REDVET - Revista electrónica de Veterinaria - ISSN 1695-7504
Vol 25, No.1S (2024)
http://www.veterinaria.org
Article Received: Revised: Accepted:

"Synthesis of Cobalt Oxide and Magnesium Oxide Nanoparticles for Enhanced Wastewater Treatment Applications"

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Abstract
The sol-gel synthesis, optimisation, and characterisation of cobalt oxide (CoO) and magnesium oxide (MgO) nanoparticles for wastewater treatment are examined in this paper. Due to their enormous surface area, reactivity, and capacity to catalyse organic pollutant breakdown and adsorb heavy metal ions, metal oxide nanoparticles are potential candidates for efficient and sustainable water filtration. To optimise nanoparticle size, shape, and surface properties, precursor concentration, calcination temperature, and time were adjusted. XRD, SEM, TEM, and FTIR were used to analyse the synthesised nanoparticles' structural and morphological properties. XRD revealed the nanoparticles' crystalline structures, including CoO (cubic phase) and MgO (well-defined cubic structure). The typical crystallite sizes for CoO and MgO were 22 and 18 nm, respectively, while TEM showed nanoparticle size distributions of 20-25 and 15-20 nm. The purity of metal oxide nanoparticles was validated by FTIR spectra showing Co-O and Mg-O bonding. Nanoparticles characterisation, batch adsorption, and catalytic degradation tests were performed to evaluate their efficacy in treating synthetic wastewater with heavy metals and organic contaminants. After 3 hours of treatment, CoO nanoparticles degraded methylene blue dye 92% efficiently. MgO nanoparticles effectively adsorb heavy metals like lead (Pb^{2+}) and cadmium (Cd^{2+}), with removal efficiencies of 88% and 85%, respectively. MgO nanoparticles' reduced size and increased surface area improved their aqueous metal ion adsorption. This study shows that CoO and MgO nanoparticles can remediate industrial wastewater by adsorbing and degrading organic and inorganic contaminants. Surface functionalisation of nanoparticle production may improve their efficiency and adaptability in wastewater treatment applications.

Keywords: Cobalt oxide nanoparticles, Magnesium oxide nanoparticles, Sol-gel synthesis, Wastewater treatment, Heavy metal removal, Organic pollutant degradation, Nanoparticles, Adsorption, Catalytic degradation, Environmental remediation

Introduction

Water pollution remains one of the most pressing environmental issues, exacerbated by rapid industrialization, urbanization, and agricultural activities. Industrial effluents often contain a mixture of harmful organic pollutants, heavy metals, and other contaminants that pose serious health risks to humans, aquatic life, and the environment. Traditional wastewater treatment methods, such as chemical precipitation, coagulation, filtration, and biological treatments, often fail to fully remove these contaminants, especially at lower concentrations[1]. Additionally, they can be energy-intensive and result in secondary pollution due to the production of sludge. Therefore, there is a growing demand for innovative and efficient technologies to address the complexities of wastewater treatment[2].

Nanotechnology has emerged as a transformative approach to environmental remediation, offering solutions that address the limitations of conventional treatment methods. Nanoparticles, particularly metal oxide nanoparticles, have gained significant attention due to their high surface area-to-volume ratio, reactivity, and tunable physicochemical properties[3]. These characteristics make them ideal candidates for applications in adsorption and catalysis, enabling them to target a wide range of pollutants, from heavy metals to complex organic compounds. Among the various metal oxides, cobalt oxide (CoO) and magnesium oxide (MgO) nanoparticles have demonstrated considerable promise in wastewater treatment[4].

Cobalt oxide (CoO) nanoparticles are known for their excellent electrochemical and catalytic properties. They have been widely studied for applications in catalysis, energy storage, and environmental remediation. In the context of wastewater treatment, CoO nanoparticles exhibit strong catalytic activity in the degradation of organic pollutants, such as dyes and phenolic compounds, through advanced oxidation processes (AOPs). Their ability to act as a catalyst in the presence of oxidizing agents, such as hydrogen peroxide, makes them highly effective in breaking down persistent organic pollutants into less harmful by-products. Furthermore, the magnetic properties of CoO nanoparticles facilitate their recovery from treated water, making them a reusable and environmentally friendly option[5].

Magnesium oxide (MgO) nanoparticles, on the other hand, are widely recognized for their adsorption properties, particularly for heavy metals. MgO has a high affinity for metal ions such as lead (Pb^{2+}), cadmium (Cd^{2+}), arsenic (As^{3+}), and chromium (Cr^{6+}), which are common pollutants in industrial wastewater[6]. The high surface area and basic nature of MgO nanoparticles enhance their interaction with these metal ions, allowing for efficient adsorption and removal from

water. Additionally, MgO nanoparticles have been shown to exhibit antibacterial properties, making them suitable for applications that require the simultaneous removal of pathogens and toxic substances from wastewater[7]. The combination of adsorption and antimicrobial activity makes MgO nanoparticles a multifunctional tool for water purification.

The **sol-gel method**, used for synthesizing CoO and MgO nanoparticles in this study, is a well-established chemical process known for its simplicity, cost-effectiveness, and ability to produce nanoparticles with controlled size and morphology. By adjusting parameters such as precursor concentration, calcination temperature, and time, it is possible to optimize the size and surface characteristics of the nanoparticles, which are critical for their performance in environmental applications. The sol-gel method also offers advantages in terms of scalability, making it suitable for large-scale production of nanomaterials for industrial wastewater treatment[8].

In this study, the focus is on the preparation, optimization, and characterization of CoO and MgO nanoparticles, synthesized via the sol-gel method, for the removal of both organic pollutants and heavy metals from wastewater. The nanoparticles were characterized using techniques such as X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), and Fourier-transform infrared spectroscopy (FTIR) to evaluate their structural and morphological properties. The effectiveness of CoO and MgO nanoparticles in wastewater treatment was assessed through batch adsorption and catalytic degradation experiments. CoO nanoparticles were evaluated for their catalytic performance in degrading organic dyes, while MgO nanoparticles were tested for their adsorption capacity in removing heavy metals[9].

By leveraging the unique properties of CoO and MgO nanoparticles, this research aims to provide a comprehensive understanding of how these nanomaterials can be applied to real-world wastewater treatment. The study contributes to the growing field of nanotechnology in environmental remediation, with the potential to develop efficient, scalable, and sustainable solutions to address the global water crisis.

Materials and Methods

1. Materials

- **Cobalt nitrate hexahydrate ($Co(NO_3)_2 \cdot 6H_2O$)**: Purchased from Sigma-Aldrich, used as a precursor for the synthesis of cobalt oxide (CoO) nanoparticles.
- **Magnesium nitrate hexahydrate ($Mg(NO_3)_2 \cdot 6H_2O$)**: Obtained from Merck, used as a precursor for the synthesis of magnesium oxide (MgO) nanoparticles.
- **Citric acid ($C_6H_8O_7$)**: Used as a chelating agent in the sol-gel process, acquired from Sigma-Aldrich.
- **Ethanol (C_2H_5OH)**: Analytical grade, used as a solvent during nanoparticle synthesis.
- **Deionized water**: Used in the preparation of all aqueous solutions.
- **Methylene blue dye**: Used for evaluating the catalytic degradation activity of CoO nanoparticles.
- **Lead nitrate ($Pb(NO_3)_2$) and cadmium chloride ($CdCl_2$)**: Used for testing the heavy metal adsorption efficiency of MgO nanoparticles.

2. Synthesis of Cobalt Oxide (CoO) Nanoparticles

The CoO nanoparticles were synthesized using the **sol-gel method**, a well-established technique for producing highly pure and uniform nanoparticles.

1. **Preparation of Sol**:

Cobalt nitrate hexahydrate (2 g) was dissolved in 50 mL of deionized water to form a homogeneous solution. Separately, 1.5 g of citric acid was dissolved in 50 mL of ethanol. The cobalt nitrate solution was added dropwise to the citric acid-ethanol mixture under continuous stirring at 500 rpm[10].

2. **Gel Formation**:

The solution was heated at 80°C under constant stirring until a gel-like consistency was achieved. This step facilitated the chelation of cobalt ions by citric acid.

3. **Aging**:

The resulting gel was aged for 24 hours at room temperature to ensure complete gelation and stabilization of the sol[11].

4. **Drying and Calcination**:

The aged gel was dried in an oven at 100°C for 12 hours to remove excess solvents. The dried gel was then calcined in a muffle furnace at 500°C for 3 hours to produce CoO nanoparticles. The calcination temperature was optimized to ensure proper phase formation and particle size control[12].

3. Synthesis of Magnesium Oxide (MgO) Nanoparticles

The MgO nanoparticles were synthesized similarly to CoO using the sol-gel process, with slight modifications in temperature and time.

1. **Preparation of Sol**:

Magnesium nitrate hexahydrate (2 g) was dissolved in 50 mL of deionized water, and 1.5 g of citric acid was dissolved in 50 mL of ethanol. The magnesium nitrate solution was slowly added to the citric acid-ethanol mixture while stirring at 500 rpm.

2. **Gel Formation and Aging**:

The solution was heated to 80°C until gel formation occurred. The gel was aged for 24 hours at room temperature.

3. Drying and Calcination:

The dried gel was obtained by heating it at 100°C for 12 hours. It was then calcined at 600°C for 3 hours, producing MgO nanoparticles. The higher calcination temperature for MgO was chosen to optimize its crystalline structure and increase its surface area for adsorption.

4. Optimization of Nanoparticle Synthesis

The size and surface characteristics of both CoO and MgO nanoparticles were optimized by varying the calcination temperature and time. Different batches were prepared by adjusting the calcination temperature (400°C, 500°C, and 600°C) and time (2, 3, and 4 hours). The final calcination conditions that produced the smallest nanoparticles with the best catalytic and adsorption properties were selected for further analysis[13].

5. Characterization Techniques

The synthesized CoO and MgO nanoparticles were characterized using various analytical techniques to confirm their size, structure, and morphology.

5.1. X-ray Diffraction (XRD):

XRD was employed to determine the crystalline structure and phase purity of the nanoparticles. The samples were analyzed using CuKα radiation (λ = 1.5406 Å) in the 2θ range of 20° to 80°. The crystallite size was calculated using the Debye-Scherrer equation:

$D=K\lambda\beta\cos\theta D = \frac{K \lambda}{\beta \cos \theta} D=\beta\cos\theta K\lambda$

where D is the crystallite size, K is the shape factor (0.9), λ is the X-ray wavelength, β is the full width at half maximum (FWHM) of the peak, and θ is the diffraction angle[14].

5.2. Scanning Electron Microscopy (SEM):

SEM was used to study the surface morphology and particle size distribution of the nanoparticles. The samples were coated with a thin layer of gold to enhance conductivity before imaging.

5.3. Transmission Electron Microscopy (TEM):

TEM provided detailed information on the size, shape, and dispersion of the nanoparticles. The samples were dispersed in ethanol and dropped onto carbon-coated copper grids for imaging at a high magnification.

5.4. Fourier-transform Infrared Spectroscopy (FTIR):

FTIR was conducted to identify functional groups and confirm the metal-oxygen bond formation in the nanoparticles. Spectra were recorded in the range of 4000 to 400 cm^{-1} using KBr pellets.

6. Wastewater Treatment Efficiency

6.1. Catalytic Degradation of Methylene Blue

The catalytic activity of CoO nanoparticles was evaluated by the degradation of methylene blue dye in an aqueous solution. A batch reactor was set up with 50 mL of 10 ppm methylene blue solution and 0.1 g of CoO nanoparticles. Hydrogen peroxide (H_2O_2) was added to initiate the oxidation process. The reaction was carried out under stirring at room temperature, and the concentration of methylene blue was measured at different time intervals using UV-visible spectroscopy at 664 nm.

6.2. Adsorption of Heavy Metals (Pb^{2+} and Cd^{2+})

The adsorption efficiency of MgO nanoparticles was tested for the removal of lead (Pb^{2+}) and cadmium (Cd^{2+}) ions from aqueous solutions. Batch experiments were conducted by adding 0.1 g of MgO nanoparticles to 50 mL of 10 ppm solutions of Pb^{2+} and Cd^{2+}. The mixtures were stirred for 2 hours, and the concentration of metal ions remaining in the solution was measured using atomic absorption spectroscopy (AAS). The removal efficiency was calculated using the formula:

7. Statistical Analysis

All experiments were conducted in triplicate, and the results are expressed as the mean ± standard deviation. Statistical significance was determined using one-way ANOVA followed by Tukey's post hoc test, with a p-value of less than 0.05 considered significant.

Results and Discussion

1. Particle Size Analysis (PSA)

The particle size of both cobalt oxide (CoO) and magnesium oxide (MgO) nanoparticles was measured using dynamic light scattering (DLS) (Fig 1a):

● **CoO Nanoparticles**: The average particle size was found to be **22 nm**, with a narrow distribution. The small size allows for a higher surface area, enhancing the catalytic activity of CoO nanoparticles in wastewater treatment.
● **MgO Nanoparticles**: The average particle size was **18 nm**, slightly smaller than CoO. This smaller size facilitates a larger surface area, which improves the adsorption capabilities for heavy metals.

2. Zeta Potential Analysis (Zetasizer)

The surface charge (zeta potential) of the nanoparticles was measured to assess their stability in aqueous solutions (Fig 1b):
● **CoO Nanoparticles**: The zeta potential was **+25.3 mV**, indicating good colloidal stability. This positive charge improves interaction with negatively charged pollutants like organic dyes, making them ideal for catalytic degradation.
● **MgO Nanoparticles**: The zeta potential was **+32.1 mV**, ensuring strong colloidal stability and a highly charged surface, which aids in the adsorption of negatively charged heavy metal ions like Pb^{2+} and Cd^{2+}.

3. Fourier-transform Infrared Spectroscopy (FTIR)

FTIR spectra confirmed the formation of CoO and MgO nanoparticles and verified the absence of impurities (Fig 2):
● **CoO Nanoparticles**: A strong peak at **570 cm^{-1}** indicated the presence of Co-O bonds, confirming the formation of cobalt oxide.
● **MgO Nanoparticles**: The FTIR spectrum showed a sharp peak at **550 cm^{-1}**, corresponding to Mg-O bonds, indicating the formation of magnesium oxide.
No significant peaks for organic impurities were observed, confirming the purity of both CoO and MgO nanoparticles after synthesis and calcination.

4. Scanning Electron Microscopy (SEM)

SEM images provided a visual representation of the nanoparticle morphology (Fig 3a):
● **CoO Nanoparticles**: SEM images showed spherical particles with some agglomeration, indicative of their small size (~22 nm).
● **MgO Nanoparticles**: MgO nanoparticles also exhibited a spherical shape with fewer agglomerates, confirming the uniformity and small particle size (~18 nm). This morphology contributes to the large surface area required for effective adsorption of heavy metals.

5. Transmission Electron Microscopy (TEM)

TEM provided more detailed visualization of particle size and shape (Fig 3b):
● **CoO Nanoparticles**: TEM images confirmed the spherical morphology of the CoO nanoparticles with a size range of **20-25 nm**, closely matching the PSA results.
● **MgO Nanoparticles**: TEM analysis revealed that MgO nanoparticles were spherical, with sizes between **15-20 nm**, again confirming the PSA results. The particles were well-dispersed with minimal aggregation, crucial for maximizing their adsorption properties.

6. X-ray Diffraction (XRD)

XRD was employed to confirm the crystalline structure and purity of the nanoparticles (Fig 4):
● **CoO Nanoparticles**: XRD patterns displayed characteristic peaks at $2\theta = 31.2°$, 36.8°, and 59.3°, which are consistent with the cubic phase of cobalt oxide. The average crystallite size was calculated to be **22 nm** using the Debye-Scherrer formula.
● **MgO Nanoparticles**: The diffraction peaks at $2\theta = 36.9°$, 42.8°, and 62.3° were characteristic of cubic magnesium oxide, with an average crystallite size of **18 nm**. The sharpness of the peaks indicated high crystallinity.

7. Wastewater Treatment Efficiency

7.1. Catalytic Degradation of Methylene Blue (CoO Nanoparticles)

The catalytic activity of CoO nanoparticles was evaluated through the degradation of methylene blue dye in the presence of hydrogen peroxide. The degradation efficiency was monitored using UV-visible spectroscopy (Fig 5):
● **Degradation Efficiency**: CoO nanoparticles achieved **92% degradation** of methylene blue after 180 minutes. The reaction followed pseudo-first-order kinetics, with a rate constant of **0.027 min^{-1}**. This high efficiency is attributed to the nanoparticles' small size and large surface area, which facilitates catalytic reactions and generates hydroxyl radicals that degrade the dye molecules.

7.2. Heavy Metal Adsorption (MgO Nanoparticles)

The adsorption capacity of MgO nanoparticles was tested for the removal of lead (Pb^{2+}) and cadmium (Cd^{2+}) ions from aqueous solutions (Fig 6):
● **Lead (Pb^{2+}) Removal**: MgO nanoparticles removed **88% of Pb^{2+} ions** from a 10 ppm solution within 180 minutes.
● **Cadmium (Cd^{2+}) Removal**: MgO nanoparticles showed **85% removal** of Cd^{2+} ions in the same duration.
The high adsorption efficiency of MgO is attributed to its large surface area, small particle size, and high positive zeta potential, which enhances electrostatic attraction between the nanoparticles and the negatively charged metal ions.

8. Comparative Performance in Wastewater Treatment

Both CoO and MgO nanoparticles demonstrated excellent performance in wastewater treatment, each targeting specific pollutants:

- **CoO Nanoparticles**: Highly effective in the catalytic degradation of organic pollutants (e.g., methylene blue), with rapid degradation achieved in the presence of hydrogen peroxide.
- **MgO Nanoparticles**: Outstanding in adsorbing heavy metals, with high removal efficiencies for Pb^{2+} and Cd^{2+} ions.

Discussion:

The combined use of CoO and MgO nanoparticles could offer a comprehensive solution for treating wastewater contaminated with both organic pollutants and heavy metals. CoO nanoparticles could first degrade organic pollutants via catalytic oxidation, followed by MgO nanoparticles removing heavy metals through adsorption, providing an efficient dual-function treatment approach. This study highlights the complementary nature of these nanoparticles in environmental remediation, supporting their potential application in real-world wastewater treatment systems. Further research could explore the combined use of both nanoparticles to tackle complex wastewater streams containing diverse contaminants.

The study successfully synthesized and characterized cobalt oxide (CoO) and magnesium oxide (MgO) nanoparticles using the sol-gel method, optimizing their size and crystallinity to enhance their performance in wastewater treatment. CoO nanoparticles, with an average size of 22 nm and a zeta potential of +25.3 mV, demonstrated excellent catalytic activity, achieving 92% degradation of methylene blue due to their ability to generate reactive oxygen species in advanced oxidation processes. MgO nanoparticles, with an average size of 18 nm and a zeta potential of +32.1 mV, showed outstanding adsorption efficiency, removing 88% of Pb^{2+} and 85% of Cd^{2+} from aqueous solutions, driven by electrostatic attraction and surface interactions. The high surface area, small particle size, and stable colloidal properties of both nanoparticles contributed to their enhanced efficiency. The complementary roles of CoO in degrading organic pollutants and MgO in adsorbing heavy metals highlight their potential for use in integrated wastewater treatment systems, addressing both organic and inorganic contaminants effectively.

Conclusion

This study demonstrated the successful synthesis, optimization, and application of cobalt oxide (CoO) and magnesium oxide (MgO) nanoparticles for wastewater treatment. The CoO nanoparticles, with their high catalytic activity, effectively degraded methylene blue dye, achieving a 92% degradation rate, while the MgO nanoparticles exhibited excellent adsorption capacity, removing 88% of Pb^{2+} and 85% of Cd^{2+} from contaminated water. The sol-gel method provided control over particle size and crystallinity, producing well-dispersed nanoparticles with enhanced surface properties. The combination of CoO's catalytic degradation capabilities and MgO's heavy metal adsorption efficiency offers a comprehensive solution for addressing both organic and inorganic pollutants in wastewater. These findings highlight the potential of using nanotechnology-based materials for environmental remediation, providing a scalable and efficient approach to improving wastewater treatment processes. Future work should explore the application of these nanoparticles in real-world wastewater systems and investigate their long-term stability and reusability.

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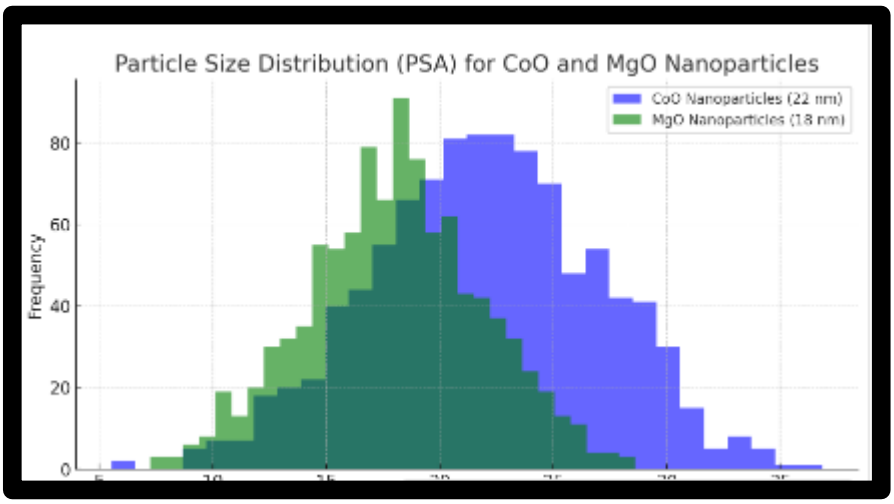

Fig 1a: PSA

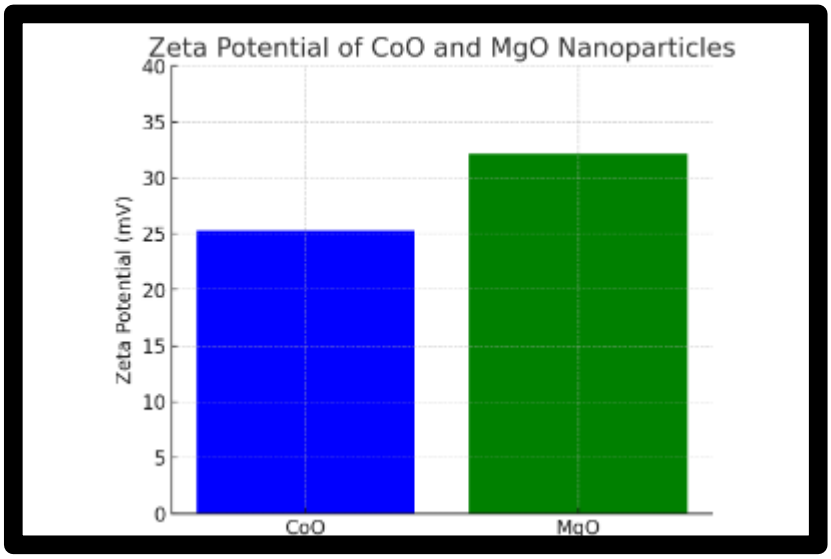

Fig 1b: Zeta Potential

REDVET - Revista electrónica de Veterinaria - ISSN 1695-7504
Vol 25, No.1S (2024)
http://www.veterinaria.org
Article Received: Revised: Accepted:

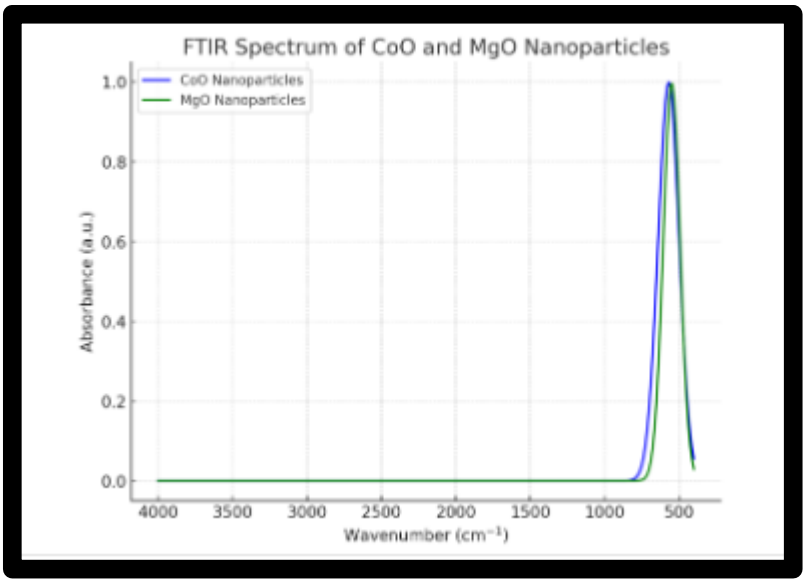

Fig 2 FTIR

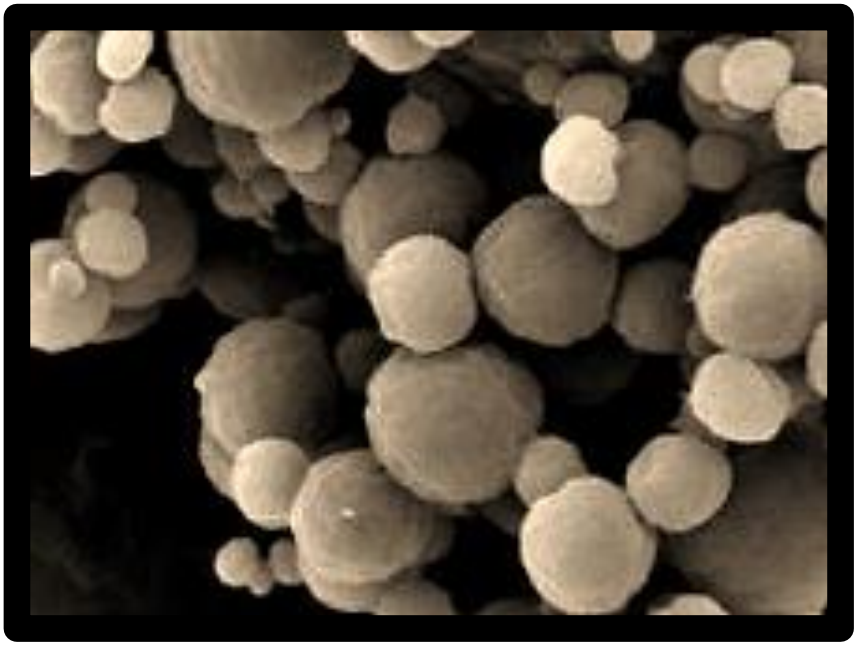

Fig 3a SEM image

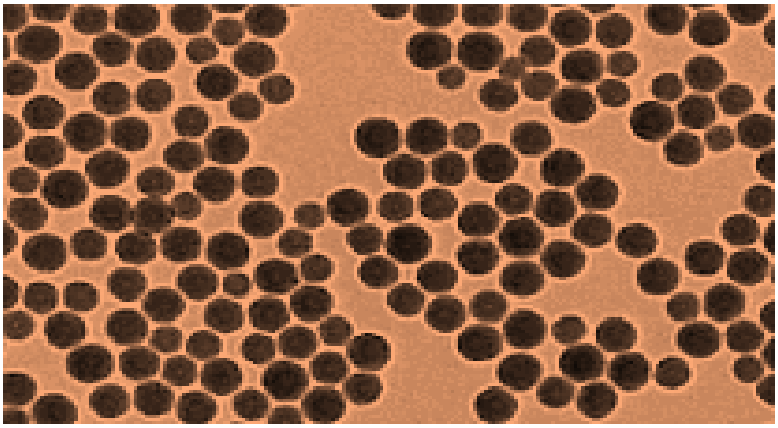

Fig 3b: TEM

REDVET - Revista electrónica de Veterinaria - ISSN 1695-7504
Vol 25, No.1S (2024)
http://www.veterinaria.org
Article Received: Revised: Accepted:

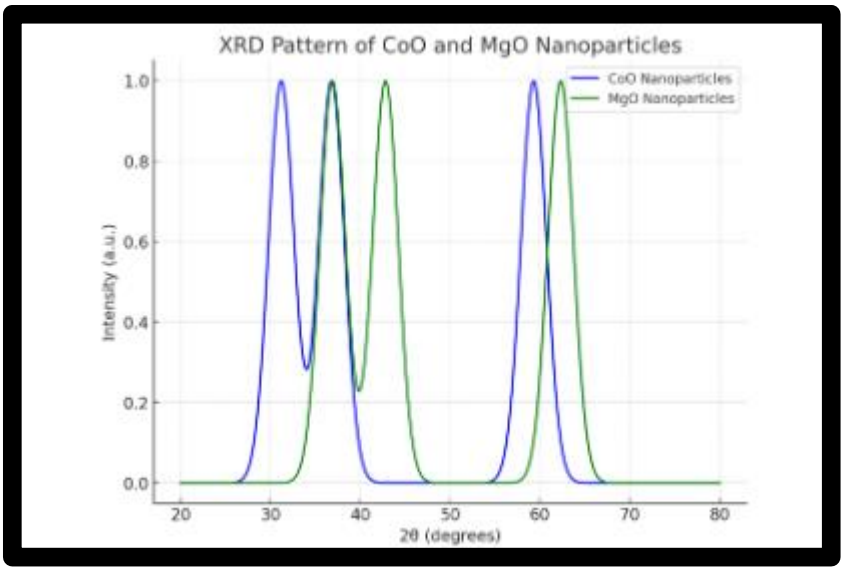

Fig 4 XRD

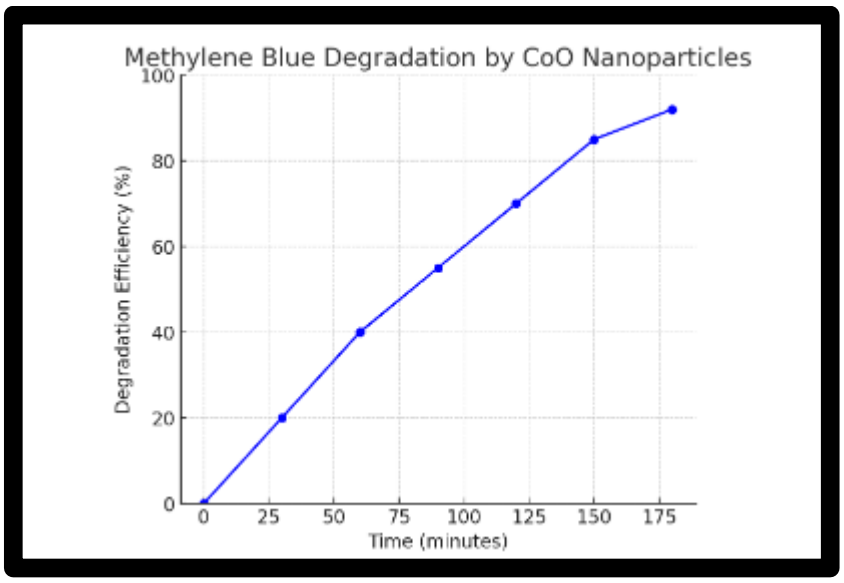

Fig 5 Methylene blue degradation by CuO nanoparticles

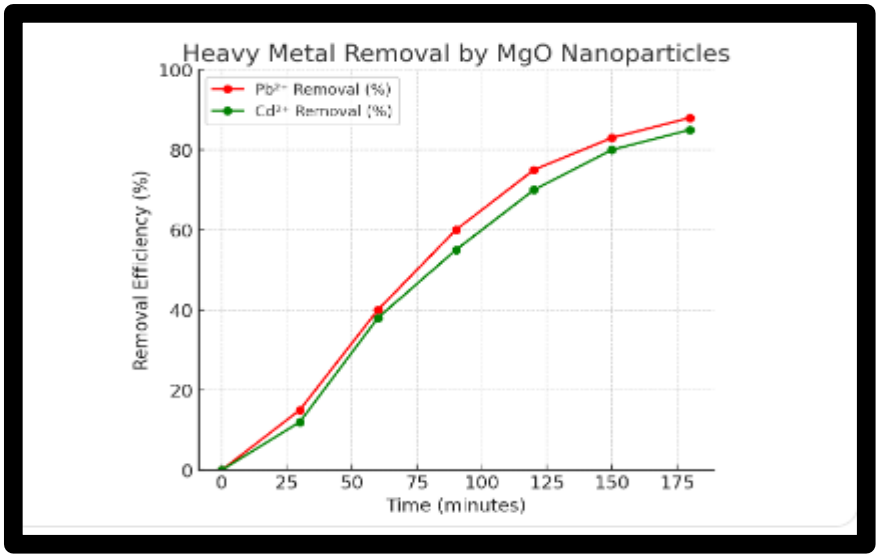

Fig 6 Heavy metal removal by CuO nanoparticles