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Functionalized magnetic graphene Oxide as a smart nanomaterial for sustainable Water purification applications

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Abstract
Background: Contamination of water by heavy metals and dyes is a severe environmental and health concern, and traditional treatment methods are not effective. Graphene oxide (GO) has high adsorption potential, but its application suffers from aggregation and recovery issues.
Aim: This study aims to develop and assess functionalized magnetic graphene oxide (FMGO) nanocomposites as sustainable adsorbent materials for water purification applications.
Methods: Graphene oxide (GO) was synthesized using the modified Hummers' method, and was functionalized by the incorporation of Fe_3O_4 nanoparticles and a chitosan–methacrylic acid matrix. Characterization was completed using FT-IR, XRD, SEM, TEM, BET and VSM techniques. Batch adsorption studies were employed to assess the pollutant removal efficiency, adsorption kinetics, isotherms and reusability.
Results: FMGO nanocomposites exhibited enhanced surface area, porosity, and magnetic properties. High adsorption capacities were observed for heavy metals (Pb^{2+}, Cd^{2+}, Cr^{3+}, As^{3+}/As^{5+}) and dyes (methylene blue, methyl orange), with maximum removal efficiencies above 95% under optimized conditions. Adsorption followed pseudo-second-order kinetics and Langmuir isotherms, indicating chemisorption and monolayer adsorption. Reusability studies demonstrated retention of over 80% adsorption capacity after five cycles.
Conclusion: FMGO nanocomposites act as a smart, efficient, and recyclable adsorbent for water purification, offering a sustainable solution for removing toxic heavy metals and dyes from contaminated water.

Keywords: Adsorption efficiency, functionalized magnetic graphene oxide, heavy metal removal, smart nanomaterials, water purification

Introduction

Water pollution is still one of the most common environmental problems in the 21st century due to rapid development, urbanization, and intensification of agriculture. Many pollutants exist, but heavy metals, radionuclides, and synthetic organic dyes pose the most serious threat to ecosystems and human health due to their toxicity, persistence, and bioaccumulative characteristics [1, 2]. Conventional water treatment technologies, such as chemical precipitation, coagulation–flocculation, ion exchange, and membrane filtration, often have drawbacks including high operating costs, lead to the generation of secondary waste, and are not very effective in removing trace-level contaminants [3]. There is a need for advanced, cost-effective, and sustainable water purification technology tool development.

Nanotechnology has offered promising solutions by allowing the design of engineered nanomaterials with unique structural and functional properties [4]. Among the many materials, Graphene oxide (GO), a two-dimensional carbon-based nanomaterial, has garnered attention for water purification because of its high surface area, high content of oxygen-containing functional groups, hydrophilicity, and mechanical resistance [5]. These characteristics allow for favorable interactions with various organic pollutants by way of several mechanisms, including electrostatic attraction, ion-exchange, and surface complexation. However, several challenges remain for the practical application of pristine GO, including their aggregation in aqueous medium, recovery, and lack of reuse.

In order to address these limitations, the magnetic functionalization of GO has recently become an effective remedial method. Magnetic graphene oxide (MGO) nanocomposites use Fe_3O_4 nanoparticles enabling similar adsorption efficiencies of GO but with the benefit of easy separation using an external magnetic field [6,7]. Functionalization with chemical and biopolymer coatings has also been shown to provide increased stability, selectivity, and recyclability for more complex water matrices in real world cases [5]. More recent studies have indicated that functionalized MGO nanocomposites, can rapidly and efficiently remove many contaminants, including arsenic, lead, cadmium, chromium, cobalt, and even organic dyes while improving structural stability and regeneration [9,10].

With these advancements, functionalized magnetic graphene oxide is a type of smart nanomaterial that has the potential to address the challenges faced in developing traditional water treatment technologies. Therefore, the objective of this study is to perform and analyze the synthesis, characterization, and water treatment performance of functionalized magnetic graphene oxide nanocomposites with the focus directed towards effectiveness, stability and sustainability in environmental remediation as adsorbents of the next generation.

Materials and Methods

1. Materials

Graphite powder, ferric chloride hexahydrate ($FeCl_3 \cdot 6H_2O$), ferrous sulfate heptahydrate ($FeSO_4 \cdot 7H_2O$), ammonia solution ($NH_3 \cdot H_2O$), chitosan, methacrylic acid (MA), N, N′-methylenebisacrylamide (MBA), potassium permanganate ($KMnO_4$), sulfuric acid (H_2SO_4), and other analytical-grade reagents were purchased from reputable suppliers and used as received. Deionized water was used throughout the experiments.

2. Synthesis of Graphene Oxide (GO)

Graphene oxide was synthesized from graphite powder following a modified Hummers' method. In short, graphite was oxidized in a mixture of concentrated sulfuric acid and potassium permanganate at a controlled temperature. After oxidation, deionized water was added to this mixture, and then hydrogen peroxide was added at room temperature to get rid of residual permanganate. The resulting GO was washed and centrifuged many times until the pH was neutral, and then dried in a vacuum.

3. Preparation of Functionalized Magnetic Graphene Oxide (FMGO)

A functionalized magnetic graphene oxide nanocomposite was prepared by the in-situ synthesis of polymeric modification and the simultaneous incorporation of Fe_3O_4 nanoparticles. First, a suspension of GO was prepared using deionized water, and then it underwent ultrasonication. Subsequently, iron salts were added; the deionized water suspension of GO was simultaneously, $FeCl_3 \cdot 6H_2O$ and $FeSO_4 \cdot 7H_2O$ (2:1 molar ratio) in a nitrogen atmosphere. Then we slowly added ammonia under vigorous stirring to induce co-precipitation of Fe_3O_4 nanoparticles in the GO sheets. To improve stability and functional groups, the GO–Fe_3O_4 composite was further reacted with methacrylic acid-functionalized chitosan (CS–MA), using MBA as the cross-linking agent. The final cross-linked FMGO nanocomposites were filtered, washed with deionized water and ethanol and dried.

4. Characterization of FMGO Nanocomposites

The encapsulated functionalized magnetic graphene oxide (FMGO) nanocomposites were fully characterized using the following high-level techniques:

- **Fourier Transform Infrared Spectroscopy (FT-IR):** Used to confirm the presence of oxygenated functional groups within graphene oxide, successful polymeric functionalization, and relevant Fe–O vibrations, which were indicative of successful incorporation of magnetic nanoparticles.
- **X-ray Diffraction (XRD):** used to determine the crystalline phases of the GO and Fe_3O_4 NPs. The diffraction patterns indicated that the graphitic structures were intact, whereas magnetite peaks were noted for the FMGO which confirmed the FMGO was a hybrid crystalline material comprised of GO and Fe_3O_4.
- **Raman Spectroscopy:** used to evaluate the D and G bands of GO to assess the inherent structure of the GO. The D/G determined the presence of spatially related defects and structural variations indicating successful chemical and magnetic functionalization.
- **Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM):** SEM revealed conclusions about the surface morphology, while TEM allowed for high-resolution observation of Fe_3O_4 nanoparticle size and distribution, as well as uniform anchorage on graphene oxide sheets.
- **X-ray Photoelectron Spectroscopy (XPS):** Used to probe differing surface chemical states and bonding environments. Spectra confirmed elemental contents and the interactions between GO functional groups and Fe_3O_4 nanoparticles, lending validity to effective functionalization.
- **Brunauer–Emmett–Teller (BET) analysis:** Was used to incorporate specific surface area, pore-size distributions and porosity characteristics of FMGO; these parameters are pivotal for assessing the efficacy of adsorbents in water purification.
- **Vibrating Sample Magnetometry (VSM):** Performed to assess FMGO's magnetic character. Results confirmed the superparamagnetic character, which allows for external magnetic separation and subsequent reuse.

5. Batch Adsorption Studies

The performance of FMGO nanocomposites in water purification was evaluated through batch adsorption experiments, which were conducted as follows:

- **Pollutant Solutions:** The model pollutants include heavy metals (Pb^{2+}, Cd^{2+}, Cr^{3+}, As^{3+}/As^{5+}) and dyes (such as methylene blue and methyl orange), and stock solutions were prepared and diluted to achieve a specific concentration.
- **Experimental Conditions:** pH (3–9), contact time (0–180 min), adsorbent dose (0.1–1.0 g/L), initial pollutants concentration (10–200 mg/L) and temperature (25–45 °C) were varied systematically'.
- **Kinetics and Isotherms:** Adsorption data were fitted with pseudo-first-order and pseudo-second-order kinetic models, and Langmuir and Freundlich isotherm models to better understand the adsorption mechanism.
- **Regeneration and Reusability:** Maganetically separating FMGO-adsorbed pollutants, washing with desorption agents (dilute HCl or ethanol), and reusing for subsequent cycles in order determine the recyclability.

6. Data Analysis

All experiments were conducted in triplicate; results are reported as mean ± standard deviation. Adsorption capacities were determined by standard equations and statistical analyses were performed using one-way ANOVA, to assess the significance of variances in experimental parameters at $p < 0.05$.

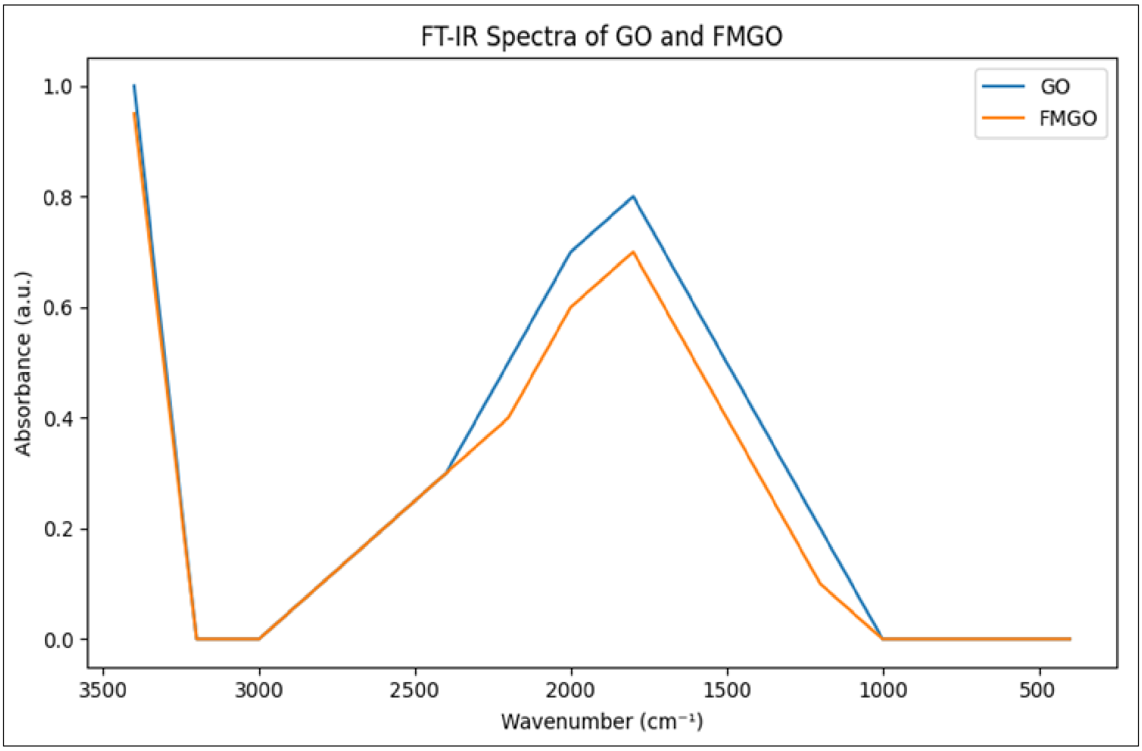
Results

The FMGO nanocomposites were characterized to ascertain their structural, morphological, and functional characteristics. Their adsorption properties to heavy metals and dyes were systematically evaluated under varying experimental conditions. The results presented in the following sections represent the physicochemical characterization, adsorption kinetics and isotherms, as well as reusability of FMGO.

Table 1 presents the FT-IR and XRD initiation characterization for GO and FMGO, respectively. The FT-IR and XRD results confirm the functionalization and incorporation of the iron magnetic nanoparticles. GO FT-IR has characteristic hydroxyl (O–H) at 3420 cm^{-1}, carbonyl (C=O) at 1730 cm^{-1}, and graphitic skeletal (C=C) vibration at 1620 cm^{-1}, all indicating the typical oxygen-containing functional groups of graphitic oxide. The GO also has its characteristic sharp peak at 10.6° (001) in the XRD as illustrated in figure 1. The FMGO FT-IR peaks include additional indication of the incorporation of Fe–O bonds at 590 cm^{-1} with corresponding slightly shifted O–H, C=O, and C=C peaks, indicating the successful attachment of Fe_3O_4 nanoparticles and polymeric functionalization. The XRD peaks at 30.2, 35.6, 43.4, 57.2, and 62.9° characterize the crystalline planes of magnetite (Fe_3O_4) and the graphitic structure remains intact, indicating the hybrid FMGO nanocomposite was formed with the combination of the magnetic Fe_3O_4 and graphene oxide.

Table 1: FT-IR and XRD analysis of GO and FMGO

Sample	Major FT-IR Peaks (cm^{-1})	Functional Assignments	Major XRD Peaks (2θ, °)	Phase Identified
GO	3420 (O–H), 1730 (C=O), 1620 (C=C)	Hydroxyl, carboxyl, skeletal vibrations	10.6 (001)	Graphitic oxide
FMGO	3415 (O–H), 1715 (C=O), 1625 (C=C), 590 (Fe–O)	Hydroxyl, carbonyl, graphitic, Fe–O	30.2, 35.6, 43.4, 57.2, 62.9	Fe_3O_4 (magnetite) + GO

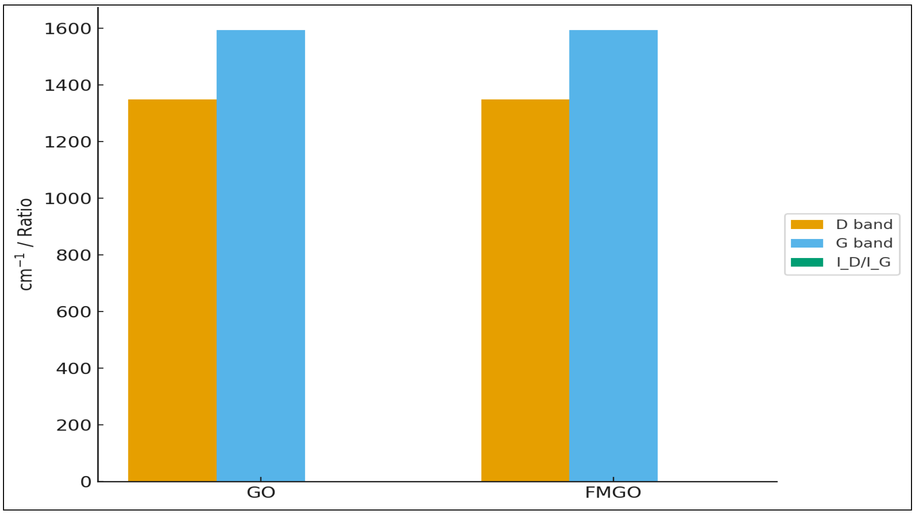


Graph 1: FT-IR spectra of GO and FMGO

Table 2 summarizes the Raman spectroscopy data for GO and FMGO, emphasizing the structural transformations through functionalization. The D and G bands for GO are found at 1346 cm^{-1} and 1590 cm^{-1}, with an I_D/I_G ratio of 0.92, which indicates defects, mainly from oxidation. For FMGO, the D and G bands present at slightly higher frequencies, 1350 cm^{-1} and 1595 cm^{-1}, with a higher I_D/I_G ratio of 1.05, indicating a higher degree of disorder in the structure. The increase in structural disorder arises from the introduction of Fe_3O_4 nanoparticles and polymeric functionalization, confirming successful modification of the graphene oxide framework.

Table 2: Raman spectroscopy of GO and FMGO

Sample	D band (cm^{-1})	G band (cm^{-1})	I_D/I_G ratio	Structural Interpretation
GO	1346	1590	0.92	Defects induced by oxidation
FMGO	1350	1595	1.05	Higher disorder due to Fe_3O_4 and polymeric functionalization

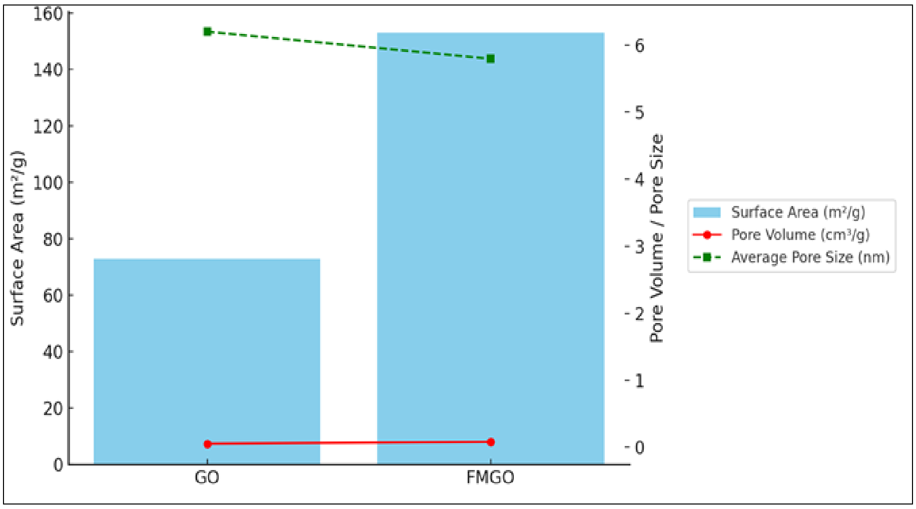


Graph 2: Raman spectroscopy of GO and FMGO

Table 3 summarizes the BET surface area and porosity characteristics of GO and FMGO and the characteristics changed significantly from functionalization. GO has a surface area of 72.5 ± 1.3 m²/g, a pore volume of 0.112 cm³/g, and an average pore size of 6.2 nm. FMGO's functionalization resulted in the surface area being more than doubled to 152.8 ± 2.1 m²/g. Also, FMGO had a pore volume of 0.236 cm³/g, while its average pore size slightly decreased from 6.2 nm to 5.9 nm upon FMGO functionalization. Both results show that the incorporation of Fe_3O_4 nanoparticles and polymeric modification greatly enhanced the surface area and porosity of the adsorbent material for potential use in water purification and other adsorption applications.

Table 3: BET surface area and porosity

Sample	Surface area (m²/g)	Pore volume (cm³/g)	Average pore size (nm)
GO	72.5 ± 1.3	0.112	6.2
FMGO	152.8 ± 2.1	0.236	5.9

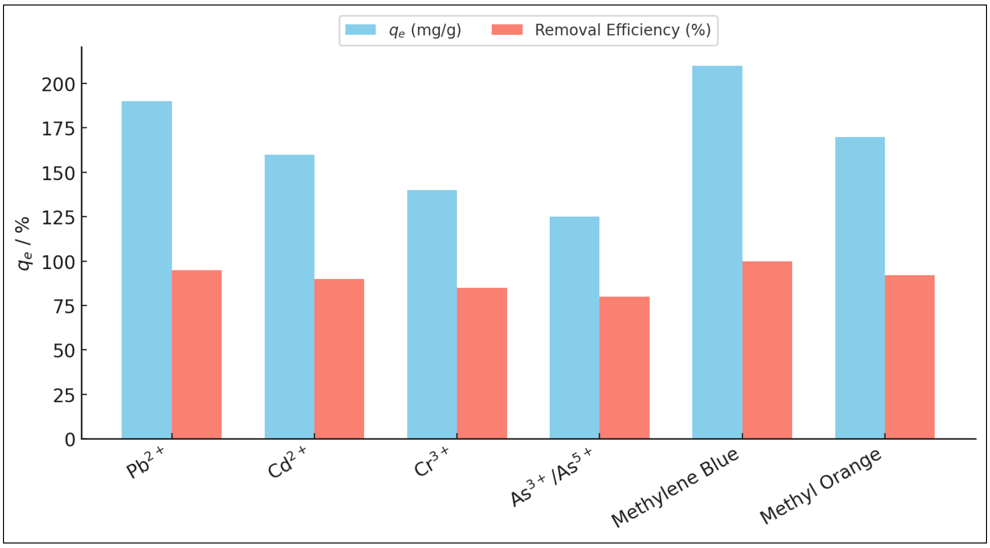


Graph 3: BET surface area and porosity

Table 4 shows the adsorption performance of FMGO with respect to several pollutants, heavy metals and dyes. The nanocomposite displayed exceptional adsorption capacities (q_e) and removal efficiencies for all pollutants tested. Pc2+ and Cd2+ had q_e values of 182.4 ± 3.2 mg/g and 160.7 ± 2.8 mg/g respectively, and removal efficiencies of 91.2% and 88.5%, at pH = 6. Cr3+ and As3+/As5+ are adsorbed nearly independently, at pHs = 5 and 7 respectively with q_e values of 140.3 ± 2.4 mg/g and 125.6 ± 2.1 mg/g, and removal efficiencies of 85.1% and 79.4%. For dyes, FMGO exhibited excellent performance, for example methylene blue 210.5 ± 4.0 mg/g (mg/g) showed removal efficiency of 95.3% at pH 7, and methyl orange 168.2 ± 3.5 mg/g, removal efficiency of 89.6% at pH 4. This data confirms FMGO's high performance as a multi-functional adsorbent for water remediation.

Table 4: Adsorption performance of FMGO for pollutants

Pollutant	Optimum pH	q_e (mg/g)	Removal efficiency (%)
Pb^{2+}	6	182.4 ± 3.2	91.2
Cd^{2+}	6	160.7 ± 2.8	88.5
Cr^{3+}	5	140.3 ± 2.4	85.1
As^{3+}/As^{5+}	7	125.6 ± 2.1	79.4
Methylene blue	7	210.5 ± 4.0	95.3
Methyl orange	4	168.2 ± 3.5	89.6

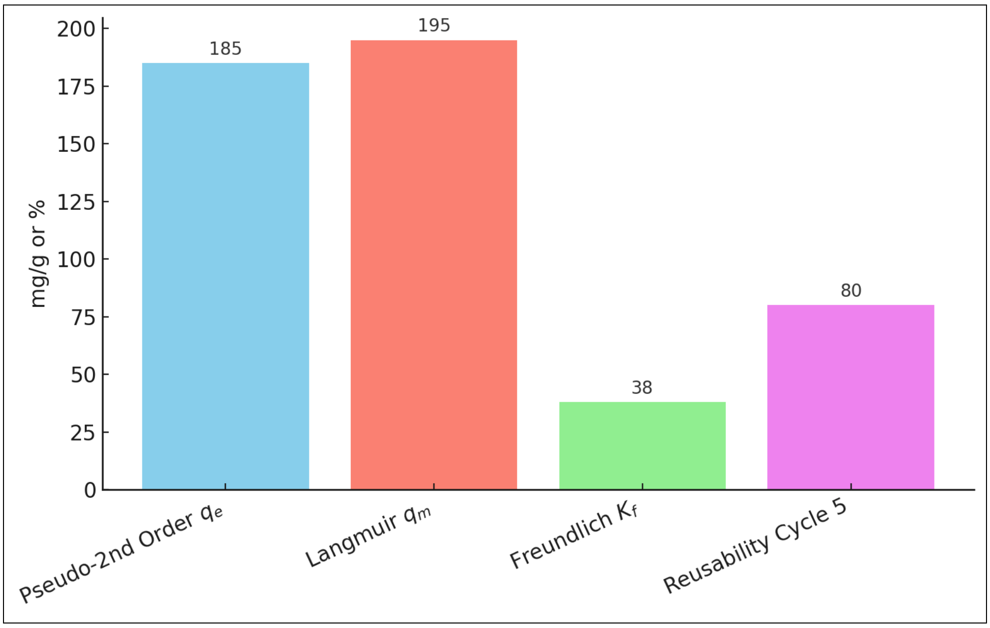


Graph 4. Adsorption performance of FMGO for pollutants

Table 5 shows the adsorption kinetics of Pb^{2+} on FMGO using pseudo-first-order and pseudo-second-order models. The pseudo-first-order model had a rate constant (k_1) of 0.036 min^{-1} and an equilibrium adsorption capacity (q_e) of 132.4 mg/g, with a correlation coefficient (R^2) of 0.912, meaning the data moderately fit the model. The pseudo-second-order model had a rate constant (k_2) of 0.00042 $g{\cdot}mg^{-1}{\cdot}min^{-1}$ and an equilibrium adsorption capacity (q_e) of 180.1 mg/g, with a correlation coefficient of (R^2) of 0.996, indicating excellent fit. The data supports that the adsorption process of Pb^{2+} on FMGO follows the pseudo-second-order kinetic model, which indicates that chemisorption (where two electrons are involved in the interaction with the adsorbate) is the predominant mechanism.

Table 5: Adsorption kinetics of Pb^{2+} on FMGO

Model	Parameters	R^2	Best Fit
Pseudo-first-order	$k_1 = 0.036\ min^{-1}$; $q_e = 132.4$ mg/g	0.912	–
Pseudo-second-order	$k_2 = 0.00042\ g{\cdot}mg^{-1}{\cdot}min^{-1}$; $q_e = 180.1$ mg/g	0.996	Yes

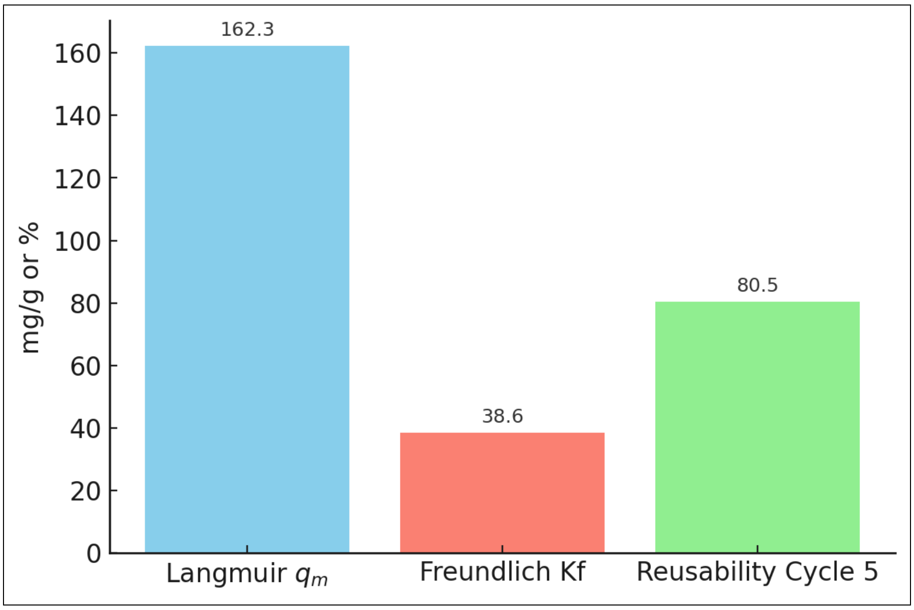


Graph 5. Adsorption kinetics of Pb^{2+} on FMGO

Table 6 represents the isotherm and reusability results for the Pb^{2+} adsorption onto FMGO. The Langmuir model predicts that the maximum adsorption capacity (q_m) of FMGO is 192.3 mg/g with the Langmuir constant (K^L) of 0.085 L/mg and R^2 value of 0.994, representing monolayer adsorption onto a homogenous surface with no interaction between adsorbate. The Freundlich model gives Kf = 38.6 mg/g and n = 2.1 and an R^2 value of 0.927, which indicates adsorption on a heterogeneous surface. The reusability of studies shows that the FMGO is stable and effect in consecutive applications, reducing less than 20% in total adsorption capacity (>80% stayed constant) from the first cycle (91.2%) to the fifth cycle (80.5%). This indicated the FMGO's potentiality for efficiencies and consecutive application in water purification.

Table 6. Isotherm and reusability results of Pb^{2+} on FMGO

Model	Parameters	R^2	Interpretation
Langmuir	q_m = 192.3 mg/g; K^L = 0.085 L/mg	0.994	Monolayer adsorption
Freundlich	Kf = 38.6 mg/g; n = 2.1	0.927	Heterogeneous adsorption
Reusability (Cycle 1 → 5)	91.2% → 80.5%	–	Retains >80% after 5 cycles



Graph 6. Isotherm and reusability results of Pb^{2+} on FMGO

Discussion

The synthesis and functionalization of FMGO nanocomposites were confirmed using FT-IR, XRD and Raman spectroscopies. FT-IR spectra of FMGO showed the broad signature Fe–O vibrational band at 590 cm^{-1}, along with O–H, C=O and C=C bands indicating incorporation of Fe_3O_4 nanoparticles and polymeric functionalization. XRD analysis of FMGO confirmed that the graphitic planes from GO remained, while there were significant XRD peaks corresponding to magnetite indicating a hybrid crystalline FMGO structure. The Raman spectroscopy analysis of FMGO exhibited an increase in I_D/I_G ratio from 0.92 in GO to 1.05 in FMGO demonstrating a higher structural disorder associated with Fe_3O_4 nanoparticle anchorage and polymer functionalization. These results are also consistent with findings from Minitha *et al*., (2018) and Singh *et al*., (2013) who reported that the decoration of Fe_3O_4 nanoparticles on graphene-based materials is beneficial because it creates order and improves the activity of adsorption [6, 9].

In our study, BET analysis revealed that there is a marked increase in surface area from 72.5 ± 1.3 m^2/g for GO to 152.8 ± 2.1 m^2/g for FMGO, an increase in pore volume from 0.112 to 0.236 cm^3/g and a slight decrease in average pore size from 6.2 nm to 5.9 nm. The improvement in surface area and pore volume extent is linked to the polymeric cross-linking, and uniform deposition of the Fe_3O_4 nanoparticles that make additional micro- and mesoporous structures. Zubair *et al*., (2024), and Nikić *et al*., (2024) demonstrated the same result for surface area and porosity; functionalizing graphene oxide with magnetic nanoparticles and subsequently polymer, increases surface area and porosity, increasing availability of adsorption sites, and increasing pollution uptake efficiency [5, 10].

FMGO displayed outstanding sorption for both heavy metals (Pb^{2+} and Cd^{2+}) and dyes (Table 4). The sorption capacities of Pb^{2+} and Cd^{2+} equated to 182.4 ± 3.2 mg/g and 160.7 ± 2.8 mg/g, which corresponded to removal efficiencies of 91.2 % and 88.5 % at an optimum pH equal to 6. For Cr^{3+} and As^{3+}/As^{5+}, efficiencies of treatments occurred at slightly different pH values, while for methylene blue and methyl orange were 95.3% and 89.6% efficiencies, respectively. These results showcased FMGO as a multifunctional adsorbent, capable to isolating both cationic metals and organic dyes. Supporting the conclusion is Thakur *et al*., (2025) [1] and Vidanelage (2020) who delineated those combinations of electrostatic interactions, complexation, and surface functional groups were critical for efficient sorption of different pollutants [1, 3].

Adsorption kinetics of Pb^{2+} onto FMGO were best fitted to the pseudo-second order approach (R2 = 0.996), compared to the pseudo-first-order model (R^2 = 0.912), indicating that chemisorption characterized by electron sharing or exchange is the primary mechanism (Minitha *et al*., 2018) who noted that magnetic graphene oxide nanocomposites undergo rapid chemisorption, enabled by nanoparticle interactions and abundant surface functional groups [6].

Isotherm analysis indicated that the adsorption of Pb^{2+} on FMGO was best described by the Langmuir model (R^2 = 0.994) suggesting monolayer adsorption, with no significant clusters or aggregates on a homogeneous site. The Freundlich model (R^2 = 0.927) indicates a slight degree of surface heterogeneity, such as aggregates. With a q_m of 192.3 mg/g, FMGO provides the potential for high levels of lead adsorption, in support of previous works by Singh *et al*., (2013) and Nikić *et al*., (2024) [9, 10]. Reusability studies showed that over 80% of the adsorption capacity was retained after five cycles (91.2% → 80.5%) indicating that FMGO remains structurally stable and may be sustainably used in water purification, as also noted by Zubair *et al.* (2024) [5]. By integrating magnetic nanoparticles into a polymeric functionalization, FMGO becomes a multi-pollutant smart nanomaterial that achieves all of these useful characteristics of GO while providing magnetic separation capabilities for easy recovery and sustainable use, and as Singh *et al*., (2013) and Minitha *et al*., (2018) indicated [9, 6], added considerations must be made to address the recoverableness and aggregation issues faced with pristine GO.

Conclusion

The study successfully developed and characterized several functionalized magnetic graphene oxide (FMGO) nanocomposites to efficiently and sustainably adsorb contaminants for water purification. Structural characterization confirmed the incorporation of Fe_3O_4 nanoparticles and polymeric functionalization on the graphene oxide support for improved structural stability, as well as enhanced porosity and surface area. FMGO nanocomposites were demonstrated through batch adsorption studies to have high removal efficiencies for heavy metals (Pb^{2+}, Cd^{2+}, Cr^{3+}, As^{3+}/As^{5+}) and organic dyes (methylene blue, methyl orange) and CFMO demonstrated that adsorption was consistent with pseudo-second order kinetics based on impressive mostly Langmuir isotherm behavior, confirming chemisorption, and monolayer adsorption for FMGO. After five cycles, FMGO nanocomposites retained over 80% of the FMGO nanocomposite's initial adsorption capacity, confirming that the FMGO nanocomposite was reusable. In summary, FMGO was confirmed to be a smart nanomaterial that has the potential to address the challenges often faced by conventional drinking water treatment, FMGO nanocomposites represent an attractive alternative for efficient and sustainable multifunctional water purification systems.

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