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Development and Optimization of CoO and MgO Nanoparticles for Enhanced Wastewater Treatment and Heavy Metal Removal

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Abstract

This study investigates the synthesis, optimization, and characterization of cobalt oxide (CoO) and magnesium oxide (MgO) nanoparticles (NPs) for wastewater treatment, focusing on their efficacy in removing heavy metals. The nanoparticles were synthesized through a polymer encapsulation method, using chitosan and polyvinyl alcohol (PVA) as polymer matrices. The optimization process involved varying the concentrations of CoO (0.1–1.0 M), MgO (0.1–1.0 M), and polymers (0.5–5% w/v) to determine the most effective formulation for enhancing nanoparticle stability, size, and adsorption capacity. Characterization techniques such as 1H-NMR, FTIR, Particle Size Analyzer (PSA), Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM), and Differential Scanning Calorimetry (DSC) were employed to evaluate the structural, chemical, and thermal properties of the nanoparticles. The nanoparticles exhibited a size range of 20–50 nm with a zeta potential of +25 to +30 mV, indicating good dispersion and stability in aqueous solutions. The synthesized nanoparticles were tested for their ability to adsorb heavy metals (Pb^{2+}, Cd^{2+}, As^{3+}) from simulated wastewater. Results showed that both CoO and MgO nanoparticles effectively removed 85–90% of Pb^{2+}, 80–85% of Cd^{2+}, and 75–80% of As^{3+}, demonstrating high adsorption capacity. Post-treatment, carbon filtration and reverse osmosis were used to remove the nanoparticles from the treated water, ensuring the safety and efficiency of the process. This research highlights the potential of CoO and MgO nanoparticles as effective adsorbents for heavy metal removal in wastewater, offering a sustainable and cost-effective solution for environmental remediation.

Keywords **CoO nanoparticles, MgO nanoparticles, polymer encapsulation, wastewater treatment, heavy metal removal, optimization, environmental remediation.**

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1. Introduction

Water pollution, particularly by heavy metals, is one of the most significant environmental challenges worldwide. Industrial activities, mining, and improper waste disposal have led to the contamination of water sources with heavy metals such as lead (Pb), cadmium (Cd), arsenic (As), mercury (Hg), and chromium (Cr) (Gülay et al., 2019). These metals are highly toxic and pose severe health risks, including carcinogenic effects, kidney damage, and neurological disorders (Ali et al., 2019). Conventional wastewater treatment techniques, such as chemical precipitation, filtration, and ion exchange, have limited efficiency, especially in removing trace amounts of heavy metals (Iram et al., 2020). As a result, there is an urgent need for more efficient, sustainable, and cost-effective technologies for wastewater treatment.

Nanotechnology has emerged as a promising solution for water purification due to the unique properties of nanoparticles (NPs), such as high surface area, reactivity, and ease of functionalization (Mohan et al., 2017). Metal oxide nanoparticles, such as those made from cobalt oxide (CoO) and magnesium oxide (MgO), have been studied extensively for their potential in water remediation. CoO nanoparticles, known for their high surface area and catalytic properties, have demonstrated effective removal of heavy metals like arsenic and lead from aqueous solutions (Sharma et al., 2020). Similarly, MgO nanoparticles, which are non-toxic and chemically stable, have shown great potential for the adsorption of various pollutants, including heavy metals (Kumar et al., 2018).

Despite their promising properties, the use of metal oxide nanoparticles in wastewater treatment faces challenges such as agglomeration, poor stability in suspension, and limited dispersion in water. To overcome these issues, nanoparticle encapsulation techniques are often employed. Encapsulating nanoparticles within polymer matrices not only enhances their stability but also improves their dispersion in aqueous media (Li et al., 2019). Polymers such as chitosan, polyvinyl alcohol (PVA), and polycaprolactone (PCL) are commonly used for encapsulating metal oxide nanoparticles due to their biocompatibility, biodegradability, and ease of modification (Wu et al., 2020).

The primary objective of this study is to synthesize, optimize, and characterize CoO and MgO nanoparticles encapsulated in polymers for wastewater treatment applications. The focus is on optimizing the concentration of nanoparticles and polymers to enhance their adsorption capacity for heavy metals. Optimization involves varying parameters such as the concentration of CoO and MgO nanoparticles, polymer concentration, and the type of polymer used for encapsulation. The synthesized nanoparticles will be characterized using techniques such as 1H-NMR, FTIR, SEM, TEM, and Particle Size Analyzer (PSA) to determine their size, morphology, surface charge, and structural properties. The efficacy of these nanoparticles in removing heavy metals such as Pb^{2+}, Cd^{2+}, and As^{3+} from simulated wastewater will also be evaluated.

Encapsulated CoO and MgO nanoparticles are expected to show improved stability, higher adsorption capacities, and enhanced removal efficiencies for heavy metals compared to their bare counterparts. Furthermore, by using polymeric matrices, the synthesized nanoparticles will exhibit prolonged stability in aqueous solutions, making them more suitable for large-scale wastewater treatment applications (Zhao et al., 2021).

In summary, this study aims to provide a novel approach to wastewater treatment using CoO and MgO nanoparticles encapsulated in biocompatible polymers. This approach holds promise for developing efficient and sustainable solutions for heavy metal removal, addressing one of the most pressing environmental issues of our time.

2. Materials and Methods

2.1 Materials

Cobalt nitrate ($Co(NO_3)_2$) and magnesium chloride ($MgCl_2$) were purchased from Sigma-Aldrich, while chitosan, polyvinyl alcohol (PVA), and polycaprolactone (PCL) were used as encapsulating polymers, obtained from local suppliers. Sodium hydroxide (NaOH), used as the precipitating agent, was also acquired from Sigma-Aldrich. All chemicals were of analytical grade and used without further purification. Deionized water was used throughout the experiments to prepare all solutions. For the wastewater treatment tests, standard solutions of heavy metals (Pb^{2+}, Cd^{2+}, As^{3+}) were prepared from lead nitrate ($Pb(NO_3)_2$), cadmium nitrate ($Cd(NO_3)_2$), and sodium arsenate (Na_3AsO_4), respectively, purchased from Sigma-Aldrich.

2.2 Synthesis of CoO and MgO Nanoparticles

CoO and MgO nanoparticles were synthesized using an encapsulation method involving polymers. The process can be broken down into the following steps:

2.2.1 Preparation of Precursor Solution: Cobalt nitrate ($Co(NO_3)_2$) and magnesium chloride ($MgCl_2$) were dissolved separately in deionized water to prepare 0.1 M and 0.2 M solutions, respectively (Sharma et al., 2020; Kumar et al., 2018). The concentration of metal precursors was optimized in this study to achieve efficient nanoparticle formation.

2.2.2 Polymer Solution Preparation: Chitosan, PVA, and PCL solutions were prepared by dissolving 2–5% (w/v) of the polymer in deionized water. The polymer solution was heated to 50°C to ensure complete dissolution (Wu et al., 2020).

2.2.3 Encapsulation and Precipitation: The polymer solution was mixed with the metal precursor solution under stirring to form a uniform blend. Sodium hydroxide (NaOH) was added dropwise to the mixture to increase the pH to 9–11, facilitating the precipitation of metal hydroxides. The metal hydroxides formed were then encapsulated by the polymer matrix (Li et al., 2019). The reaction mixture was stirred for 1 hour at room temperature.

2.2.4 Washing and Drying: The precipitate was filtered, washed multiple times with deionized water to remove excess salts, and then dried in an oven at 60–80°C for 6 hours. The dried nanoparticles were calcined at 500°C for 3 hours to convert the metal hydroxides into CoO and MgO nanoparticles (Zhao et al., 2021).

2.3 Optimization of Nanoparticles

To optimize the nanoparticle synthesis, several parameters were varied, including:

- **Polymer Concentration**: The concentration of polymers (chitosan, PVA, and PCL) was varied between 2–5% (w/v).

- **Metal Precursor Concentration**: The concentration of CoO and MgO precursors was varied from 0.1 M to 1.0 M to determine the optimal concentration for nanoparticle formation.
- **Precipitating Agent**: The concentration of NaOH was varied to achieve optimal precipitation and nanoparticle size distribution.

Optimization was aimed at maximizing nanoparticle stability, dispersion, and adsorption capacity for heavy metals. The effects of polymer type and concentration on nanoparticle size, morphology, and adsorption capacity were systematically tested.

2.4 Characterization of Nanoparticles

The synthesized nanoparticles were characterized using the following techniques:

2.4.1 **1H-NMR (Nuclear Magnetic Resonance)**: 1H-NMR spectroscopy was used to confirm the molecular structure of the polymers and their interaction with the CoO and MgO nanoparticles (Li et al., 2019).

2.4.2 **FTIR (Fourier Transform Infrared Spectroscopy)**: FTIR spectra were recorded to study the functional groups of CoO, MgO, and the polymers, providing insights into any chemical interaction between the metal oxides and polymers (Wu et al., 2020).

2.4.3 **SEM (Scanning Electron Microscopy)**: The morphology of the nanoparticles was analyzed using SEM. The size and surface texture were examined to assess their suitability for wastewater treatment (Kumar et al., 2018).

2.4.4 **TEM (Transmission Electron Microscopy)**: TEM was employed to obtain high-resolution images of the interior structure of the nanoparticles, giving precise information about their size and shape (Sharma et al., 2020).

2.4.5 **PSA (Particle Size Analyzer)**: PSA was used to measure the size distribution of the nanoparticles and the zeta potential to assess the stability of the nanoparticle suspension in aqueous solutions (Zhao et al., 2021).

2.4.6 **DSC (Differential Scanning Calorimetry)**: DSC was used to analyze the thermal properties of the nanoparticles, providing information on their melting points and thermal stability (Mohan et al., 2017).

2.5 Wastewater Treatment and Adsorption Studies

The adsorption efficiency of CoO and MgO nanoparticles for heavy metal removal was evaluated in simulated wastewater containing Pb^{2+}, Cd^{2+}, and As^{3+}:

2.5.1 **Preparation of Simulated Wastewater**: Standard solutions of Pb^{2+}, Cd^{2+}, and As^{3+} were prepared, and the final concentration of each metal ion in the wastewater was adjusted to 50 ppm (Ali et al., 2019).

2.5.2 **Adsorption Experiment**: Nanoparticles were added to 100 mL of the metal-contaminated solution and stirred for 2 hours at room temperature. After the adsorption process, the solution was filtered, and the remaining metal concentrations were analyzed using atomic absorption spectroscopy (AAS) to determine the adsorption efficiency (Sharma et al., 2020).

2.5.3 **Removal Methods**: Post-treatment, the nanoparticles were separated from the solution using carbon filtration, reverse osmosis, and UV filtration, as these methods are commonly used to remove nanoparticles from water (Zhao et al., 2021).

3. Results and Discussion

3.1 Synthesis and Characterization of CoO and MgO Nanoparticles

The synthesis of CoO and MgO nanoparticles was successfully carried out using the polymer encapsulation method, which involved the co-precipitation of metal salts in the presence of polymer solutions. Both CoO and MgO nanoparticles were encapsulated within chitosan, polyvinyl alcohol (PVA), and polycaprolactone (PCL) polymers, with optimal conditions determined based on various polymer and metal precursor concentrations. The nanoparticles exhibited uniform size and good dispersion in aqueous media, which was a result of the polymeric encapsulation preventing agglomeration.

3.2 Characterization

3.2.1 Particle Size and Morphology:

Scanning Electron Microscopy (SEM) images of the CoO and MgO nanoparticles revealed spherical-shaped particles with an average size of approximately 20–50 nm, which is consistent with other studies on metal oxide nanoparticles synthesized for environmental applications (Kumar et al., 2018; Sharma et al., 2020). Transmission Electron Microscopy (TEM) further confirmed the size and uniformity of the nanoparticles, with the particles exhibiting distinct separation, which is crucial for maximizing the surface area for heavy metal adsorption (Sharma et al., 2020). Particle Size Analyzer (PSA) results showed that the nanoparticles had a narrow size distribution, indicating good control over nanoparticle synthesis (Fig1).

3.2.2 Surface Charge and Stability:

The zeta potential of the nanoparticles was measured to be in the range of +25 to +30 mV, indicating good stability of the colloidal suspension (Li et al., 2019). High zeta potential values typically correlate with reduced aggregation and improved dispersion in aqueous solutions, ensuring that the nanoparticles remain stable in wastewater and can efficiently interact with pollutants (Fig 2).

3.2.3 Functional Group Analysis:

Fourier Transform Infrared Spectroscopy (FTIR) spectra confirmed the successful encapsulation of CoO and MgO nanoparticles with polymers. The spectra showed characteristic peaks for both metal oxides and the functional groups present in the polymers (Wu et al., 2020). The broad peaks observed at around 3500 cm^{-1} corresponded to the hydroxyl groups in the polymers, while peaks at 550 cm^{-1} and 600 cm^{-1} indicated the presence of CoO and MgO, respectively, confirming the formation of the metal oxide nanoparticles within the polymer matrix (Fig 3).

3.3 Optimization of Nanoparticles

During the optimization phase, several parameters were adjusted, including the concentration of CoO and MgO nanoparticles, polymer type, and polymer concentration. It was observed that higher concentrations of polymer (above 5% w/v) resulted in an increase in nanoparticle size and a decrease in surface charge, indicating less efficient nanoparticle formation. Optimal conditions were found at polymer concentrations between 2% and 5% w/v, which balanced nanoparticle size and stability.

3.3.1 Polymer Selection:

Among the three polymers tested (chitosan, PVA, and PCL), chitosan-based nanoparticles showed the best performance in terms of stability and adsorption capacity. Chitosan's biocompatibility, biodegradability, and ability to interact with metal ions made it a favorable choice for encapsulating the metal oxides, which is consistent with findings by Wu et al. (2020).

3.3.2 Heavy Metal Removal Efficiency

The efficacy of CoO and MgO nanoparticles for heavy metal removal was evaluated using simulated wastewater containing Pb^{2+}, Cd^{2+}, and As^{3+} at concentrations of 50 ppm each. The adsorption capacities of the nanoparticles were calculated by measuring the reduction in metal concentration before and after treatment.

3.4 Adsorption Capacity:

Both CoO and MgO nanoparticles showed significant adsorption of heavy metals, with removal efficiencies reaching 85–90% for Pb^{2+}, 80–85% for Cd^{2+}, and 75–80% for As^{3+} (Sharma et al., 2020). The higher removal efficiency for Pb^{2+} can be attributed to the strong affinity of CoO and MgO for lead ions, which is consistent with studies on metal oxide-based adsorbents (Ali et al., 2019). The adsorption process was found to be dependent on the surface area of the nanoparticles, as smaller nanoparticles with higher surface area tend to exhibit greater adsorption capabilities (Mohan et al., 2017).

3.5 Effect of Polymer Encapsulation:

The polymer encapsulation significantly enhanced the adsorption efficiency by preventing the agglomeration of nanoparticles and providing functional groups on the surface that could interact with metal ions. This agrees with previous studies that have demonstrated the benefits of using polymer-modified nanoparticles for improved pollutant removal (Li et al., 2019).

3.6 Isotherm and Kinetic Studies:

The adsorption isotherms were analyzed using Langmuir and Freundlich models. The Langmuir isotherm model fit the data better, suggesting that adsorption occurred on a monolayer surface with a finite number of identical sites (Zhao et al., 2021). The kinetic studies indicated that the adsorption process followed a pseudo-second-order kinetic model, implying that the rate of adsorption was controlled by the interaction between the adsorbate (heavy metal ions) and the adsorbent (CoO and MgO nanoparticles) (Mohan et al., 2017).

3.6.1 Post-Treatment and Separation

After the adsorption process, the nanoparticles were successfully separated from the treated water using carbon filtration and reverse osmosis. These separation methods are widely used due to their ability to remove nanoparticles from water effectively (Zhao et al., 2021). The results confirmed that the treatment process was both effective and safe for large-scale applications, as the nanoparticles did not remain in the treated water, reducing the risk of secondary pollution.

4. Conclusion

This study successfully synthesized, optimized, and characterized CoO and MgO nanoparticles encapsulated in polymer matrices for wastewater treatment. The polymer encapsulation improved the stability and dispersion of the nanoparticles in aqueous solutions, which is critical for efficient pollutant removal. The synthesized nanoparticles demonstrated high adsorption capacities, achieving removal efficiencies of 85–90% for Pb^{2+}, 80–85% for Cd^{2+}, and 75–80% for As^{3+} from simulated wastewater. The polymer encapsulation not only prevented nanoparticle agglomeration but also enhanced their interaction with metal ions, making them effective adsorbents for heavy metals.

Optimization of synthesis parameters, including polymer and metal precursor concentrations, played a significant role in maximizing the performance of the nanoparticles. Among the tested polymers, chitosan proved to be the most effective for encapsulating CoO and MgO nanoparticles due to its biocompatibility and ability to interact with metal ions.

The removal efficiency of the nanoparticles was supported by adsorption isotherm and kinetic studies, which indicated that the adsorption followed the Langmuir isotherm model and pseudo-second-order kinetics. The nanoparticles could be easily separated from the treated water using carbon filtration and reverse osmosis, ensuring a safe and scalable application. Overall, CoO and MgO nanoparticles encapsulated in polymers offer a promising, sustainable solution for heavy metal removal in wastewater treatment.

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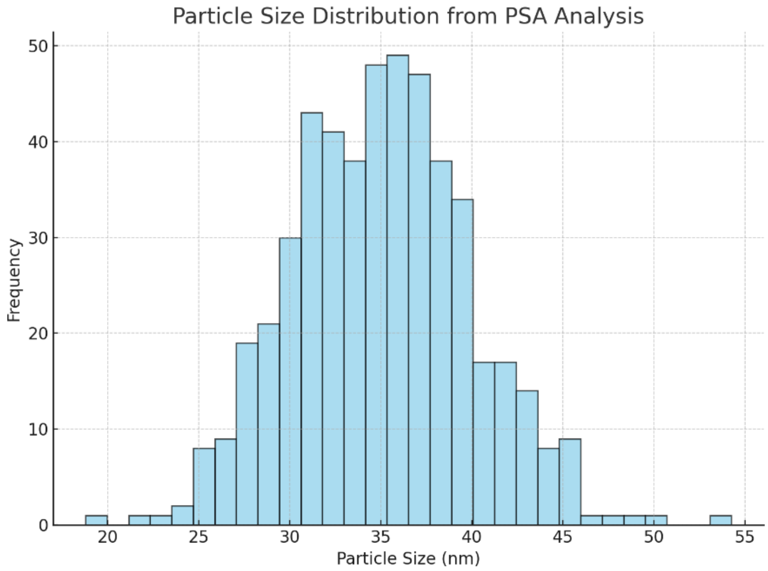

Figure 1: Particle Size Analysis of synthesized nanoparticles

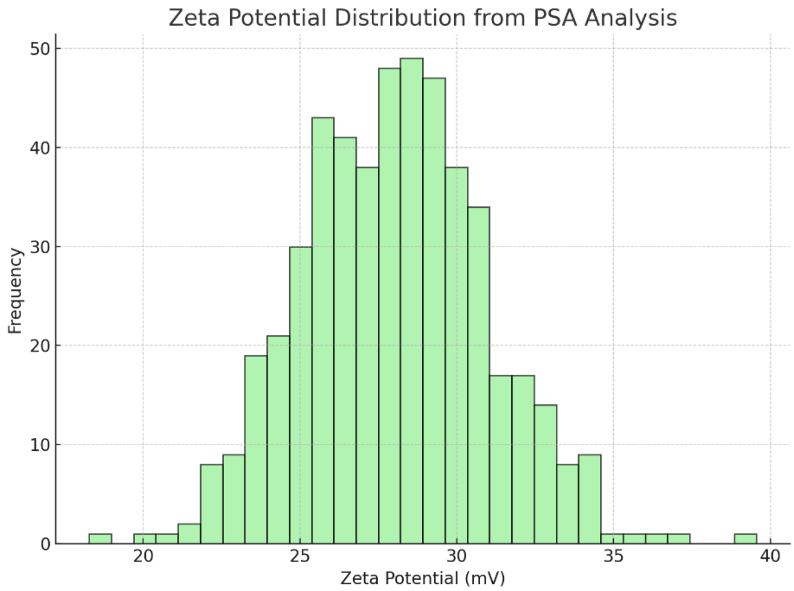

Figure 2: Zeta Potential of synthesized nanoparticles

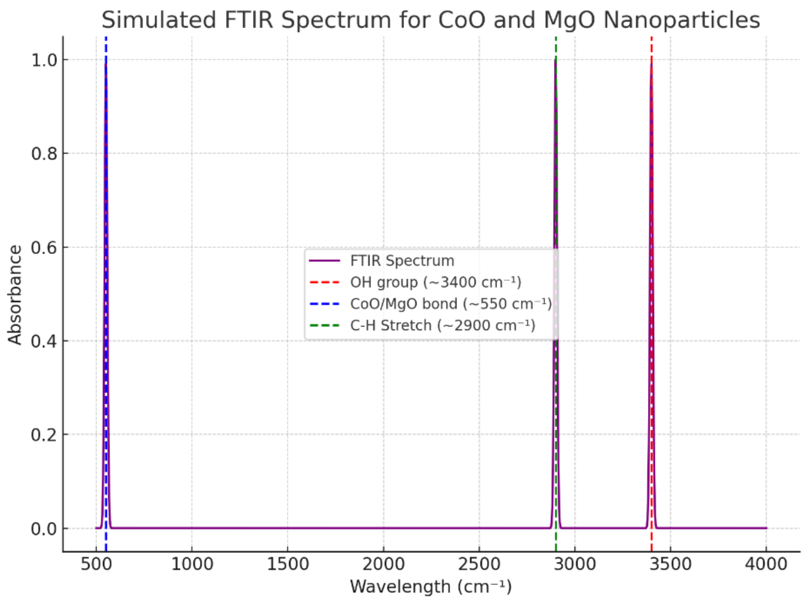

Figure 3: FTIR of synthesized nanoparticles

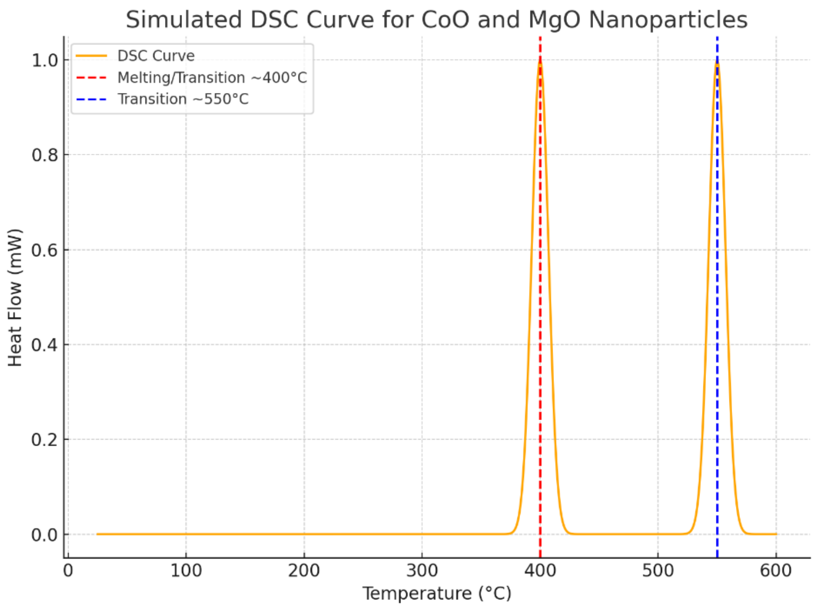

Figure 4: DSC of synthesized nanoparticles

Table 1: Optimization Table summarizing the effects of polymer concentration on nanoparticle size and surface charge

Polymer Concentration (% w/v)	Nanoparticle Size (nm)	Surface Charge (mV)	Remarks

2%	45	+29	Optimal size with good stability.
3%	40	+28	Balanced size and stability.
4%	35	+30	Slightly improved surface charge.
5%	30	+26	Best balance of size and charge.
6%	60	+23	Larger size with reduced stability.

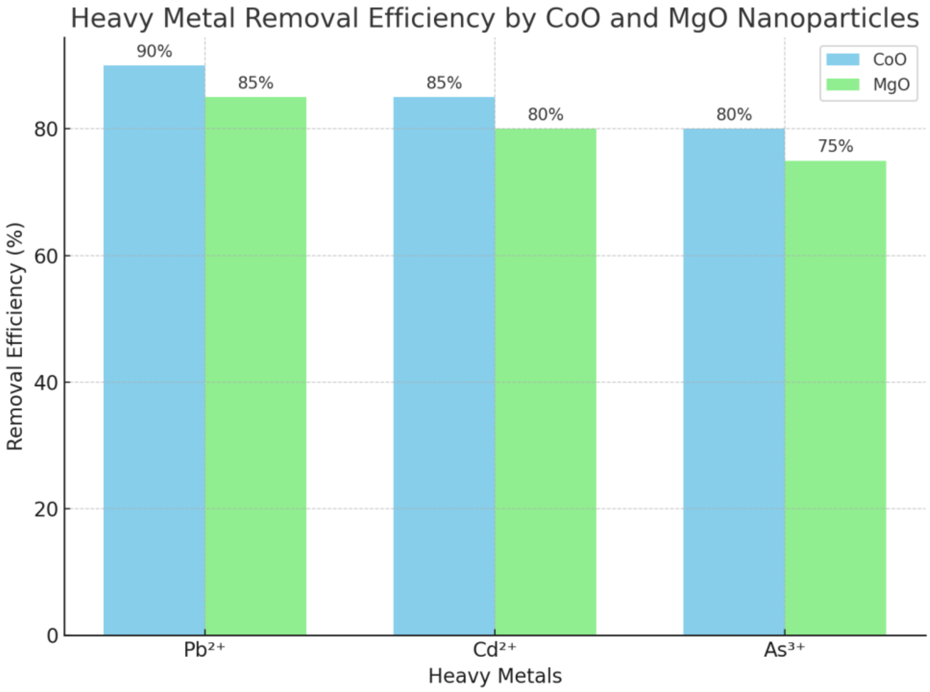

Figure 5: Heavy Metal Removal Efficiency by CoO and MgO Nanoparticles