

RESEARCH ARTICLE

Green Synthesis of Fe₃O₄ Nanoparticles Using Tea Leaf Extract for Polyacrylamide Adsorption

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ABSTRACT

Magnetic Fe₃O₄ nanoparticles (Fe₃O₄ NPs) were synthesized via a green method using *Camellia sinensis* leaf extract as a reducing and stabilizing agent. The obtained superparamagnetic nanoparticles exhibited a saturation magnetization of ~50 emu/g and an average crystallite size of ~20 nm, formed at 180°C over 8 h. These nanoparticles were applied as adsorbents for polyacrylamide (PAM) removal from aqueous solutions. Adsorption followed a pseudo-second-order kinetic model and fitted the Langmuir isotherm, with optimal conditions at pH 8, 50 min contact time, and 1.5 g/L dosage, achieving 99% removal efficiency. The Fe₃O₄ NPs were easily recovered using an external magnetic field, highlighting their potential as low-cost, eco-friendly, and reusable adsorbents for water treatment applications.

1 | Introduction

With the rapid advancement of industrial activities, the global demand for oil has markedly increased, leading to a corresponding rise in environmental pollution associated with wastewater discharged from oil production processes. This type of wastewater poses a significant threat to aquatic environments due to its large volume, low biodegradability, high concentrations of organic pollutants, and strong accumulation potential [1]. In oil extraction operations, polyacrylamide (PAM) is extensively utilized and subsequently released into natural water bodies along with production water [2], exerting detrimental effects on the ecological system. PAM is a nonionic, water-soluble organic polymer characterized by poor biodegradability, high viscosity, and inefficient oil–water separation performance, all of which complicate treatment processes and elevate operational costs [3]. Moreover, the polymer backbone of PAM is susceptible to degradation, resulting in the release of acrylamide (AM) monomers. These monomers are known to exert neurotoxic effects on both humans and animals, particularly targeting the peripheral nervous system [4].

Currently, the primary methods employed for the treatment of PAM-containing wastewater include physical adsorption, chemical coagulation, and biodegradation [5–7]. However, these conventional approaches exhibit several limitations, such as low PAM removal efficiency, prolonged treatment duration, complex operational procedures, high costs, and the potential for secondary pollution [8]. It has been reported that particle removal using magnetic fields offers higher selectivity and efficiency compared to traditional techniques such as centrifugation or filtration [9]. In recent years, magnetic materials have attracted considerable attention owing to their high performance, facile synthesis, and derivation from abundant raw materials. Their use also contributes to reduced energy consumption, enhanced nanoparticle biocompatibility, and improved environmental and human health safety [10–12]. These materials have demonstrated broad applicability across diverse fields, including adsorbents, catalysts, capacitors, energy storage systems, and sensors [13, 9, 14–18]. Notably, magnetic materials have shown promising results in catalytic processes and the removal of organic pollutants from aquatic environments [19, 20]. Furthermore, their inherent magnetic properties facilitate easy recovery and reuse, thereby

reducing posttreatment costs [21]. Owing to these advantages, magnetic materials are expected to play an increasingly important role in future environmental and industrial applications.

Green synthesis technology has emerged as a promising alternative to conventional nanoparticle synthesis methods, offering higher efficiency, enhanced selectivity, and scalability [21–25]. This approach employs environmentally friendly and renewable resources—such as plant and microbial extracts—as reducing and stabilizing agents, thereby minimizing the ecological footprint associated with nanoparticle production [26]. Recent studies have successfully demonstrated the synthesis of iron oxide nanoparticles using green routes with natural precursors such as tangerine peel, lemon peel, eucalyptus extract, fenugreek, and honey. These nanoparticles have been effectively applied for the removal of heavy metals from aqueous environments [27]. Notably, a sustainable strategy utilizing *Prosopis farcta* biomass as a precursor has been reported for the green synthesis of magnetic nanoparticles [5, 28]. Such techniques offer advantages in terms of lower energy consumption [26, 27] and efficient thermal control, which contributes to the production of nanoparticles with narrow size distributions [29, 30].

Controlled heating in green synthesis processes limits side reactions, thus enhancing both the efficiency and reproducibility of nanoparticle production [31, 32]. A number of studies have employed plant-based extracts in the synthesis of magnetic nanoparticles. For instance, Koli et al. [33] reported a simple and cost-effective method for synthesizing biocompatible Fe₃O₄ nanoparticles (Fe₃O₄ NPs) with an average particle size of 13 nm. Similarly, Yusefi et al. [34] synthesized Fe₃O₄ nanofluids using crude extracts from *Garcinia mangostana* fruit peel, demonstrating their potential application in magnetic hyperthermia, as the nanofluids were able to induce temperature increases within the safe hyperthermia range (42–47°C) and exhibited acceptable specific absorption rate (SAR) values under alternating magnetic fields.

In the present study, magnetic Fe₃O₄ NPs were synthesized via a green method using green tea (*Camellia sinensis*) leaf extract as both a reducing and stabilizing agent. The physicochemical properties of the synthesized nanoparticles were characterized comprehensively. Furthermore, the study systematically evaluated key parameters influencing the adsorption of PAM, including contact time, adsorbent dosage, initial PAM concentration, and solution pH. Kinetic isotherm analyses of the adsorption process were also conducted. The findings provide new insights into the development and optimization of environmentally sustainable treatment strategies for PAM-laden wastewater generated during petroleum production.

2 | Experimental Procedures

2.1 | Materials

Iron(III) nitrate nonahydrate (Fe(NO₃)₃·9H₂O, ≥98%), sodium hydroxide (NaOH, ≥98%), and ethanol (C₂H₅OH, 96%) were procured from Sigma-Aldrich. Green tea leaves were collected from areas within the university campus. All reagents were of analytical grade and were used without further purification.

Nonionic PAM was supplied by Korona Co. Ltd. Deionized water was utilized in all experiments.

2.2 | Preparation of *Camellia sinensis* Leaf Extract

Fresh *C. sinensis* leaves were thoroughly washed with distilled water to remove surface impurities and subsequently air-dried at room temperature. A total of 35 g of dried leaves were ground and extracted in a Soxhlet apparatus for 4 h using 350 mL of a distilled water–ethanol mixture (1:5, v/v). The resulting extract was filtered, and ethanol was completely evaporated under reduced pressure. The final extract was cooled to room temperature and stored at 4°C for further use.

2.3 | Synthesis of Magnetic Fe₃O₄ NPs

Magnetic Fe₃O₄ NPs were synthesized via a green hydrothermal route. Specifically, 10 mL of 0.1 M Fe(NO₃)₃·9H₂O solution was added to a 20 mL of *C. sinensis* leaf extract and stirred at 800 rpm for 30 min at room temperature. The pH of the solution was adjusted to 8 using 0.1 M NaOH. The resulting mixture was transferred into a Teflon-lined stainless-steel autoclave and subjected to hydrothermal treatment at various temperatures and durations. Upon completion, the reaction mixture was cooled to room temperature. The black precipitate was collected using an external magnet, thoroughly washed with distilled water and ethanol to remove impurities, and dried in a vacuum oven at 65°C for 12 h, yielding magnetic Fe₃O₄ NPs.

2.4 | Adsorption of PAM

A stock solution of PAM was prepared by dissolving 0.1 g of the dry polymer in 100 mL of deionized water at 45°C under continuous stirring to ensure complete solubilization. For each adsorption experiment, 50 mL of PAM solution with a known initial concentration was utilized to evaluate the effects of pH, adsorbent dosage, and adsorption kinetics. A predetermined amount of Fe₃O₄ NPs was introduced into the solution, and the resulting suspension was maintained under constant agitation at a stirring speed of 300 rpm. At designated time intervals, 3 mL aliquots were withdrawn for analysis. Following magnetic separation of the Fe₃O₄ NPs, the residual concentration of PAM in the supernatant was determined using a UV–Vis spectrophotometer at a wavelength of 191 nm. The removal efficiency (η) was calculated according to the following equation:

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

where C_o and C_t (mg/L) are the initial and time-dependent concentrations of PAM, respectively.

2.5 | Kinetics and Isotherm Models

To investigate the adsorption mechanism of PAM onto magnetic Fe₃O₄ NPs, three different kinetic and isotherm models were examined. The Langmuir isotherm model assumes a monolayer

adsorption process occurring on a homogeneous surface with a finite number of identical adsorption sites, each possessing equal affinity for the adsorbate. The linear form of the Langmuir equation is expressed as

$$\frac{C_e}{q_e} = \frac{C_e}{q_{\max}} + \frac{1}{q_{\max}K_L} \quad (2)$$

where C_e is the equilibrium concentration of PAM (mg/L), q_e is the equilibrium adsorption capacity (mg/g), $q_{\max}$ is the maximum adsorption capacity (mg/g), and K_L is the Langmuir constant.

The Freundlich isotherm model describes adsorption on heterogeneous surfaces and assumes that adsorption sites have varying affinities, with stronger binding sites being occupied first. The linear form of the Freundlich equation is given by

$$q_e = K_F C_e^{\frac{1}{n}} \quad (3)$$

where K_F is the Freundlich constant indicative of adsorption capacity ($L^{1/n} \text{mg}^{1-1/n} \text{g}^{-1}$) and $1/n$ is a dimensionless constant that represents the adsorption intensity.

The pseudo-first-order kinetic model is generally applied to describe physical adsorption processes in solid-liquid systems. Its linear form is expressed as

$$\ln \frac{q_e - q_t}{q_e} = -k_1 t \quad (4)$$

where q_e and q_t are the amounts of adsorbate adsorbed at equilibrium and at time t , respectively (mg/g), and k_1 is the pseudo-first-order rate constant (min^{-1}).

The pseudo-second-order kinetic model, on the other hand, assumes that chemisorption is the rate-limiting step, involving valency forces through sharing or exchange of electrons. The linear form is given by

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (5)$$

where k_2 is the pseudo-second-order rate constant (g/mg/min).

2.6 | Characterizations

The crystal structure of the synthesized Fe_3O_4 NPs was analyzed using x-ray diffraction (XRD). Surface morphology was examined using field emission scanning electron microscopy (FE-SEM, Hitachi S-4800-3000F). Fourier-transform infrared (FTIR) spectroscopy (Jasco 4700) was employed to identify functional groups and bonding characteristics. Magnetic properties, including saturation magnetization, were measured using a vibrating sample magnetometer (VSM). The hydrodynamic size and particle size distribution were determined via dynamic light scattering (DLS) using a Malvern Zetasizer Pro (Malvern Panalytical, UK) at 25°C . For this analysis, a 0.1% (w/v) nanoparticle suspension was prepared in deionized water. X-ray photoelectron spectroscopy (XPS) analysis was performed using an ESCALab 250 spectrometer (Thermo VG, UK) to characterize the surface elemental

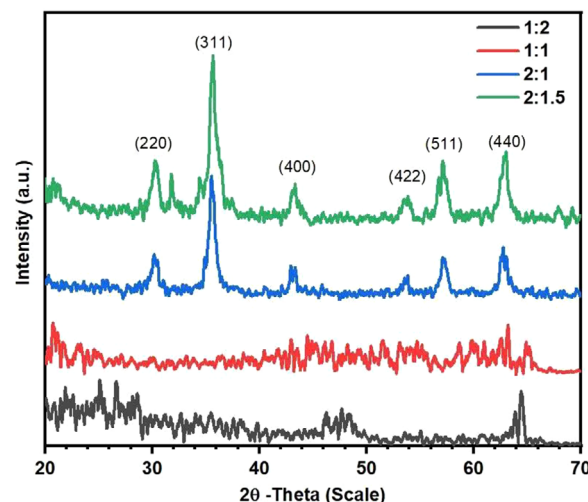


FIGURE 1 | XRD patterns of Fe_3O_4 NPs synthesized with varying *Camellia sinensis* extract to Fe^{3+} precursor volume ratios (1:2, 1:1, 2:1, and 2:1.5, v/v).

composition and corresponding chemical states. The UV-vis diffuse reflectance (UV-vis DRS) spectra were acquired using a UV-2600 spectrophotometer (Shimadzu).

3 | Results and Discussion

3.1 | Structural Characteristics

Figure 1 presents XRD patterns of Fe_3O_4 NPs synthesized using varying volume ratios of *C. sinensis* leaf extract to Fe^{3+} precursor solution, specifically 1:2, 1:1, 2:1, and 2:1.5. The diffraction peaks observed in all samples are in good agreement with the standard diffraction pattern of Fe_3O_4 , corresponding to the (2 2 0), (3 1 1), (4 0 0), (4 2 2), (5 1 1), and (4 4 0) crystal planes (JCPDS card no. 19-0629), confirming the successful formation of the spinel cubic phase of Fe_3O_4 .

Changes in the extract-to-iron ratio were found to influence the relative intensity and sharpness of the diffraction peaks, suggesting variations in crystallinity and particle size. Specifically, the sample synthesized with a 2:1 extract-to- Fe^{3+} ratio exhibited the sharpest and most intense peaks, indicating higher crystallinity and more uniform particle formation. In contrast, lower extract concentrations resulted in broader peaks, possibly due to reduced nucleation control and increased defect formation during the crystallization process. These results demonstrate that the concentration of the green tea extract plays a significant role in modulating the crystal structure and quality of the synthesized Fe_3O_4 NPs.

Figure 2 shows the XRD patterns illustrating the effect of crystallization time on the phase formation of magnetic nanoparticles synthesized at 3, 5, 8, and 12 h. At shorter crystallization durations of 3 to 5 h, the characteristic diffraction peaks corresponding to Fe_3O_4 NPs begin to emerge, indicating the initial formation of the magnetite phase. However, when the crystallization time is extended to 12 h, additional diffraction peaks appear at $2\theta = 37.6^\circ$, which can be attributed to the $\gamma\text{-Fe}_2\text{O}_3$ phase, corresponding to

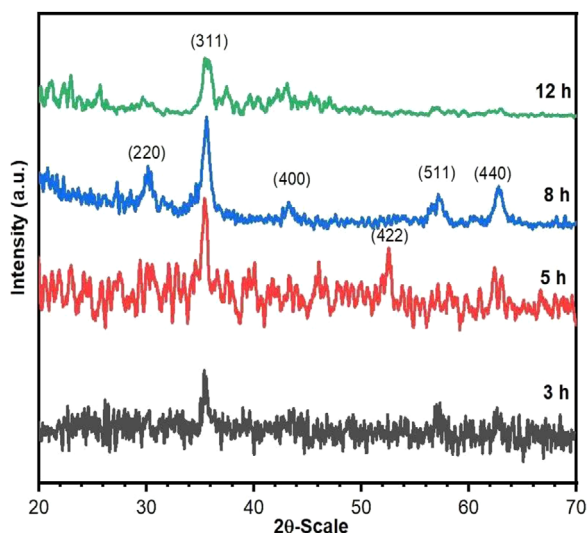


FIGURE 2 | XRD patterns of Fe_3O_4 NPs synthesized at different crystallization times: 3, 5, 8, and 12 h.

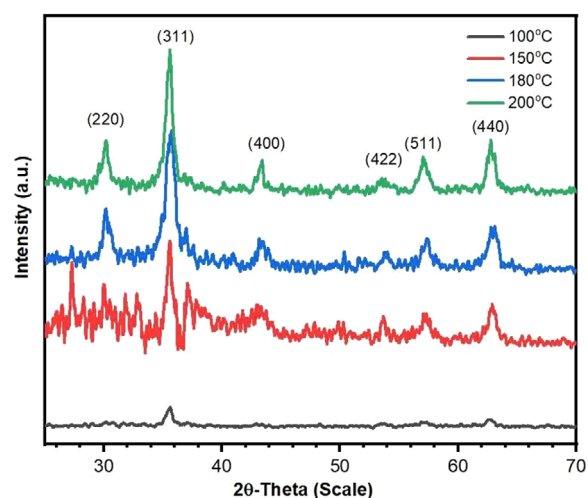


FIGURE 3 | XRD patterns of Fe_3O_4 NPs synthesized at different crystallization temperatures: 100°C, 150°C, 180°C, and 200°C.

the (2 2 2) crystal plane [35]. The presence of this hematite phase at prolonged crystallization times has a significant impact on the magnetic properties of the synthesized Fe_3O_4 NPs.

Temperature is a critical parameter influencing the crystallization behavior of Fe_3O_4 NPs, as it directly impacts both the kinetics and efficiency of the reduction process. The influence of temperature on the formation of Fe_3O_4 magnetic nanocrystals was systematically investigated, and the resulting crystallographic structures were characterized using XRD analysis, as illustrated in Figure 3. As shown in Figure 3, in the temperature range of 100–150°C, characteristic diffraction peaks begin to emerge in the XRD patterns; however, these peaks exhibit low intensity, indicating poor crystallinity and incomplete phase formation. At 180°C, the diffraction peaks become significantly more intense and sharper, suggesting well-defined crystalline structure and higher degree of crystallinity. When the synthesis temperature reaches 200°C, some peak broadening and additional diffraction

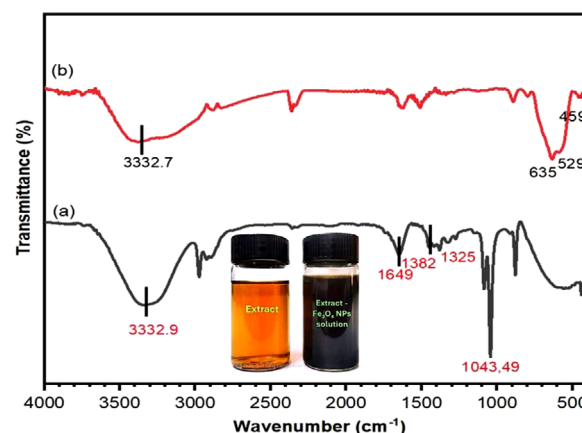


FIGURE 4 | FT-IR spectra of *Camellia sinensis* leaf extract: (a) before the synthesis reaction and (b) after the synthesis reaction (insert: Extract and Extract- Fe_3O_4 NPs solution).

signals are observed, implying the onset of structural distortion or the formation of secondary phases.

The FT-IR spectra of the green tea (*C. sinensis*) extract before and after the synthesis reaction were analyzed to identify the functional groups involved in the reduction and stabilization of Fe_3O_4 NPs (Figure 4). A broad absorption band observed at 3332.9 cm^{-1} corresponds to the stretching vibrations of O–H groups, indicating the presence of hydroxyl-containing compounds. The distinct peak at 1649.11 cm^{-1} is attributed to the C=C stretching vibrations within aromatic rings, suggesting the presence of polyphenolic compounds. Additional peaks at 1325 and 1382 cm^{-1} are characteristic of N–H bending vibrations of aromatic amines. The absorption band at 1043.49 cm^{-1} is associated with C–N stretching vibrations of aliphatic amines. These findings confirm the presence of phenolic and amine-based functional groups in the green tea extract, which likely play a key role in the reduction of iron ions and stabilization of the synthesized Fe_3O_4 NPs.

FT-IR spectra of *C. sinensis* leaf extract: (a) before the synthesis reaction and (b) after the synthesis reaction. The broad band around 3332.9 cm^{-1} corresponds to O–H stretching vibrations, whereas the peak at 1649.1 cm^{-1} is attributed to C=C stretching in aromatic rings, indicating the presence of polyphenolic compounds. Peaks at 1325 and 1382 cm^{-1} are associated with N–H bending of aromatic amines, and the band at 1043.5 cm^{-1} corresponds to C–N stretching of aliphatic amines. After the reaction, a new absorption peak at 459 cm^{-1} appears, confirming the formation of Fe–O bonds in the synthesized Fe_3O_4 NPs [34].

On the basis of the XRD analysis, the theoretical crystallite size of the synthesized Fe_3O_4 NPs was estimated using the Scherrer equation. However, SEM analysis provides more direct and visual insight into the actual morphology and particle size of the synthesized material. The results indicate that Fe_3O_4 NPs exhibit a cubic morphology, and the crystallite size, as determined using the Scherrer equation, is approximately 20 nm.

The SEM images reveal that the Fe_3O_4 NPs exhibit relatively uniform morphology with a predominantly spherical shape

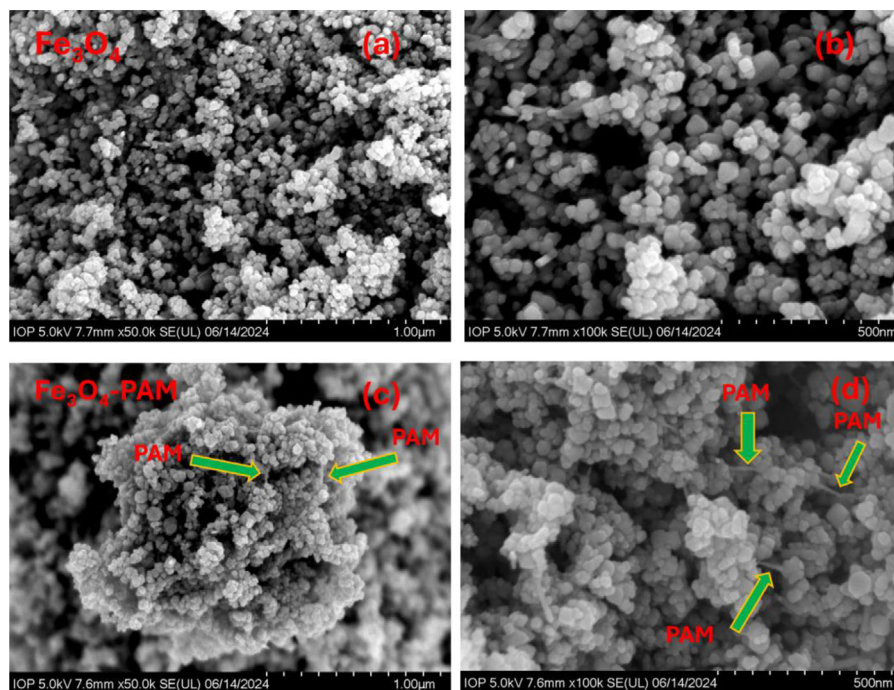


FIGURE 5 | (a) and (b) SEM images of pure Fe_3O_4 , and (c) and (d) PAM adsorption onto nano- Fe_3O_4 . PAM, polyacrylamide.

(Figure 5a,b). The particle size observed from the SEM micrographs is generally consistent with the crystallite size estimated from XRD, although slight aggregation is present, which is typical for magnetite nanoparticles due to their magnetic interactions. These results confirm the successful formation of nanoscale Fe_3O_4 particles via the green synthesis route. Figure 5c,d shows the surface morphology of Fe_3O_4 NPs after PAM adsorption, indicating the attachment of PAM onto the Fe_3O_4 surface, which may be attributed to electrostatic interactions. It is also noteworthy that the PAM chains adhere without any apparent surface alteration, suggesting a simple adsorption process rather than mineralization, thereby avoiding secondary contamination.

The magnetic behavior of the synthesized Fe_3O_4 NPs was investigated using VSM at room temperature. As shown in Figure 6, the magnetization curve reveals negligible coercivity ($H_c \approx 0$ Oe) and remanent magnetization ($M_r \approx 0$ emu/g), suggesting that the nanoparticles exhibit superparamagnetic behavior. This phenomenon is commonly observed in Fe_3O_4 NPs with diameters below the critical size for multi-domain formation, where thermal fluctuations are sufficient to overcome magnetic anisotropy energy and randomize the magnetic moments in the absence of an external field [36]. The measured saturation magnetization (M_s) was approximately 50 emu/g, which is lower than the value reported for bulk Fe_3O_4 (~ 92 emu/g) [37]. This reduction in M_s can be attributed to surface spin disorder, surface oxidation, or finite-size effects that commonly occur in nanoparticles [37]. These findings are consistent with previous studies on superparamagnetic Fe_3O_4 nanostructures synthesized via chemical methods [38, 39], further confirming the successful synthesis of nanoscale Fe_3O_4 with size-dependent magnetic behavior.

The Fe 2p XPS spectrum of Fe_3O_4 is presented in Figure 7a. In this spectrum, the principal binding-energy peak appears

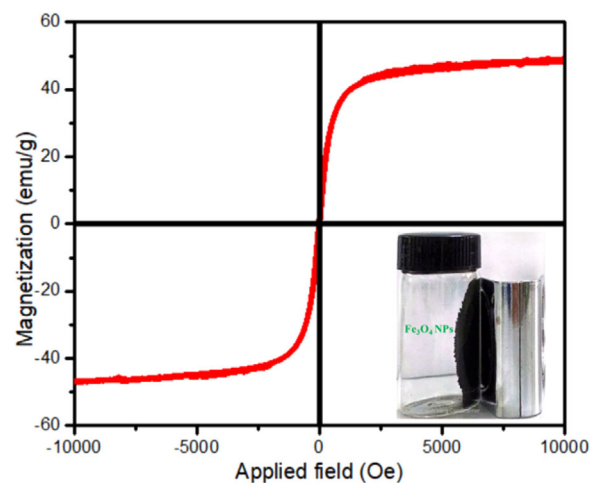


FIGURE 6 | Magnetic hysteresis of Fe_3O_4 NPs (Inset: image of Fe_3O_4 NPs sample).

at approximately 710.6 eV, corresponding to the Fe $2p_{3/2}$ state, whereas the peak located at 724–725 eV is assigned to the Fe $2p_{1/2}$ state. These features are consistent with the presence of shake-up satellites and two spin–orbit doublets, indicating the coexistence of both Fe^{2+} and Fe^{3+} species distributed in octahedral and tetrahedral coordination sites [40]. The lowest energy component at 710.2 eV is attributed to Fe^{2+} , accompanied by its satellite at 717.08 eV. Octahedrally coordinated Fe^{3+} is observed at a binding energy of 710.6 eV, whereas tetrahedral Fe^{3+} appears at 713.3 eV. These values are in good agreement with those reported in the literature [41, 42].

Furthermore, the O 1s XPS spectrum of Fe_3O_4 NPs (Figure 7b) reveals two characteristic peaks: The main peak at 529.8 eV

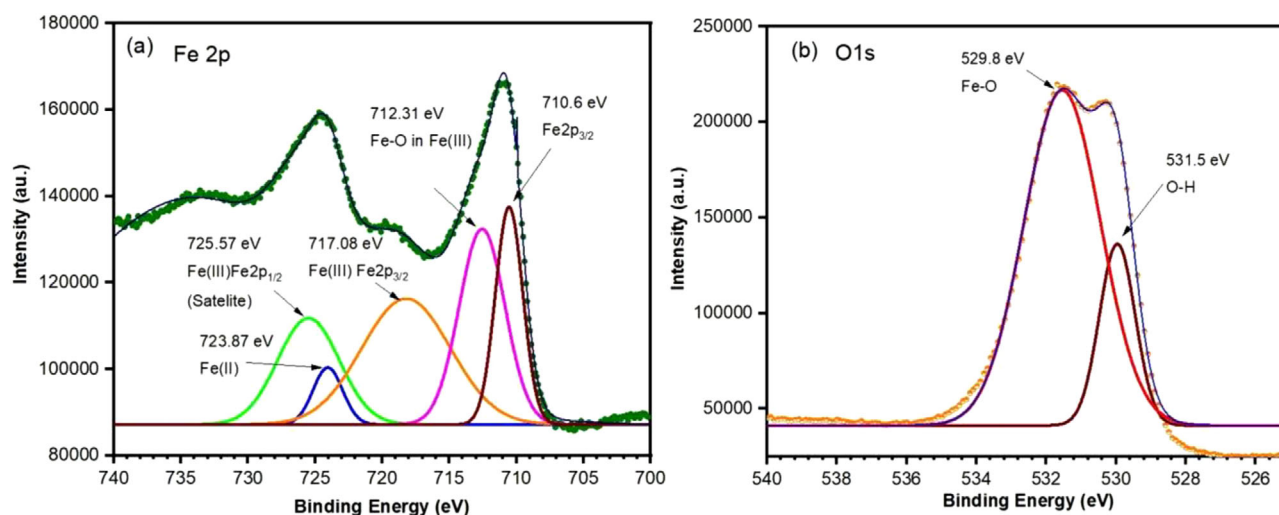


FIGURE 7 | XPS of Fe_3O_4 NPs (a) Fe 2p and (b) O 1s.

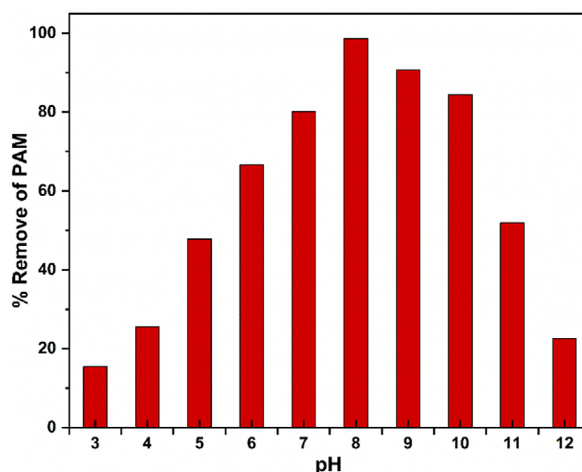


FIGURE 8 | Effect of pH on the adsorption efficiency of PAM onto Fe_3O_4 NPs (30 ppm PAM, 1.5 g/L Fe_3O_4 NPs, contact time of 50 min, temperature of 27°C). PAM, polyacrylamide.

corresponds to lattice oxygen (Fe—O) in the magnetite structure, whereas the signal at 531.5 eV is associated with surface hydroxyl groups (O—H) and/or adsorbed water [43].

3.2 | Adsorption Studies

3.2.1 | Effect of pH on PAM Adsorption Efficiency

Adsorption experiments were conducted at varying pH levels using 35 mL of PAM solution (30 ppm) and 1.5 g/L of Fe_3O_4 NPs as the adsorbent at room temperature (27°C). The pH of the solution was adjusted within the range of 3–12 using 0.01 M HCl and 0.01 M NaOH solutions. The adsorption of PAM onto Fe_3O_4 NPs was found to be strongly pH-dependent.

Maximum adsorption efficiency of 98.7% was observed at pH = 8.4 (Figure 8). In acidic conditions, a significant decrease in adsorp-

tion efficiency was recorded: at pH = 5.0, the efficiency dropped to 51.5% and further decreased to 15.5% at pH = 3.5. Similarly, in highly alkaline environments, the adsorption performance also declined markedly, with efficiencies of 84.4% and 22.5% observed at pH = 10 and pH = 12, respectively.

These results suggest that the surface charge of the Fe_3O_4 NPs and the ionization state of PAM play critical roles in the adsorption process. The optimal adsorption observed at near-neutral to mildly basic conditions may be attributed to enhanced electrostatic interactions between the negatively charged PAM molecules and the positively charged sites on the nanoparticle surface.

Figure 9a shows the zeta potential of PAM, measured at -63.9 mV, indicating that the polymer particles possess a negatively charged surface and exhibit good electrokinetic stability. The high magnitude of the negative zeta potential suggests strong interparticle repulsion, which prevents aggregation and promotes colloidal stability. The average hydrodynamic diameter (Z-average) of PAM particles, obtained from DLS analysis, was approximately 354.5 nm (Figure 9b).

At low pH values (acidic conditions), the carboxylate groups ($-\text{COO}^-$) of PAM become protonated to form $-\text{COOH}$, which reduces the negative surface charge. This results in a decrease in zeta potential toward zero, increasing the likelihood of particle agglomeration and precipitation due to reduced electrostatic repulsion. Conversely, under strongly basic conditions (pH > 10), functional groups on the polymer may undergo degradation or conformational changes, leading to irregular fluctuations in zeta potential and destabilization of the colloidal system.

In near-neutral to mildly alkaline conditions (pH 7–10), the carboxyl groups on PAM are almost fully ionized to $-\text{COO}^-$, resulting in a highly negative zeta potential. This electrostatic repulsion stabilizes the colloidal system and prevents aggregation, thereby enhancing adsorption efficiency—reaching a maximum of approximately 99% at pH 8.

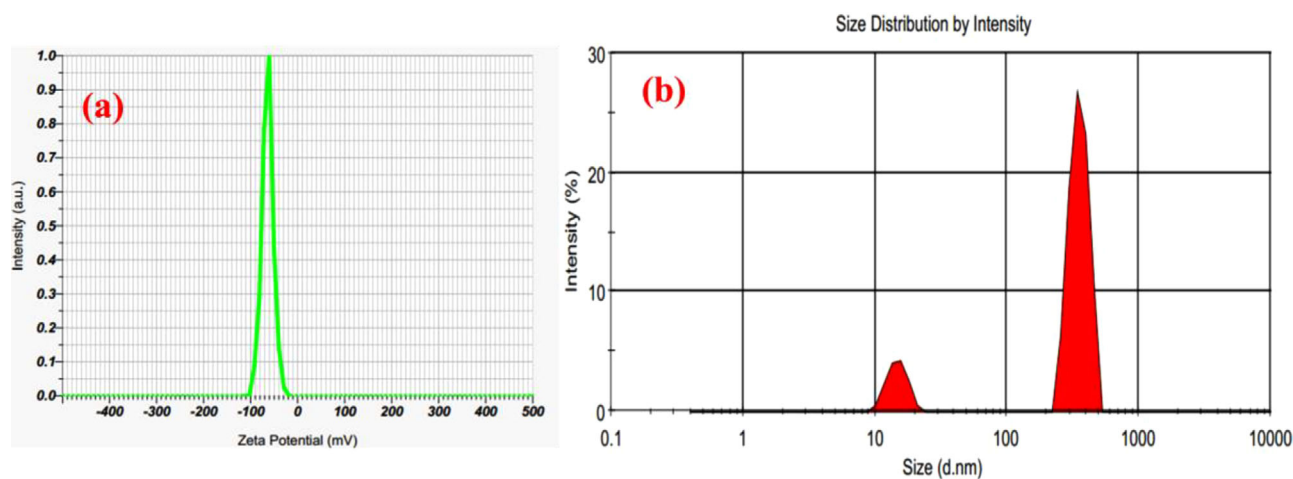


FIGURE 9 | (a) Zeta potential (ζ) and (b) particle size (Z-average) of 30 ppm PAM solution.

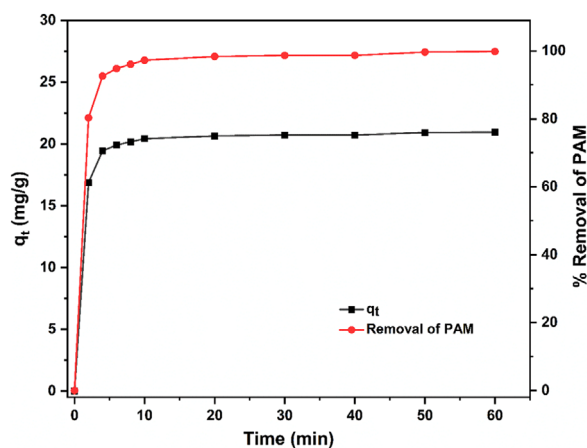


FIGURE 10 | Effect of contact time on PAM adsorption efficiency (30 ppm PAM, 1.5 g/L Fe_3O_4 NPs, temperature of 27°C, and pH = 8). PAM, polyacrylamide.

3.2.2 | Effect of Contact Time on PAM Adsorption Efficiency

As presented in Figure 10, the adsorption of PAM onto Fe_3O_4 NPs proceeded rapidly during the initial stage (0–10 min), primarily due to the abundance of available active sites on the nanoparticle surface. In the subsequent period (10–50 min), a gradual saturation of these active sites was observed, leading to increased competition among PAM molecules for the remaining adsorption sites and consequently a reduction in the adsorption rate. This trend can be attributed to the high molecular weight of PAM, which limits its ability to penetrate deeply into the agglomerated structures of the nanoparticles. In the final stage (50–60 min), the adsorption process approached equilibrium, with approximately 99% of PAM being adsorbed onto the Fe_3O_4 surface.

3.2.3 | Effect of Fe_3O_4 NPs Dosage on Adsorption Efficiency

The influence of varying Fe_3O_4 NPs dosages (0.25, 0.5, 1.0, 1.5, and 2.0 g/L) on the adsorption efficiency of PAM was investigated at

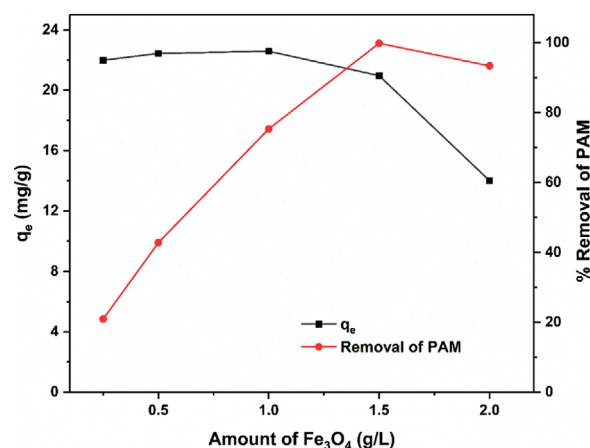


FIGURE 11 | Effect of Fe_3O_4 NPs dosage on PAM adsorption efficiency (30 ppm PAM, contact time of 50 min, temperature of 27°C, and pH = 8). PAM, polyacrylamide.

an initial PAM concentration of 30 ppm, temperature of 27°C, and a contact time of 50 min. The results are presented in Figure 11.

An increase in adsorption efficiency from 20.94% to 42.74% was observed as the Fe_3O_4 NPs dosage increased from 0.25 to 0.5 g/L, indicating that the initial surface area was insufficient to adsorb all available PAM molecules. As the adsorbent dosage was further increased to 1.0 g/L, the efficiency rose to 75.27%, eventually reaching a maximum of 99.82% at 1.5 g/L. This enhancement is attributed to the increase in total surface area and the corresponding rise in the number of accessible active sites, such as $-\text{OH}$ and $-\text{FeOH}_2^+$ groups on the nanoparticle surface. At 1.5 g/L, the quantity of adsorbent was sufficient to capture nearly all PAM molecules present in the solution. However, a further increase in Fe_3O_4 dosage to 2.0 g/L resulted in a decline in adsorption efficiency to 93.25%. This reduction is likely due to the agglomeration of Fe_3O_4 NPs at higher concentrations, driven by magnetic or van der Waals interactions, which reduces the effective surface area available for PAM adsorption. Additionally, at elevated dosages, particle–particle shielding may occur, wherein nanoparticles obscure each other, limiting access to active adsorption sites. Furthermore, increasing the Fe_3O_4

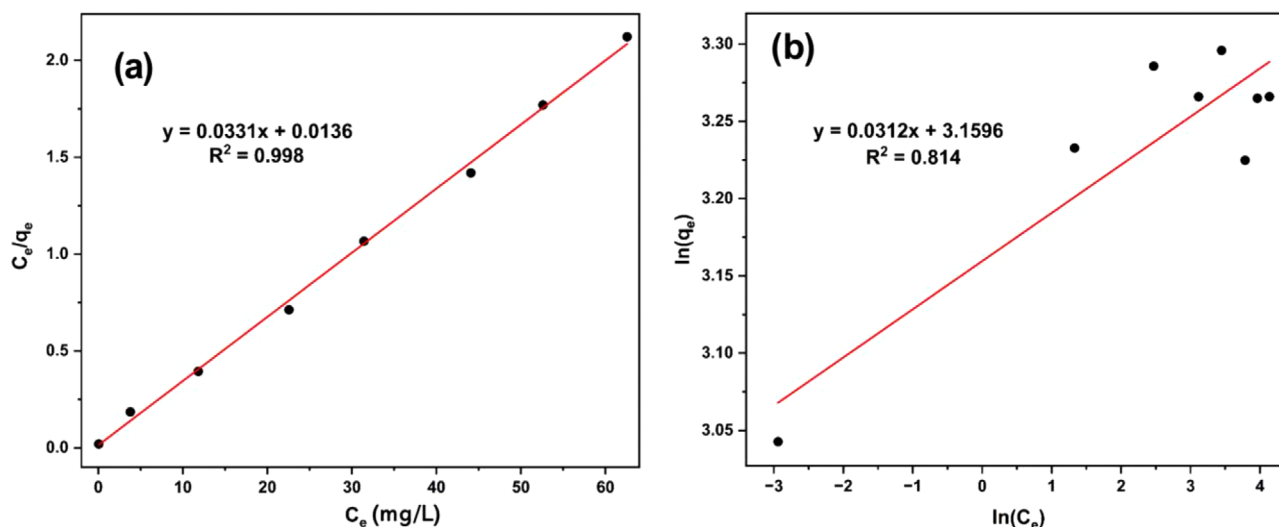


FIGURE 12 | Langmuir (a) and Freundlich (b) isotherm plots for PAM adsorption onto Fe_3O_4 NPs (1.5 g/L of Fe_3O_4 NPs dosage, contact time of 50 min, temperature of 27°C , and pH = 8).

TABLE 1 | Adsorption isotherm constants for polyacrylamide (PAM) adsorption onto Fe_3O_4 NPs.

Langmuir			Freundlich		
$q_{\max}$ [mg/g]	K_L [L/mg]	R^2	K_F [$\text{L}^{1/n} \text{mg}^{1-1/n} \text{g}^{-1}$]	$1/n$	R^2
30.21	2.43	0.99	23.56	0.0312	0.814

concentration may alter the pH of the solution or the system's zeta potential, thereby diminishing electrostatic interactions between Fe_3O_4 and PAM.

3.2.4 | Adsorption Isotherm

The Langmuir isotherm plot for PAM adsorption onto Fe_3O_4 NPs is presented in Figure 12, and the corresponding isotherm parameters are summarized in Table 1.

The correlation coefficient R^2 for the Langmuir isotherm model was found to be 0.99, indicating that the adsorption process of PAM onto Fe_3O_4 NPs follows the Langmuir isotherm model closely. The maximum monolayer adsorption capacity $q_{\max}$ of Fe_3O_4 NPs was determined to be 30.21 mg/g at ambient temperature (27°C). This adsorption capacity is comparable to or exceeds that of hybrid adsorbent materials such as PAM/montmorillonite (32.8 mg/g for Pb^{2+}) [44] and PAM/nano- TiO_2 (~ 26.5 mg/g for Cr(VI)) [45]. These findings highlight the significant potential of Fe_3O_4 NPs as an effective adsorbent for wastewater treatment applications.

The linear form of the Freundlich isotherm equation derived from Equation (5) is expressed as follows:

$$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e \quad (6)$$

The Freundlich constants were calculated from the plot shown in Figure 10b and are summarized in Table 1. The correlation coefficient $R^2 = 0.814$ indicates that the experimental data do not fit well with the Freundlich isotherm model.

3.2.5 | Adsorption Kinetics

Kinetic experiments were conducted to determine the adsorption model describing the interaction between PAM and Fe_3O_4 NPs. Among the two kinetic models evaluated, the pseudo-second-order kinetic model exhibited a better fit to the experimental data, with a correlation coefficient $R^2 = 0.999$ (Figure 13 and Table 2).

The values of k_1 and q_e obtained from the plot in Figure 13a and Table 2 show a low correlation coefficient $R^2 = 0.889$. Moreover, the experimental adsorption capacity $q_{e,\text{exp}}$ (mg/g) significantly differs from the calculated value $q_{e,\text{cal}}$ (mg/g). These observations indicate that the pseudo-first-order kinetic model does not adequately describe the adsorption of PAM onto Fe_3O_4 NPs. In contrast, the pseudo-second-order kinetic model exhibits a high correlation coefficient $R^2 = 0.999$ (Figure 12b), suggesting that the adsorption process of PAM on Fe_3O_4 NPs follows this model. Additionally, the calculated adsorption capacities $q_{e,\text{cal}}$ closely match the experimental values $q_{e,\text{exp}}$. Similar studies investigating the adsorption of Pb^{2+} and Cd^{2+} using Fe_3O_4 NPs coated with SiO_2 and PAM support these findings. Kinetic data were evaluated using both pseudo-first-order and pseudo-second-order models. The results demonstrated that the pseudo-second-order model provided a better fit with a higher correlation coefficient ($R^2 \approx 0.99$) compared to the pseudo-first-order model ($R^2 < 0.94$). Furthermore, the calculated q_e values from the pseudo-second-order model showed excellent agreement with the experimental data [46].

The magnetic Fe_3O_4 NPs coated with PEG were used for the adsorption of methylene blue (MB). Kinetic results showed that the pseudo-first-order model yielded a correlation coefficient $R^2 \approx 0.937$, whereas the pseudo-second-order model exhibited a

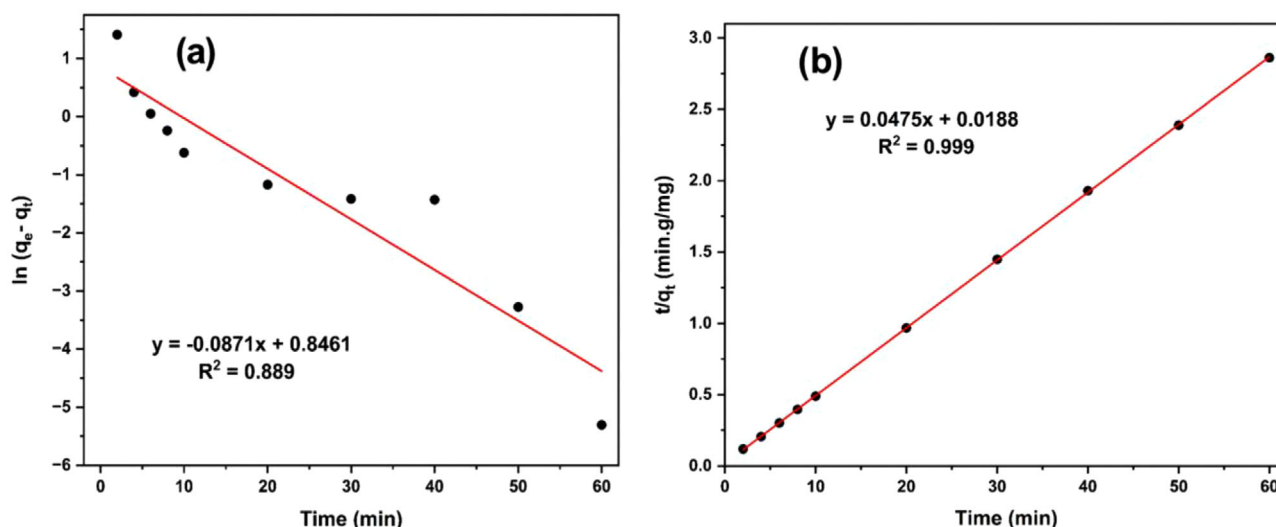


FIGURE 13 | Kinetic model plots for PAM adsorption onto Fe_3O_4 NPs at pH = 8, temperature = 27°C , 1.5 g/L of Fe_3O_4 NPs dosage, and initial PAM concentration of 30 ppm: (a) pseudo-first-order model and (b) pseudo-second-order model.

TABLE 2 | Kinetic parameters for polyacrylamide (PAM) adsorption onto Fe_3O_4 NPs.

Pseudo first order				Pseudo second order			
$C_t = C_o e^{-k_1 t}$				$\frac{1}{C_t} = k_2 t + \frac{1}{C_o}$			
R^2	k_1 [min^{-1}]	$q_{e, \text{exp}}$ [mg/g]	$q_{e, \text{cal}}$ [mg/g]	R^2	k_2 [min^{-1}]	$q_{e, \text{exp}}$ [mg/g]	$q_{e, \text{cal}}$ [mg/g]
0.889	0.0889	20.96	2.416	0.999	0.120	20.96	21.053

perfect fit with $R^2 = 1.0$. The pseudo-second-order model provided an excellent agreement between the calculated adsorption capacity q_e and the experimental value $q_{e, \text{exp}}$ [47]. Magnetite combined with chitosan was employed for the adsorption of Orange I dye. The plots and kinetic parameters from both pseudo-first-order and pseudo-second-order models indicated that the pseudo-second-order model provided a better fit, with a correlation coefficient of approximately $R^2 = 0.9561$. Therefore, the pseudo-second-order kinetic model is more appropriate for describing the adsorption process [48].

3.2.6 | Adsorption Mechanism

On the basis of the adsorption model and the previous discussion, the predominant adsorption mechanism is physical adsorption driven by the electrostatic interaction between the negatively charged $-\text{COO}^-$ groups of PAM and the positively charged Fe_3O_4 surface plays a crucial role in the adsorption process. This strong attraction facilitates the rapid and efficient capture of PAM molecules onto the nanoparticle surface, significantly enhancing the overall adsorption capacity. The high negative zeta potential of PAM (-63.9 mV) ensures strong repulsion among PAM molecules, preventing aggregation and promoting better dispersion, which further contributes to increased accessibility of adsorption sites. Additionally, hydrogen bonding between the $-\text{CONH}_2$ groups of PAM and the surface hydroxyl groups ($-\text{OH}$) of Fe_3O_4 provides additional binding forces that stabilize the

adsorbed molecules. This interaction not only strengthens the retention of PAM on the nanoparticle surface but also improves the selectivity toward PAM over other competing species in aqueous solutions. Furthermore, weak complexation or mild chemical interactions between functional groups of PAM and $\text{Fe}^{3+}/\text{Fe}^{2+}$ ions on the Fe_3O_4 surface may contribute to a more robust and durable adsorption. Although these interactions are less dominant compared to electrostatic forces, they enhance the binding stability, particularly under varying environmental conditions such as pH and ionic strength. Together, these interactions synergistically improve the adsorption efficiency of PAM onto Fe_3O_4 NPs, leading to a high adsorption capacity and stability, which are essential for practical applications in water treatment.

4 | Conclusions

In this study, magnetic Fe_3O_4 NPs were synthesized via an environmentally friendly method utilizing green tea leaf extract, a renewable and abundant natural resource. The synthesized Fe_3O_4 NPs exhibit eco-friendly characteristics, with their magnetic properties enabling efficient separation from mixtures, thereby preventing secondary environmental contamination. Increasing the adsorbent dosage (1.5 g/L of Fe_3O_4 NPs) and operating under neutral to mildly alkaline conditions (pH = 8) enhanced the adsorption rate, which reached its maximum within 50 min. These results support the potential application of plant-derived magnetic nanoadsorbents for water and wastewater treatment,

particularly for the removal of residual organic pollutants. Future studies could focus on the integration of photocatalytic–magnetic materials activated by solar irradiation to further elucidate the efficiency of these nanoparticles in eliminating various contaminants.

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Conflicts of Interest

The authors declare no conflicts of interest.

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