{O}_4@\beta\text{-cyclodextrin}$ for certain pollutants (Almutairi ST; 2024). Similarly, Habila et al. (2016) employed $\text{Fe}_3\text{O}_4@\text{SiO}_2@\text{TiO}_2$ as a magnetic adsorbent for heavy metals prior to ICP-MS analysis, achieving high adsorption capacities for Cd, Pb, Zn, and Cu under optimized conditions. These examples demonstrate the potential of magnetically separable TiO_2 -based composites in water treatment (Habila et al. 2016).

Beyond structure, the synthesis route plays a decisive role in the functional performance of nanocomposites. Conventional methods often require high temperatures, hazardous chemicals, or multiple steps, reducing scalability and sustainability. By contrast, green synthesis approaches using plant extracts or biomass as both reducing and stabilizing agents present an eco-friendly alternative. Plant extracts, rich in phenolics, flavonoids, and other functional groups, can mediate nanoparticle growth and stabilize surfaces, often introducing additional adsorption sites. Such strategies have been successfully demonstrated in various contexts (Taher et al. 2024).

Optimizing the adsorption process in multi-metal systems is nontrivial due to the interactions among metal ions and competitive adsorption. Traditional one-factor-at-a-time methods may overlook synergistic effects. The Taguchi orthogonal array design presents an efficient statistical approach to explore several factors simultaneously, determine main effects, and capture robust conditions with minimal experiments (Srivastava et al. 2008). In fact, recent studies apply Taguchi optimization for chromium removal, showing that both synthesis and process parameters can be fine-tuned to maximize adsorption (Aziri et al. 2025).

In this context, the present work uniquely combines three elements: (i) Aloe vera extract-based green synthesis of $\text{TiO}_2\text{-Fe}_3\text{O}_4$ nanocomposite, (ii) simultaneous adsorption study of three heavy metal ions (As^{3+} , Cd^{2+} , Hg^{2+}), and (iii) Taguchi L_{16} optimization of operating parameters (pH, adsorbent dosage, metal concentration, contact time). Despite the extensive literature on single-metal removal, a noticeable research gap remains in the simultaneous multi-metal adsorption utilizing fully green-synthesized magnetic nanocomposites. The novelty of the present study lies in its cost-effectiveness (utilizing abundant, low-cost plant extracts), high reliability (ensured by robust Taguchi statistical optimization), and superior performance in multi-ion environments. The main advantage of this study is the development of an eco-friendly, highly reactive, and easily recoverable adsorbent. Conversely, a potential disadvantage, common to many nano-adsorbents, is the future challenge of scaling up the green synthesis process for continuous industrial column applications (Nasrollahzadeh et al 2021). Compared to earlier studies focusing on single metal systems or non-green synthesis, our investigation aims to present a more applicable and efficient solution for real-world wastewater remediation and future large-scale environmental applications.

2 Materials and Method

2.1. Chemicals and Reagents

All chemicals used in this study were of analytical grade (purity >98%) and purchased from Sigma-Aldrich (St. Louis, MO, USA) unless otherwise stated. Titanium(IV) isopropoxide (TTIP, 97%) served as the titanium source, while ferric chloride hexahydrate ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) and ferrous chloride tetrahydrate ($\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$) provided Fe^{3+} and Fe^{2+} ions, respectively. Sodium hydroxide (NaOH, 0.5–1.0 M) and hydrochloric acid (HCl, 37%) were used to adjust pH values during synthesis. Fresh Aloe vera leaves were collected and processed to obtain the gel extract, which functioned as a natural reducing and stabilizing agent in the green synthesis process. The Fe/Ti molar ratio was fixed at 0.25, and all glassware was cleaned with diluted nitric acid and rinsed thoroughly with deionized water before use.

2.2. Green Synthesis of $\text{TiO}_2\text{-Fe}_3\text{O}_4$ Nanocomposite

The $\text{TiO}_2\text{-Fe}_3\text{O}_4$ nanocomposite was synthesized using a green sol–gel approach assisted by Aloe vera extract. Initially, 20 g of fresh Aloe vera gel was homogenized in approximately 60–70 mL of deionized water to prepare the bio-reducing extract. In a separate beaker, 0.761 g of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ and 0.28 g of $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ were dissolved in 125 mL of distilled water under constant stirring. The Aloe vera extract was added dropwise to this iron precursor solution and stirred for 30 minutes.

The pH of the solution was adjusted to 2.5–3.5 using dilute HCl to maintain Fe ion solubility and control TTIP hydrolysis. Subsequently, 5.0 mL of TTIP was added dropwise under vigorous stirring, and the temperature was maintained at 80 °C for 1 hour. Then, the pH was raised to 9–10 by adding NaOH, followed by another 1 hour of stirring. The resulting suspension was centrifuged at 6000 rpm for 5 min, washed twice with deionized water, and dried in an oven at 120 °C for 6 h. The dried composite was ground into fine powder and stored in airtight containers for further characterization and adsorption experiments. The Aloe vera biomolecules (flavonoids, polysaccharides, and phenolic compounds) acted as both reducing and capping agents, facilitating nanoparticle stabilization and surface functionalization (Villagrán et al. 2024).

2.3. Characterization Techniques

The crystalline structure of the synthesized nanocomposite was analyzed using X-ray Diffraction (XRD) (Panalytical Empyrean, Netherlands) equipped with $\text{CuK}\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$). The diffraction patterns were recorded in the 2θ range of 20–80°, and the average crystallite size was calculated using the Scherrer equation, yielding approximately 14 nm. The surface morphology and particle distribution were further investigated using Scanning Electron Microscopy (SEM) (FEI/Quanta FEG 450, Darmstadt, Germany). The SEM micrographs revealed that the nanocomposite particles possessed a quasi-spherical shape with a tendency to agglomerate, which can be attributed to the presence of biomolecules from the Aloe vera extract acting as capping and stabilizing agents. X-ray Fluorescence (XRF) (Thermo Scientific/ ARL PERFORM'X, Ecublens,

Switzerland) analysis confirmed the elemental composition, revealing 35.29 wt% Ti and 9.06 wt% Fe, consistent with the theoretical Fe/Ti ratio (0.25). Sodium (≈ 7.4 wt%) was also detected, likely due to NaOH addition during synthesis. It is worth noting that the total weight percentage (wt%) did not reach exactly 100%. This is highly attributed to the inability of standard XRF to detect light elements (e.g., oxygen, carbon) and the presence of organic surface-capping biomolecules derived from the Aloe vera extract.

Furthermore, Fourier Transform Infrared (FTIR) spectroscopy was performed in the wavenumber range of 4000–400 cm^{-1} to identify the functional groups on the adsorbent surface before and after the adsorption of As(III), Cd(II), and Hg(II) ions, thereby elucidating the binding mechanisms.

2.4. Adsorption Experiments

Batch adsorption experiments were performed to investigate the removal of As(III), Cd(II), and Hg(II) ions from synthetic wastewater solutions. The heavy metal concentrations were prepared using standard solutions (25–100 mg/L), and adsorption tests were conducted in 50 mL falcon tube containing 10 mL of solution and the required amount of adsorbent. The effects of four major parameters—pH (3, 4, 5, 6), adsorbent dosage (0.05–0.3%), initial metal ion concentration (25–100 mg/L), and contact time (30–120 min) were investigated using the Taguchi L_{16} orthogonal design. The solutions were agitated using a magnetic stirrer, and after equilibrium, the mixtures were separated magnetically and analyzed for residual metal concentration using Inductively Coupled Plasma Mass Spectrometry (ICP-MS) (Thermo Scientific iCAP Qc, Bremen, Germany). The adsorption capacity (q_e , mg/g) was calculated as:

$$q_e = \frac{(C_0 - C_e) \times V}{m} \quad (1)$$

where C_0 and C_e (mg/L) are the initial and equilibrium concentrations of metal ions, V is the solution volume (L), and m is the mass of adsorbent (g).

2.5. Desorption and Reusability Studies

Evaluating the reusability of the $\text{TiO}_2\text{-Fe}_3\text{O}_4$ nanocomposite is crucial for its practical and industrial application. Desorption experiments were conducted by treating the metal-loaded magnetic adsorbent with 0.5 M HCl an eluent solution. After separating the adsorbent via an external magnetic field, it was washed thoroughly with deionized water and dried before being introduced into the next adsorption cycle. This adsorption-desorption regeneration process was repeated for five consecutive cycles to assess the long-term stability and reusability of the synthesized nanocomposite.

2.5. Experimental Design and Optimization via Taguchi Method

The Taguchi statistical approach was employed to optimize process parameters efficiently (Srivastava et al. 2008). A L_{16} (4^4) orthogonal array was selected, allowing evaluation of four variables at four levels each with only sixteen experiments. The signal-to-noise (S/N) ratio was used to

determine the robustness of each factor, applying the “larger-the-better” criterion to maximize adsorption capacity. Similar applications of the Taguchi optimization method for adsorption process design have been reported in several recent studies, confirming its reliability and statistical robustness in environmental engineering research (Rahman et al. 2023; Aziri et al. 2025).

3. Results and Discussion

3.1. Structural Characterization (XRD Analysis)

The XRD pattern of the synthesized $\text{TiO}_2\text{-Fe}_3\text{O}_4$ nanocomposite (Fig. 1) exhibited distinct diffraction peaks at 2θ values of 31.5° , 35.5° , 43.3° , 46.9° , 56.9° , and 62.7° , corresponding to the (101), (311), (400), (511), and (440) planes of Fe_3O_4 and TiO_2 crystalline phases (JCPDS No. 21-1272 and 84-1286). The coexistence of both anatase TiO_2 and spinel Fe_3O_4 phases confirmed the successful integration of the two oxides into a single nanostructured composite.

The average crystallite size, calculated using the Scherrer equation, was approximately 14 nm, indicating the formation of ultrafine nanocrystals. The peak broadening observed in the diffractogram was consistent with nano-dimensional characteristics and potential lattice strain due to Fe–Ti interface interactions. Similar nanoscale crystallinity was reported by Cheng et al. (2012), who synthesized Fe_3O_4 nanoparticles with an average size of 15–20 nm, demonstrating comparable diffraction patterns and crystallinity (Cheng et al. 2012). This structural refinement is critical for adsorption performance because smaller crystal sizes result in increased surface-to-volume ratios and higher densities of active sites. The presence of Fe_3O_4 further enhances magnetization, allowing rapid solid–liquid separation after adsorption (Habila et al. 2016).

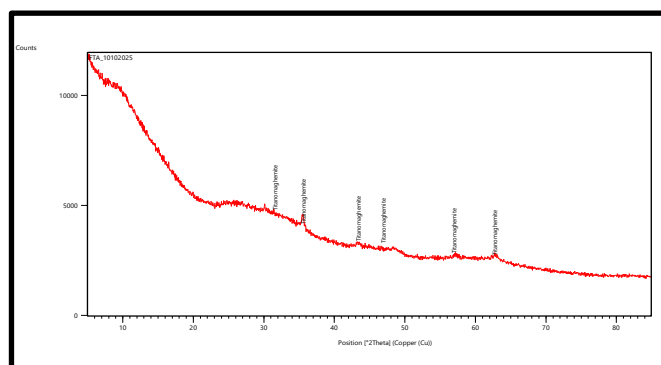


Fig. 1 XRD analysis of $\text{TiO}_2\text{-Fe}_3\text{O}_4$ nanocomposite obtained by green synthesis method

3.2. Surface Morphology (SEM Analysis)

The surface morphology and microstructural characteristics of the green-synthesized $\text{TiO}_2\text{-Fe}_3\text{O}_4$ nanocomposite were investigated using Scanning Electron Microscopy (SEM). The SEM micrographs (Fig. 2) reveal that the particles predominantly exhibit a quasi-spherical morphology with a certain degree of agglomeration. This aggregation is typically attributed to the high surface energy of the nanoparticles and

the presence of bioactive molecules (such as polysaccharides and proteins) from the Aloe vera extract, which act as capping and bridging agents on the particle surfaces. Despite the agglomeration, the composite displays a rough and porous surface texture. This porosity is a critical feature that provides an increased number of active sites, thereby facilitating the efficient adsorption of As(III), Cd(II), and Hg(II) ions from aqueous solutions. Furthermore, the microscopic analysis confirms the formation of a homogeneous nanocomposite structure, consistent with the crystallite nature indicated by the XRD results.

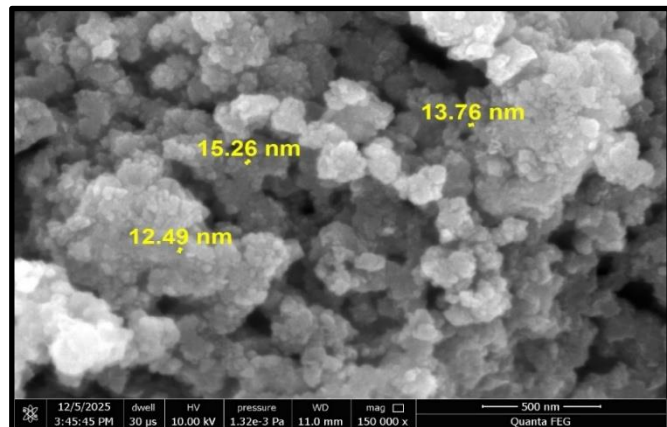


Fig. 2 SEM analysis of TiO₂-Fe₃O₄ nanocomposite obtained by green synthesis method

3.3. Elemental Composition (XRF Analysis)

The X-ray fluorescence (XRF) spectroscopy confirmed the predominant presence of Ti (35.29 wt%) and Fe (9.06 wt%), in close agreement with the theoretical Fe/Ti molar ratio of 0.25 targeted during synthesis. Additionally, minor peaks corresponding to Na (7.4 wt%), K (0.273 wt%), Ca (0.235 wt%), V (0.127 wt%), Cl (0.190 wt%), Mg (0.097 wt%), and Mn (0.056 wt%) were detected, as summarized in Table 1. The presence of sodium is attributed to the NaOH used for pH adjustment during the sol–gel process, while trace elements such as K and Ca may originate from the *Aloe vera* extract employed in the green synthesis route. It should be noted that oxygen (O) was not quantified in the XRF results, as light elements ($Z < 11$) are typically below the detection threshold of conventional XRF instruments. Furthermore, due to the presence of organic compounds derived from *Aloe vera*—including polysaccharides, flavonoids, and phenolic structures—an accurate oxygen mass percentage could not be determined within the oxide matrix. These organics act as surface capping and stabilizing agents, contributing to the observed functionalization of the nanocomposite surface rather than its inorganic elemental composition.

The observed Ti and Fe proportions indicate a balanced distribution of TiO₂ and Fe₃O₄ domains, which can synergistically contribute to adsorption and photocatalytic

efficiency. The homogeneous incorporation of Fe ions into the TiO₂ matrix is known to enhance electron mobility and suppress charge recombination, improving redox activity, as supported by previous reports (Rezqui et al. 2022; Almutairi ST. 2024).

Table 1 XRF analysis results of TiO₂-Fe₃O₄ nanocomposite obtained by green synthesis method

Element	Ti	Fe	Na	K	Ca	V	Cl
(wt %)	35.29±0.42	9.06±0.15	7.4±0.21	0.273±	0.235±0.02	0.127±0.02	0.190±0.01

3.4. Adsorption Performance and Taguchi Optimization

The adsorption experiments were conducted using a mixed-ion solution containing As(III), Cd(II), and Hg(II) ions to simulate real wastewater conditions. The removal efficiency varied significantly with pH, adsorbent dosage, contact time, and initial concentration. The Taguchi L₁₆ orthogonal array was used to systematically evaluate these four parameters and their interactions (table 2). Analysis of the signal-to-noise (S/N) ratios revealed that pH was the most influential factor across all three metals, followed by adsorbent dosage, initial concentration, and contact time. The “larger-the-better” criterion was adopted to maximize adsorption capacity.

3.4.1. Arsenic Adsorption (As³⁺)

The adsorption capacity for As(III) ranged from 3.06 to 13.30 mg/g, with optimum conditions observed at pH 6, 0.05% adsorbent dosage, 100 mg/L concentration, and 60 min contact time. Higher pH values favor deprotonation of surface hydroxyl groups, enhancing electrostatic attraction between arsenate species and active sites on the adsorbent. Similar pH-dependent trends have been observed in other adsorption studies (Cardinale et al. 2023).

3.4.2. Cadmium Adsorption (Cd²⁺)

The Taguchi optimization results revealed that the optimal conditions for Cd(II) adsorption were pH 6, 0.05 % adsorbent dosage, and 60 min contact time. Cd(II) exhibited the highest adsorption performance among the investigated metals, with a maximum adsorption capacity of 160.9 mg g⁻¹, surpassing many reported biosorbents and green nanocomposites. The strong affinity of Cd²⁺ ions toward the synthesized TiO₂-Fe₃O₄ nanocomposite can be attributed to the chelation and complexation of Cd ions with oxygen-containing functional groups (–OH, –COOH, and –C=O) derived from Aloe vera-mediated surface capping. These biomolecules enhance the density of active binding sites, promoting metal–ligand coordination on the nanocomposite surface.

Table 2 Taguchi experimental parameters and corresponding adsorption capacities of heavy metals (As, Cd, Hg) on green-synthesized TiO₂–Fe₃O₄ nanocomposite

EXPERIMENT NO	pH	Adsorbent Dosage, %	Initial Concentration, ppm	Contact time, min	Final Solution Volume, ml	75As	111Cd	202Hg
						Adsorption Capacity, mg/g	Adsorption Capacity, mg/g	Adsorption Capacity, mg/g
1	3	0,05	25	30	10	7.73 ± 0.24	3.47 ± 0.12	2.49 ± 0.09
2	3	0,1	50	60	10	9.37 ± 0.31	2.87 ± 0.10	4.18 ± 0.15
3	3	0,2	75	90	10	10.55 ± 0.38	22.25 ± 0.82	10.08 ± 0.34
4	3	0,3	100	120	10	8.18 ± 0.29	22.65 ± 0.85	7.64 ± 0.25
5	4	0,05	50	90	10	3.06 ± 0.11	59.18 ± 2.15	38.47 ± 1.42
6	4	0,1	75	120	10	5.45 ± 0.18	45.15 ± 1.68	21.57 ± 0.78
7	4	0,2	100	30	10	4.93 ± 0.15	42.80 ± 1.55	29.80 ± 1.05
8	4	0,3	25	60	10	1.37 ± 0.05	7.79 ± 0.28	7.86 ± 0.26
9	5	0,05	75	120	10	5.99 ± 0.21	67.79 ± 2.44	14.53 ± 0.52
10	5	0,1	100	30	10	8.62 ± 0.30	50.47 ± 1.92	20.49 ± 0.74
11	5	0,2	25	60	10	0.99 ± 0.04	11.82 ± 0.41	10.66 ± 0.38
12	5	0,3	50	90	10	1.93 ± 0.07	15.84 ± 0.58	14.87 ± 0.54
13	6	0,05	100	60	10	13.30 ± 0.48	160.90 ± 5.82	31.46 ± 1.15
14	6	0,1	25	90	10	n.d.	21.09 ± 0.77	7.20 ± 0.24
15	6	0,2	50	120	10	n.d.	21.22 ± 0.78	7.93 ± 0.27
16	6	0,3	75	30	10	n.d.	21.24 ± 0.79	7.77 ± 0.26

3.4.3. Mercury Adsorption (Hg²⁺)

The Hg(II) adsorption capacity varied between 2.49 and 38.47 mg/g, with the highest performance observed at pH 4 and 0.05% dosage. At higher pH, partial precipitation and competitive binding likely reduced available active sites. The high affinity at lower pH can be attributed to the strong coordination of Hg²⁺ with oxygen donor ligands and Fe–O–Ti bridges (Habila et al. 2016). The composite exhibited a selective affinity order of Cd(II) > Hg(II) > As(III). Such selectivity trends are primarily governed by ionic radii, electronegativity, and hydration energy, consistent with Pearson’s hard–soft acid–base (HSAB) principle (Srivastava et al. 2008). The adsorption capacities obtained in this study were significantly higher than those reported for most biomass- or magnetite-based adsorbents. Compared to conventional Fe₃O₄- or TiO₂-based materials, the Aloe vera-mediated TiO₂–Fe₃O₄ nanocomposite exhibited superior adsorption performance and operational simplicity, confirming the synergistic role of TiO₂ and Fe₃O₄ domains.

Specifically, Srivastava et al. (2008) reported Cd(II) adsorption capacities of 20–35 mg g^{−1} using rice husk ash, while Sachan et al. (2021) achieved approximately 80 mg g^{−1} Cd removal using biosilica nanoparticles derived from plant biomass. In contrast, the present study demonstrated a Cd(II) adsorption capacity of 160.9 mg g^{−1}, nearly twofold higher than the best-performing green nanomaterials reported so far. Furthermore, the adoption of the Taguchi optimization methodology effectively minimized the number of experimental runs (16 instead of 81 for a full factorial design), while providing statistically significant insights into the influence of pH, dosage, and concentration parameters. This confirms the reliability of the Taguchi design for multi-metal adsorption systems (Rahman et al. 2023; Aziri et al. 2025). Overall, these findings highlight the remarkable adsorption efficiency, eco-friendly synthesis route, and statistical robustness of the developed Aloe vera-assisted TiO₂–Fe₃O₄ nanocomposite, positioning it as a promising candidate for sustainable wastewater treatment applications.

3.4.4. Adsorption Mechanism and Surface Chemistry

The adsorption of As(III), Cd(II), and Hg(II) ions onto *Aloe vera*-assisted TiO₂–Fe₃O₄ nanocomposites is governed by a combination of electrostatic attraction, surface complexation, and ion exchange mechanisms. The point of zero charge (pH_{pzc}) of the adsorbent plays a critical role in determining the surface charge and, consequently, the effect of solution pH on metal binding. At low pH (pH < pH_{pzc}), the protonation of surface hydroxyl groups ($\equiv\text{Ti}-\text{OH}_2^+$ and $\equiv\text{Fe}-\text{OH}_2^+$) enhances electrostatic interactions, facilitating the adsorption of anionic species like As(III) (Goldberg 2002). Conversely, at higher pH (pH > pH_{pzc}), deprotonated oxygen sites ($\equiv\text{Ti}-\text{O}^-$ and $\equiv\text{Fe}-\text{O}^-$) serve as active centers for inner-sphere complex

formation with metal cations such as Cd(II) and Hg(II), promoting surface complexation (Loosli et al., 2015).

Additionally, *Aloe vera*-derived biomolecules introduce functional moieties (hydroxyl, carboxyl, and carbonyl groups) that significantly enhance metal ion binding through hydrogen bonding and chelation. To elucidate this binding mechanism and provide direct evidence of surface complexation, Fourier Transform Infrared (FTIR) spectra of the nanocomposite were recorded before (Fig. 3-a) and after (Fig. 3-b) the simultaneous adsorption of heavy metals. The broad absorption band observed in the range of 3200–3400 cm⁻¹ is attributed to the stretching vibrations of surface hydroxyl (–OH) groups and adsorbed water molecules.

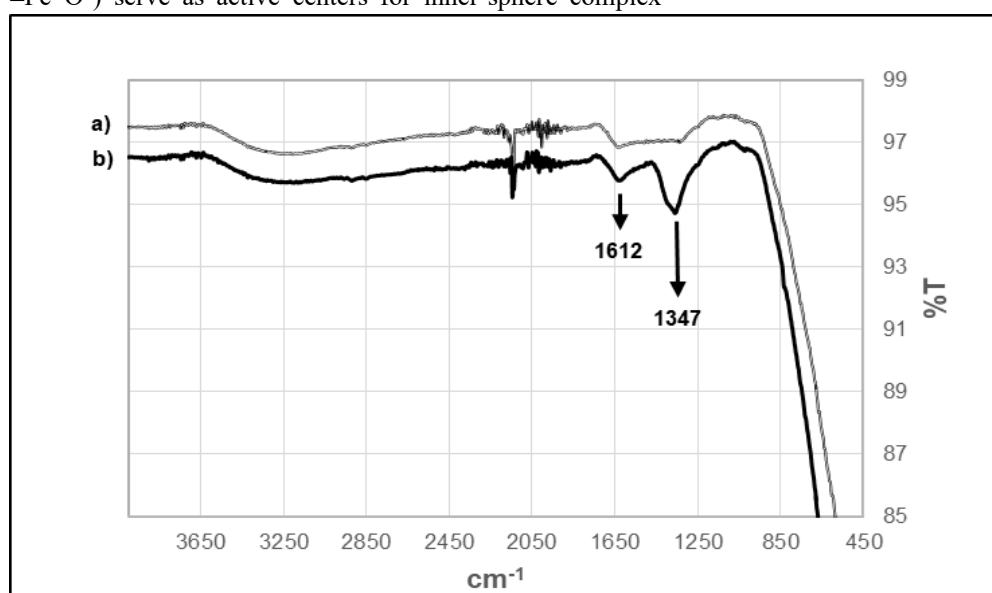


Fig. 3. Fourier Transform Infrared (FTIR) spectra of the green-synthesized *Aloe vera*-assisted TiO₂–Fe₃O₄ nanocomposite (a) before and (b) after the simultaneous adsorption of As(III), Cd(II), and Hg(II) ions.

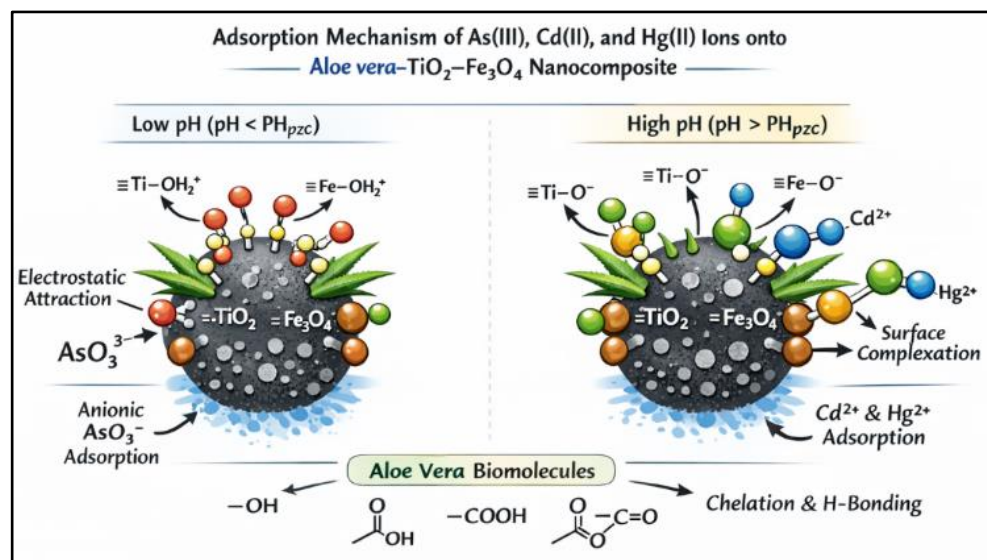


Fig. 4. Proposed adsorption mechanism of As(III), Cd(II), and Hg(II) ions onto the green-synthesized *Aloe vera*-assisted TiO₂–Fe₃O₄ nanocomposite. (Schematic representation was created/refined with the assistance of generative AI tools).

Following metal adsorption, noticeable spectral alterations occurred in this region, indicating chemical interactions. Most notably, the intensity of the peak at 1612 cm^{-1} , which corresponds to the asymmetric stretching vibrations of carboxylate ($-\text{COO}^-$) or carbonyl ($\text{C}=\text{O}$) groups, increased significantly. Furthermore, a highly distinct and sharp peak emerged at 1347 cm^{-1} in the post-adsorption spectrum, ascribed to the symmetric stretching of carboxylate groups or $\text{C}-\text{O}$ bonds. The drastic intensification and emergence of these peaks directly confirm that the biogenic oxygen-bearing functional groups actively participated in the binding process through inner-sphere surface complexation and chelation. Meanwhile, the characteristic metal-oxygen lattice vibrations ($\text{Ti}-\text{O}$ and $\text{Fe}-\text{O}$) below 850 cm^{-1} remained structurally intact, verifying the chemical stability of the core magnetic nanocomposite during the remediation process. Based on these comprehensive characterization results (XRD, SEM, XRF, and FTIR), the proposed reaction scheme illustrating the overall adsorption mechanism is provided in **Fig. 4**.

Finally, the incorporation of Fe_3O_4 domains into the adsorbent not only facilitates surface complexation but also enables magnetic recovery of the material without chemical alteration. This property allows for efficient separation and reuse, providing significant practical advantages in wastewater treatment by reducing operational costs (Moeini et al. 2026).

3.5. Desorption and Reusability Studies

The reusability of an adsorbent is a critical factor for its practical application and economic viability in industrial wastewater treatment. To evaluate the regeneration capacity of the synthesized $\text{TiO}_2\text{-Fe}_3\text{O}_4$ nanocomposite, adsorption-desorption cycles were repeated for five consecutive runs using the best-performing metal ion, Cd(II) . Desorption was carried out using 0.5 M HCl as the eluent. The selection of HCl as the desorbing agent is scientifically justified by a dual-action desorption mechanism. First, the high concentration of protons (H^+) effectively outcompetes Cd^{2+} ions for the active binding sites (such as hydroxyl and carboxylate groups) and protonates the adsorbent surface. This protonation induces strong electrostatic repulsion against the bound heavy metal cations, facilitating their release. Second, chloride ions (Cl^-) can form highly soluble coordination complexes with the desorbed Cd^{2+} ions (e.g., CdCl^+ , CdCl_2), driving the desorption equilibrium forward into the aqueous phase (Rengaraj et al., 2002). After each adsorption cycle, the metal-loaded nanocomposite was magnetically separated, treated with the eluent for 60 min, washed thoroughly with deionized water to restore surface neutrality, and dried before the next cycle.

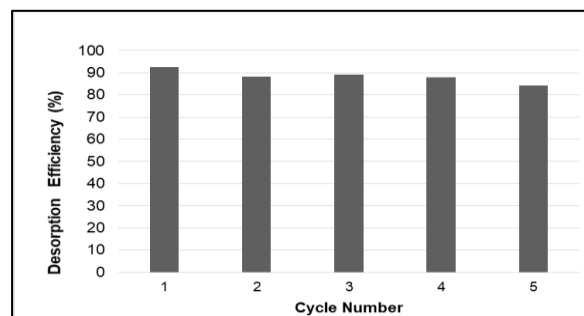


Fig. 5. Reusability performance of the green-synthesized $\text{TiO}_2\text{-Fe}_3\text{O}_4$ nanocomposite for Cd(II) ions over five consecutive adsorption-desorption cycles.

As illustrated in Fig. 5 the adsorption capacity remained highly stable across the cycles. The removal efficiency for Cd(II) slightly decreased from 92.5% in the first cycle to 84.5% after the fifth cycle. This minor decline can be attributed to the irreversible binding of some metal ions to deep active sites and the inevitable, slight loss of the adsorbent mass during the successive washing and magnetic separation steps. Nevertheless, the high retention of adsorption capacity after five cycles distinctly confirms the excellent structural stability and outstanding reusability of the green-synthesized $\text{TiO}_2\text{-Fe}_3\text{O}_4$ nanocomposite.

4. Conclusion

In this study, an eco-friendly and highly efficient $\text{TiO}_2\text{-Fe}_3\text{O}_4$ nanocomposite was successfully synthesized using an *Aloe vera* extract-mediated green sol-gel route. Moving beyond a mere presentation of material characteristics, this study highlights the synergistic role of the biogenic functional groups and the $\text{TiO}_2/\text{Fe}_3\text{O}_4$ matrix in providing a highly porous, functionalized, and magnetically separable surface. The statistical Taguchi L_{16} optimization proved highly robust for multi-metal systems, yielding an exceptional maximum adsorption capacity of 160.9 mg/g for Cd(II) under optimized conditions (pH 6), outperforming many conventional bio-adsorbents. Furthermore, FTIR analysis provided direct analytical evidence of a chemisorption-driven mechanism via inner-sphere surface complexation, while the magnetic properties ensured facile recovery and high reusability, offering a practical advantage for sustainable water treatment.

Despite these promising batch-scale results, the transition from laboratory to real-world application requires continuous exploration. Based on the current findings, the developed nanocomposite holds immense potential for broad environmental and practical applications. Specifically, its high affinity and metal selectivity at mild pH levels make it a highly suitable candidate for the purification of domestic drinking water and the remediation of heavy metal-contaminated groundwater and well water. Furthermore, the material's robust structural integrity, cost-effectiveness, and magnetic recoverability present significant opportunities for large-scale agricultural runoff management and continuous industrial processes.

To further validate its commercial and field-scale applicability, future research should prioritize continuous flow (column) operations, long-term pilot-scale studies, and the evaluation of the adsorbent's performance in authentic, highly complex industrial effluents. The combination of green synthesis and Taguchi optimization provides a robust framework for developing next-generation multifunctional nanocomposites for environmental remediation.

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