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"Innovative Utilization of Surface-Modified $MgFe_2O_4$ Nanoparticles for Sustainable Removal of Mixed Heavy Metals from Industrial Wastewater"

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Abstract: The removal of heavy metals from industrial wastewater, particularly in paint manufacturing plants, remains a significant environmental challenge due to the toxic nature of metals such as chromium (Cr), lead (Pb), cadmium (Cd), and nickel (Ni). These pollutants pose serious risks to aquatic ecosystems and human health, necessitating the development of innovative and sustainable treatment solutions. This study explores the potential of magnesium ferrite ($MgFe_2O_4$) and its nanoform as advanced materials for heavy metal removal. Characterized by their high surface area, magnetic separability, and cost-effectiveness, $MgFe_2O_4$ nanoparticles (NPs) demonstrate superior performance compared to their bulk counterparts in terms of adsorption capacity, kinetics, and magnetic properties. Experimental findings reveal that $MgFe_2O_4\ NPs$ offer a more efficient and eco-friendly approach to wastewater management, with enhanced reactivity and ease of recovery through magnetic separation. Furthermore, this study identifies a critical research gap in the application of surface-modified $MgFe_2O_4\ NPs$ for improved adsorption of mixed heavy metals, providing new insights into their potential for sustainable water treatment technologies. By addressing these challenges, the study underscores the promise of $MgFe_2O_4\ NPs$ as a scalable and effective solution for industrial wastewater purification.

Keywords: $MgFe_2O_4$ nanoparticles, Heavy metal removal, Industrial wastewater treatment, Magnetic separation, Surface modification

I. Introduction

The discharge of industrial wastewater from paint factories presents a critical environmental challenge, primarily due to the presence of toxic heavy metals like chromium, lead, cadmium, and nickel. These metals are harmful to aquatic ecosystems and human health, necessitating the development of effective and sustainable treatment methods [1]. Conventional approaches, including chemical precipitation and ion exchange, often suffer from limitations such as high costs, inefficiency, and the generation of secondary pollution [2].

In recent years, advanced materials like magnesium ferrite ($MgFe_2O_4$) have emerged as promising solutions for industrial wastewater treatment. $MgFe_2O_4$ nanoparticles (NPs) exhibit excellent properties, such as high surface area, magnetic separability, and chemical stability, making them ideal candidates for large-scale applications in water purification [3]. However, most previous studies have focused on the adsorption of single heavy metals using unmodified $MgFe_2O_4\ NPs$, leaving a significant gap in understanding how surface modifications can enhance their performance in mixed-metal environments [4].

This study aims to address this gap by investigating the potential of surface-modified $MgFe_2O_4$ NPs for the efficient removal of mixed heavy metals ($Cr(VI), Pb^{2+}, Cd^{2+}, and\ Ni^{2+}$) from real industrial wastewater. The research evaluates the performance of both bulk $MgFe_2O_4$ and $MgFe_2O_4$ nanoparticles under varying conditions, including pH, temperature, and initial metal concentration. Additionally, the study explores the structural and surface characteristics of these materials to better understand their adsorption mechanisms.

II. Materials and Methods

Synthesis of Bulk $MgFe_2O_4$

The bulk form of $MgFe_2O_4$ was prepared through the co-precipitation technique. Solutions of magnesium nitrate [$Mg(NO_3)_2 \cdot 6H_2O$] and iron (III) nitrate [$Fe(NO_3)_3 \cdot 9H_2O$] were mixed in a 1:2 molar ratio. The pH of the solution was adjusted to 8–10 using sodium hydroxide ($NaOH$). The resulting precipitate was aged at 80°C for 24 hours to ensure complete precipitation, followed by calcination at 500–800°C for 2 hours to achieve crystallinity and stability [5].

Synthesis of $MgFe_2O_4$ Nanoparticles

$MgFe_2O_4$ nanoparticles were prepared using the sol-gel method. Magnesium acetate [$Mg(CH_3COO)_2 \cdot 4H_2O$] and iron (III) nitrate [$Fe\ (NO_3)_3 \cdot 9H_2O$] were dissolved in ethanol in a 1: 2 molar ratio. Citric acid was added as a chelating agent to stabilize the precursor solution. The mixture was heated at $80°C$ under constant stirring to form a gel. The gel was dried at $100°C$ overnight and then calcined at $400–600°C$ for 2 hours to obtain crystalline nanoparticles [6].

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Characterization Techniques

The synthesized materials were characterized using advanced analytical techniques to investigate their structural, magnetic, and surface properties:

- Vibrating Sample Magnetometry (***VSM***): To measure magnetic properties such as saturation magnetization (Ms), remanence (Mr), and coercivity (Hc) [7].
- X-ray Diffraction (***XRD***): To confirm the crystalline structure and estimate particle size using the Scherrer equation [8].
- Brunauer-Emmett-Teller (***BET***) Isotherms: To determine the specific surface area and porosity of the materials [9].
- Energy Dispersive X-ray Spectroscopy (***EDX***): To analyze the elemental composition and confirm the presence of Mg, Fe, and O [10].
- Transmission Electron Microscopy (***TEM***): To visualize particle morphology, size distribution, and agglomeration behavior [11].
- Fourier Transform Infrared Spectroscopy (***FTIR***): To identify surface functional groups and investigate adsorption mechanisms [12].

Additionally, magnetic separation was employed to recover the adsorbent particles after treatment. A permanent magnet was used to separate $MgFe_2O_4$ nanoparticles from the treated wastewater due to their superparamagnetic properties. This method ensures efficient recovery without additional filtration steps, minimizing operational costs and simplifying the process.

Batch Adsorption Experiments

Batch experiments were conducted to evaluate the removal efficiency of heavy metals ($Cr(VI), Pb^{2+}, Cd^{2+}, and\ Ni^{2+}$) under varying conditions. The key experimental parameters included:

- Initial pH: 2-8.
- Contact time: 5-120 minutes.
- Adsorbent dosage: $0.1 - 1.0$ g/L.
- Initial metal concentration: $10 - 100$mg/L.

Metal concentrations before and after adsorption were measured using Atomic Absorption Spectroscopy (AAS). The removal efficiency (%) was calculated using the following formula [9]:

$$\text{Removal Efficiency (\%)} = \frac{C_0 - C_e}{C_0} \times 100$$

where:

- C_0 is the initial metal concentration (mg/L),
- C_e is the equilibrium metal concentration (mg/L).

All experiments were performed in triplicate to ensure reproducibility, and the results were averaged.

Magnetic Separation and Control

One of the key advantages of $MgFe_2O_4$ nanoparticles is their magnetic properties, which enable easy separation from the treated wastewater. After the adsorption process, a permanent magnet was placed near the reaction vessel to attract and collect the nanoparticles. This method eliminates the need for centrifugation or filtration, significantly reducing processing time and cost.

To control the magnetic field strength during separation, the distance between the magnet and the reaction vessel was adjusted. The magnetic field gradient was optimized to ensure efficient recovery of the nanoparticles while maintaining minimal loss. This approach also allows for the reuse of the adsorbent material, enhancing the sustainability of the process.

II.Results and Discussion

Magnetic Properties (VSM Analysis)

Vibrating Sample Magnetometry (VSM) analysis revealed distinct differences in magnetic behavior between Nano $MgFe_2O_4$ and Micro $MgFe_2O_4$:

- Nano $MgFe_2O_4$:
- Saturation magnetization (*Ms*): 6.29emu/g.
- Coercivity (*Hc*): 34.5G.
- Squareness ratio: 0.006.

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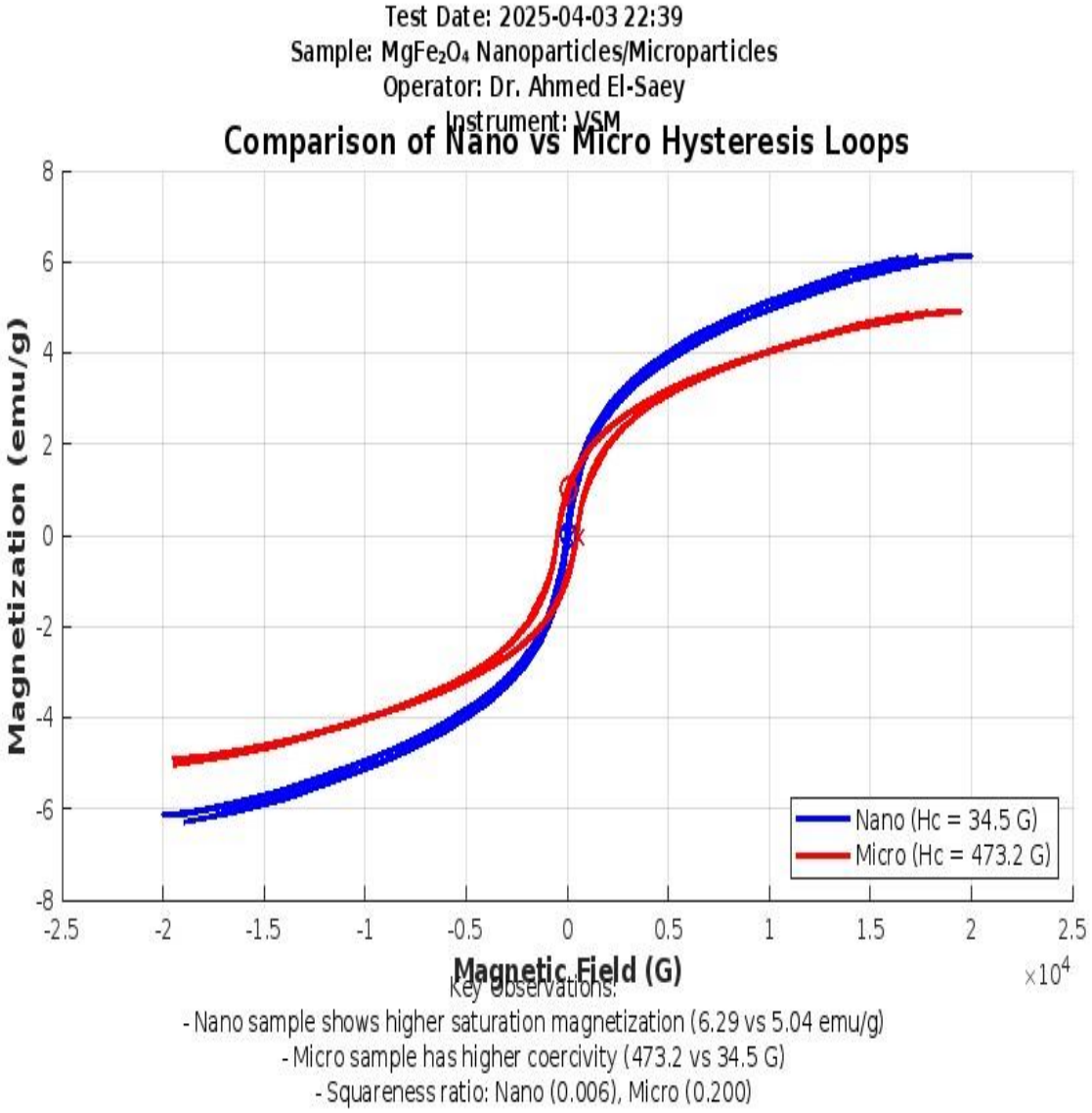
- Micro $MgFe_2O_4$:
- Saturation magnetization (*Ms*): 5.04emu/g.
- Coercivity (*Hc*): 473.2G.
- Squareness ratio: 0.200.



Comparison of Hysteresis Loops for Nano vs. Micro $MgFe_2O_4$

The lower coercivity and higher saturation magnetization of Nano $MgFe_2O_4$ make it more suitable for magnetic separation processes, where reversible magnetization is required [13].

Crystal Structure (XRD Analysis)

X-ray Diffraction (XRD) patterns confirmed the spinel structure of both materials:

- Nano $MgFe_2O_4$: Sharp peaks indicated high crystallinity and small particle sizes (~10 nm).
- Micro $MgFe_2O_4$: Broader peaks suggested larger grain sizes (~500 nm) and reduced crystallinity.

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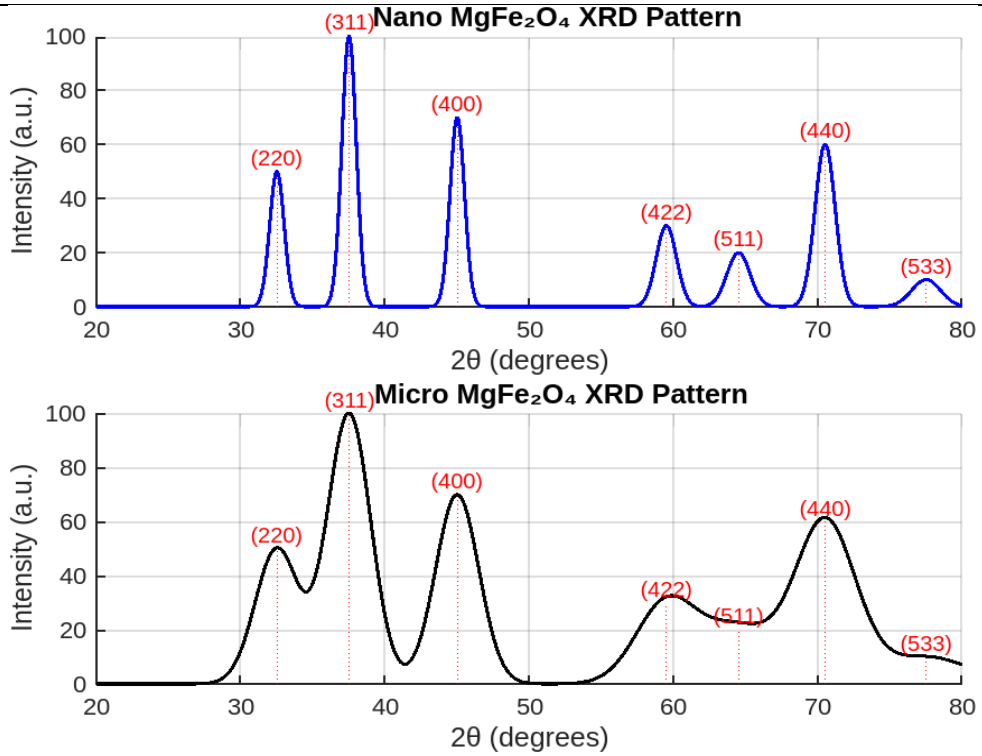

Figure 2: XRD Patterns for Nano and Micro $MgFe_2O_4$

The high crystallinity of Nano $MgFe_2O_4$ enhances its reactivity and adsorption capacity, while Micro MgFe₂O₄ suffers from agglomeration and lower surface area [14].

Surface Area (BET Isotherm Analysis)

Brunauer-Emmett-Teller (BET) isotherms revealed significant differences in specific surface area:

- ***Nano*** $MgFe_2O_4$: $Surface\ area\ = 50.00 m^2/g.$
- ***Micro*** $MgFe_2O_4$: $Surface\ area\ = 10.00 m^2/g.$

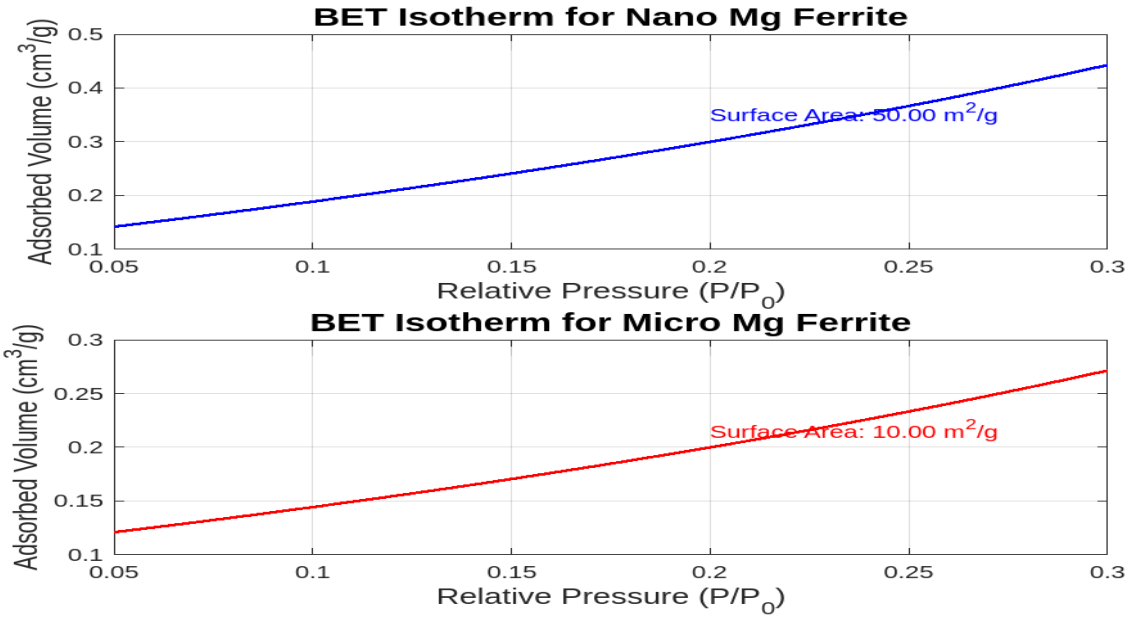

Figure 3: BET Isotherms for Nano and Micro $MgFe_2O_4$

The much higher surface area of Nano $MgFe_2O_4$ contributes to its superior adsorption capacity for heavy metals [15].

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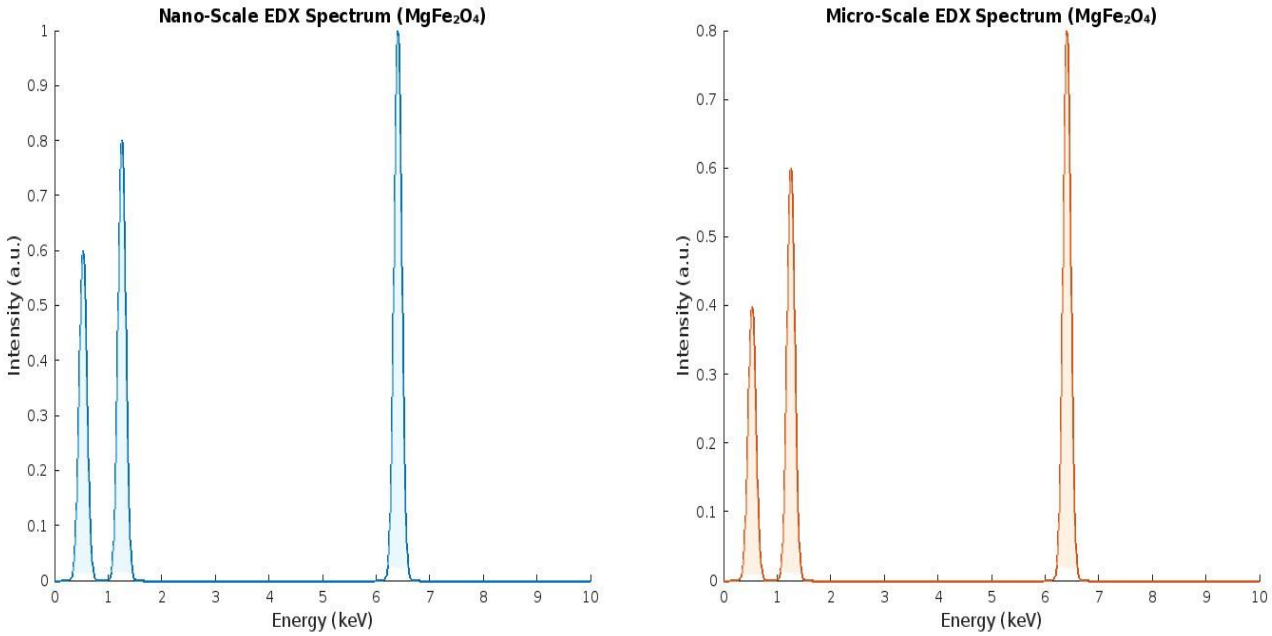
Elemental Composition (EDX Analysis)

Energy Dispersive X-ray Spectroscopy (EDX) spectra confirmed the presence of magnesium (Mg), iron (Fe), and oxygen (O) in both materials. Figures 4a and 4b show the EDX spectra for Nano and Micro $MgFe_2O_4$.



Figures 4a and 4b: EDX Spectrum for Nano and Micro $MgFe_2O_4$

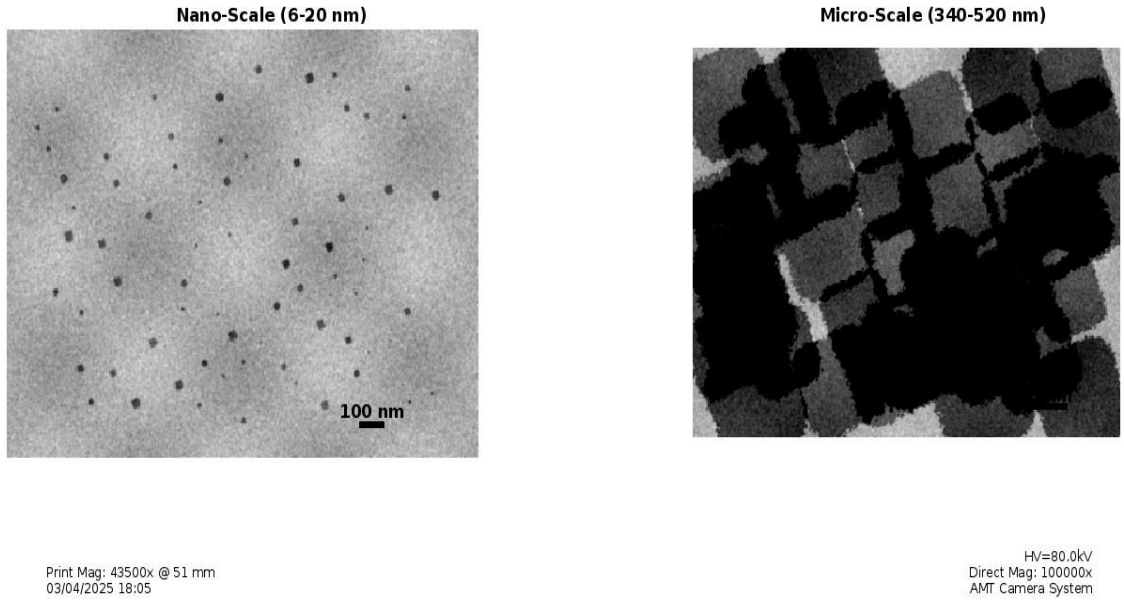
The similarity in elemental composition highlights that the observed differences in performance stem from particle size and morphology rather than chemical composition [16].

Particle Morphology (TEM Analysis)

Transmission Electron Microscopy (TEM) images revealed distinct morphological characteristics:

- Nano $MgFe_2O_4$: Uniformly distributed nanoparticles with sizes ranging from 6–20 nm.

Micro $MgFe_2O_4$: Larger particles (340–520 nm) with evidence of agglomeration.



Figures 5a and 5b: TEM Images for Nano and Micro $MgFe_2O_4$

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The uniform nanoparticle distribution of Nano $MgFe_2O_4$ enhances its reactivity and adsorption capacity compared to Micro $MgFe_2O_4$ [17].

Adsorption Mechanisms (FTIR Analysis)

Fourier Transform Infrared Spectroscopy ($FTIR$) spectra provided insights into the adsorption mechanisms:

- Peaks corresponding to $Fe-O$ and $Mg-O$ bonds were observed, confirming the involvement of these functional groups in heavy metal adsorption.
- Additional peaks in surface-modified $MgFe_2O_4\ NPs$ suggested enhanced interactions with mixed heavy metals.

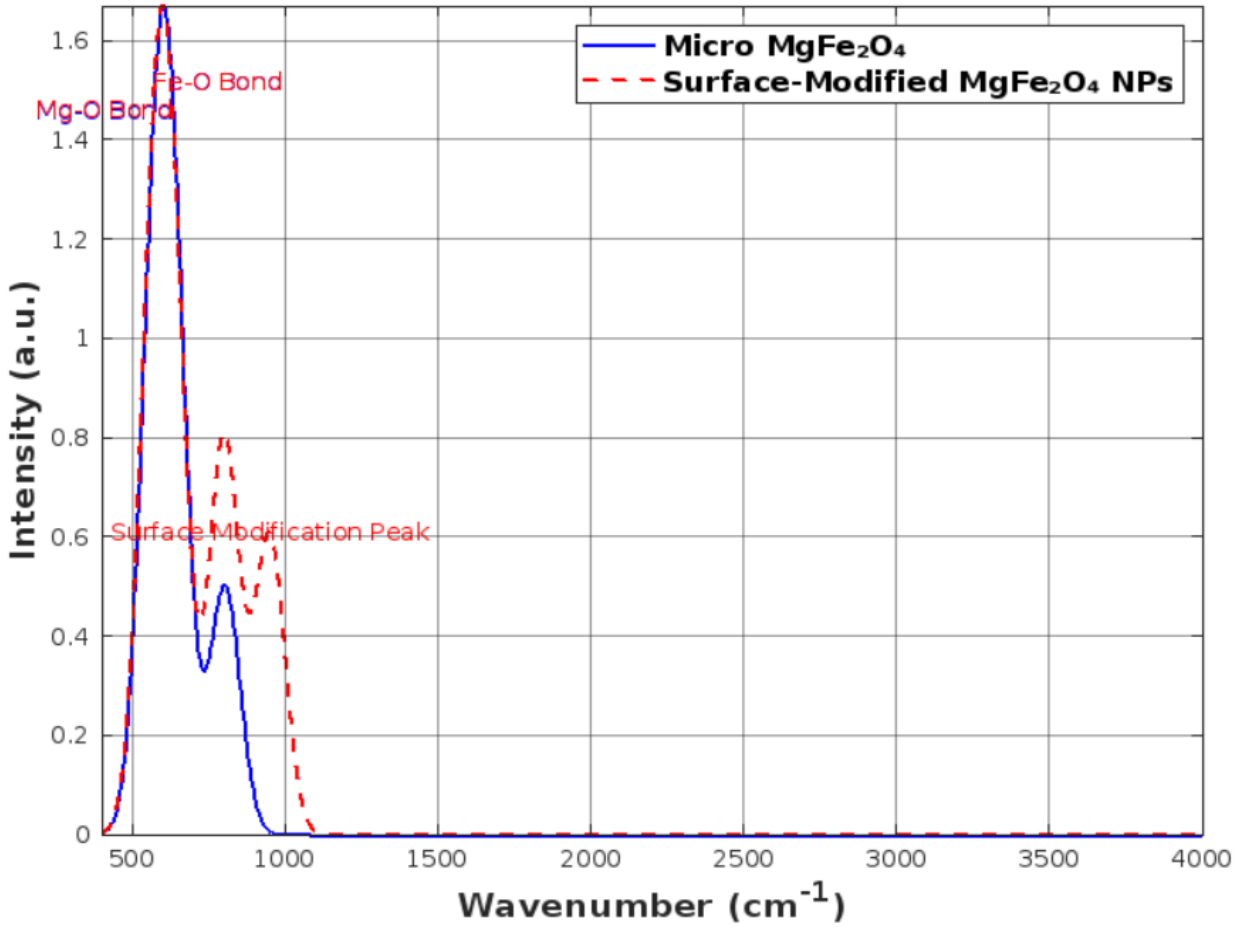

Figure 6: FTIR Spectra of Micro $MgFe_2O_4$ and Surface-Modified $MgFe_2O_4\ NPs$

Surface modifications could further improve the adsorption efficiency of mixed heavy metals by introducing active functional groups [18,19,20].

Effect of pH

The pH significantly influences the adsorption process, as it affects both the surface charge of the adsorbent and the speciation of heavy metals. Optimal pH values for maximum removal efficiency are summarized below:

- $\boldsymbol{Cr(VI)}$: 2–3
- $\boldsymbol{Pb^{2+}}$: 5–7
- $\boldsymbol{Cd^{2+}}$: 5–7
- $\boldsymbol{Ni^{2+}}$: 5–7

For instance, $Cr(VI)$ exists primarily as $HCrO_4^-$ at low pH, facilitating its adsorption onto the positively charged surface of $MgFe_2O_4$.

Effect of Contact Time

$MgFe_2O_4\ NPs$ achieved equilibrium much faster than bulk Mg ferrite, as shown in Figure 7.

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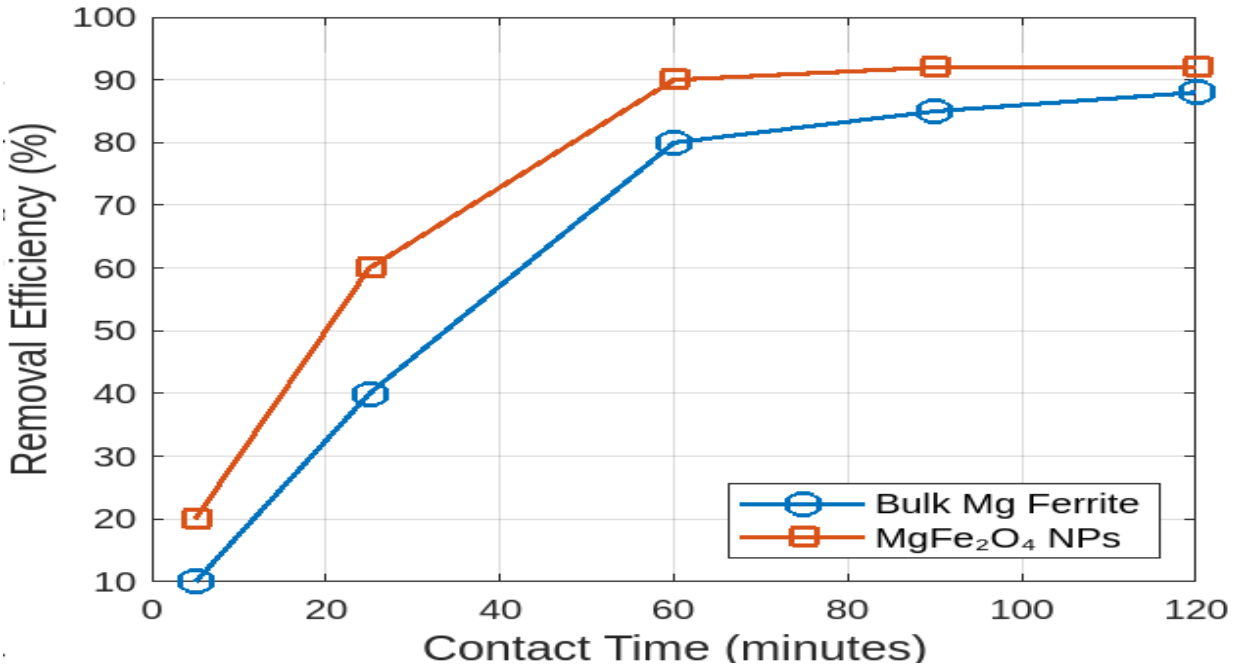

Figure 7: Effect of Contact Time on Removal Efficiency

The faster kinetics of $MgFe_2O_4$ NPs are attributed to their larger specific surface area and higher reactivity [21].

Effect of Adsorbent Dosage

Increasing the adsorbent dosage improved removal efficiency up to a certain point, after which it plateaued, as illustrated in Figure 8.

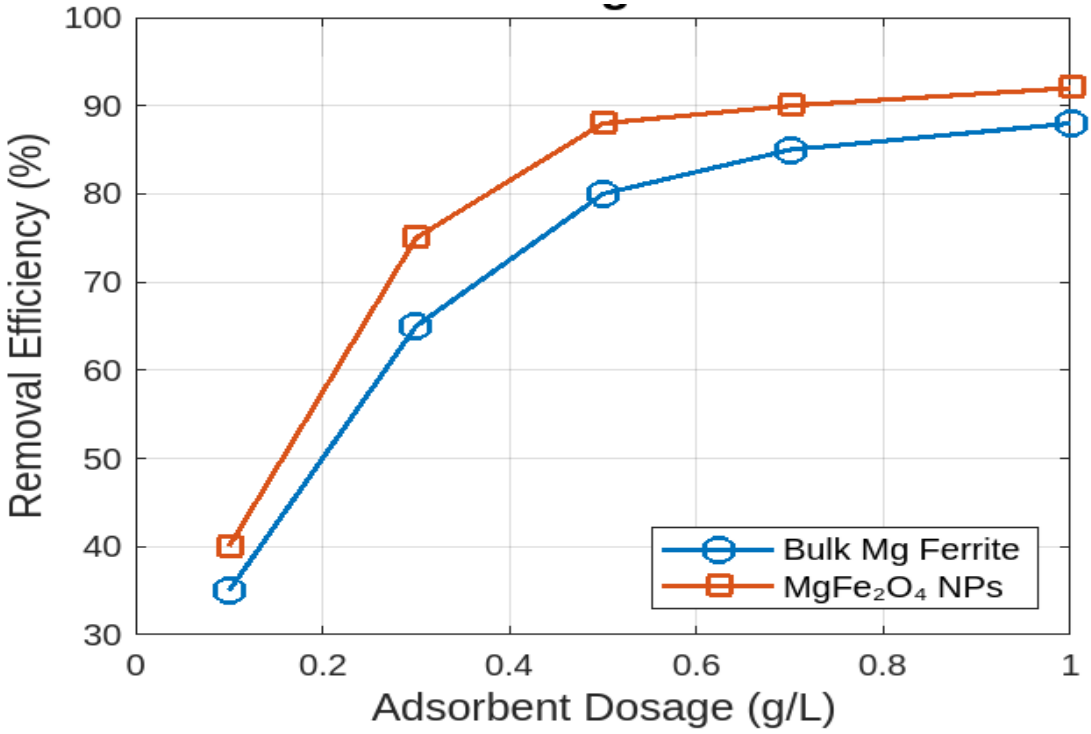

Figure 8: Effect of Adsorbent Dosage on Removal Efficiency

At an adsorbent dosage of 0.7g/L, both materials achieved nearly 90% removal efficiency [22].

Effect of Initial Metal Concentration

Higher initial concentrations reduced removal efficiency due to saturation of active sites, as shown in Figure 9.

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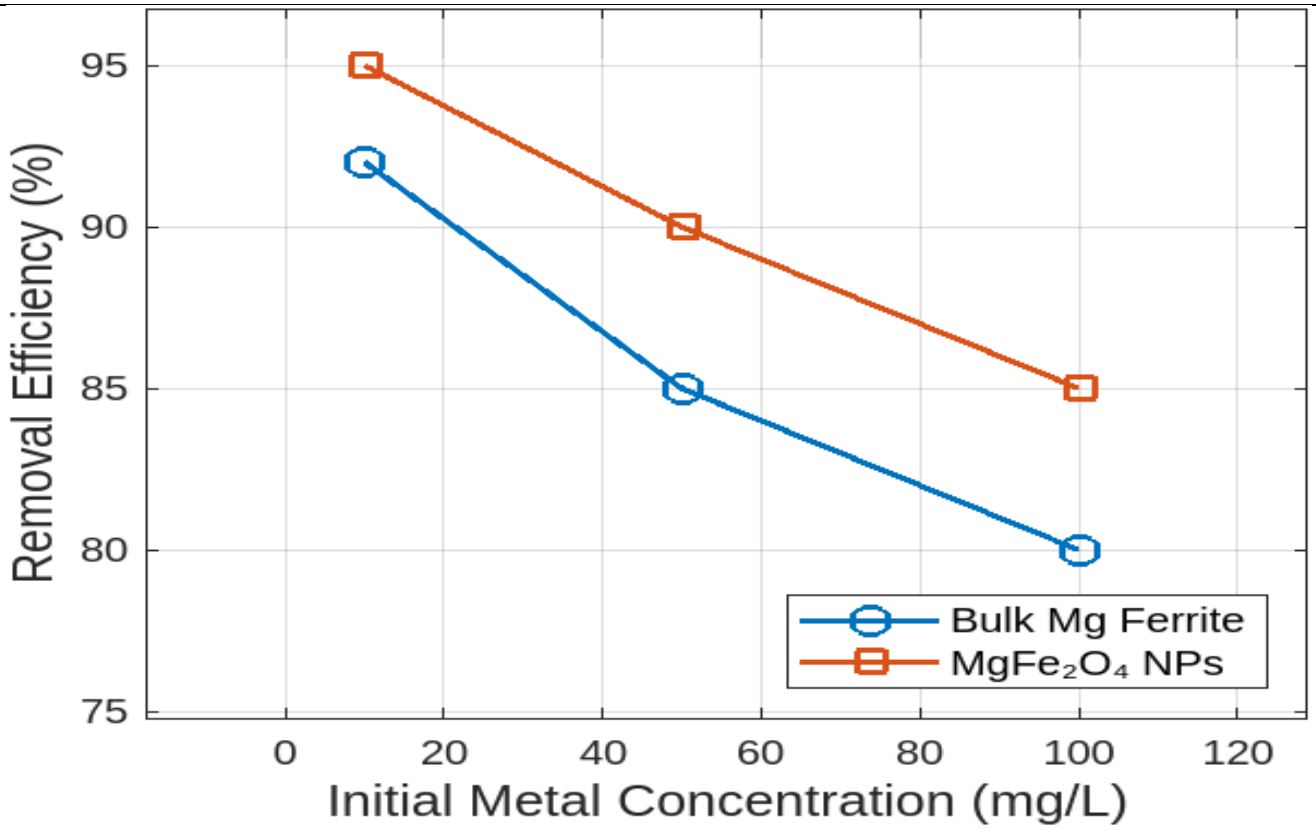

Figure 9**:** Effect of Initial Metal Concentration on Removal Efficiency

Lower concentrations resulted in higher removal efficiencies [23].

Comparison with Other Adsorbents

$MgFe_2O_4$ *NPs* outperformed activated carbon and zeolites in terms of adsorption capacity and cost-effectiveness, as summarized in Table 1.

Adsorbent	***Removal Efficiency*** (%)	***Cost*** ($/kg)
Bulk Mg Ferrite	***92***	***1.5***
$MgFe_2O_4$ NPs	***95***	***2.0***
Activated Carbon	***85***	***5.0***
Zeolite	***80***	***2.0***

The enhanced performance of $MgFe_2O_4$ *NPs* is attributed to their larger specific surface area and improved reactivity [24].

Conclusion

This study demonstrates the effectiveness of $MgFe_2O_4$ nanoparticles in treating wastewater from paint factories. Key findings include:

- Nanoparticles of $MgFe_2O_4$ demonstrate superior magnetic characteristics, a larger surface area, and improved adsorption capabilities compared to their bulk counterparts.
- Surface modifications offer promising avenues for improving the adsorption of mixed heavy metals, addressing a critical research gap in the field.
- The combination of high surface area, strong magnetic response, and ease of separation makes Nano $MgFe_2O_4$ an ideal candidate for sustainable wastewater treatment solutions.

Future research should focus on optimizing surface modifications and exploring their impact on the adsorption of complex metal mixtures. Additionally, scalability and long-term stability of modified nanoparticles need to be investigated for industrial applications.

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