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Eco-friendly Biosynthesis and Characterization: Metal Nanoparticle Fabricated by using Natural Extract

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ABSTRACT

Green synthesis has been successfully used to produce magnesium oxide (MgO) nanoparticles using *Nigella sativa* seed extract. These nanoparticles were investigated biophysically and morphologically using a variety of analytical techniques, including HR-TEM, XRD, FT-IR, SEM, and UV-Visible, in order to ascertain their structural, morphological, and functional characteristics. C-O stretching vibrations are usually attributed to the peak at 1080.25 cm^{-1}, which may be caused by NSSE biomolecules that assist in the stabilization and reduction of the MgO-NPs. Magnesium oxide nanoparticles are further supported by the peaks at 498.88 cm^{-1} and 434.43 cm^{-1}, which are indicative of metal-oxygen (Mg-O) bonding. The O K peak is close to 0.67 keV, and the MgK peak is around 1.34 keV. These results validate the successful synthesis of the nanoparticles by showing the presence of MgO.

Keywords: Bio-synthesis, Extract, Magnesium oxide Nanoparticle, *Nigella sativa*, Characterization, FT-IR, SEM, EDX.

INTRODUCTION

The eco-friendly and sustainable synthesis of metal oxide nanoparticles has garnered substantial research interest in recent years, driven by concerns over environmental pollution and the toxic implications of conventional chemical approaches. Eco-friendly green synthesis techniques are becoming more popular among researchers because they produce nanoparticles in a less dangerous manner. A wide variety of oxides can be formed by metals. Among various metal oxides, MgO-NPs have emerged as a focal point due to their remarkable physicochemical properties, particularly their high thermal stability, excellent catalytic activity, antibacterial properties, and broad applications in pharmaceuticals, wastewater treatment, and nanocomposites[1-4].

Green synthesis, an environmentally benign approach, utilizes plant extracts, microorganisms, or natural compounds as reducing and stabilizing agents to synthesize nanoparticles through environmentally benign methods, avoiding the utilization of hazardous chemicals. Consequently, a lot of effort has been put into developing eco-friendly methods for producing CQDs, CNTs, and even nanocomposites from natural biomass sources like vegetable waste[5,6].

Nigella sativa, commonly known as black cumin, is a medicinal plant rich in bioactive compounds such as alkaloids, flavonoids, phenolics, and essential oils, making it a suitable candidate for green synthesis. The bioactive compounds in *Nigella sativa* seed extract (NSSE) act as both reducing and stabilizing agents, facilitating the formation of magnesium oxide (MgO-NPs) nanoparticles while also enhancing their biocompatibility and stability. Green synthesis of magnesium oxide nanoparticles (MgO-NPs) using *Nigella sativa* (black cumin) seeds is an eco-friendly approach that leverages the plant's natural compounds to facilitate nanoparticle formation. This approach eliminates the need for toxic chemicals commonly utilized in traditional nanoparticle synthesis, enhancing sustainability and eco-friendliness. The biosynthesis of magnesium oxide nanoparticles (MgO-NPs) utilizing *Nigella sativa* seeds offers a sustainable and environmentally friendly strategy for nanoparticle fabrication. This technique harnesses the intrinsic phytochemicals within *Nigella sativa* as both reducing and stabilizing agents, thereby obviating the requirement for toxic chemicals conventionally employed in nanoparticle synthesis. The resulting MgO-NPs have demonstrated significant potential in various applications, including antimicrobial activity, antioxidant properties, and agricultural enhancements.

In a comparative context, MgO-NPs offer several advantages over other metal oxide nanoparticles. For instance, they exhibit excellent biocompatibility[7] and stability, making them suitable for biomedical applications[8-12]. Studies have shown that MgO-NPs possess substantial antioxidant[13-15] anticancer and antibacterial activity[16-18] which is attributed to their capacity for generating reactive oxygen species (ROS), which are effective against a broad spectrum of pathogens. Additionally, the synthesis cost of MgO-NPs is relatively lower than that of many other metal oxide nanoparticles, providing notable economic benefits.

In contrast, other nanoparticles like silver nanoparticles (Ag-NPs) synthesized using *Nigella sativa* extract (NSSE) have also demonstrated notable antimicrobial and antioxidant activities.

However, Ag-NPs can be associated with higher toxicity and cost, which may limit their widespread application.

Overall, the eco-friendly synthesis of MgO-NPs utilizing *Nigella sativa* seeds presents a sustainable and economically viable substitute for conventional techniques while yielding nanoparticles with advantageous properties suitable for diverse applications. This study explores the green synthesis of MgO-NPs using (NSSE) as a natural reducing agent. These are characterized by using various techniques such as UV-Visible, XRD, FT-IR, SEM, EDX, EDS and HR-TEM to determine their structural, morphological, and functional properties. The green synthesis approach provides an eco-friendly alternative and enhances the biomedical and industrial potential of MgO nanoparticles, paving the way for sustainable nanotechnology applications.

MATERIALS AND METHODS

Materials

Methanol and Magnesium nitrate hexahydrate, used for nanoparticle synthesis, was procured from Fischer scientific. Sodium hydroxide pellets from Fischer Scientific, DMSO from MDH and Acetone from Sigma Aldrich. Double distilled water was used in the whole experimental process.

Extraction of *Nigella sativa* seed extract by cold percolation method

Cleaned and fresh *Nigella sativa* seeds were purchased from the local market of Aligarh, UP, India. Seeds were washed with deionized water to remove the impurities. *Nigella sativa* was allowed to dry in the oven at 80°C for 3 days. After drying, the seeds were powdered using mortar and pestle. This powder was now used for nanoparticle synthesis. The crude methanolic extract was prepared by macerating 40 g of dried plant powder in 400 mL of methanol, followed by continuous agitation on a rotary shaker for 72 hours. The resulting extract was then filtered and stored in airtight bottles at 4°C for subsequent analysis.

Bio-synthesis of Novel MgO-NPs by NSSE

MgO-NPs were synthesized via a green synthesis approach; NSSE was incorporated into the 1M solution of magnesium nitrate hexahydrate ($Mg(NO_3)_2•6H_2O$), allowing bioactive compounds to act as reducing and capping agents. The reaction mixture was subsequently stirred for 30 min during which 100 mL of 2M NaOH solution was gradually introduced dropwise to maintain the pH within the range of 9–12. This step induced a gradual colour change, indicating the nucleation and precipitation of magnesium hydroxide $(Mg(OH)_2$. The NaOH addition was halted once the pH reached 11.2. After completing the reaction, the gel-like precipitate was separated using Whatman filter paper and allowed to settle for a specified period. The precipitate was washed multiple times with distilled water to remove any residual unreacted compounds. The resulting material was dried overnight at 80°C and then ground with an agate mortar to obtain a homogeneous powder. This dried $Mg(OH)_2$ powder was subsequently calcinated in a muffle furnace at 500–600°C for 1 h resulting in its thermal decomposition into MgO nanoparticles. The synthesized MgO nanoparticles were then characterized to confirm their morphological, structural, and compositional properties.

RESULT AND DISCUSSION

FT-IR

The provided FT-IR spectrum (Fig. 1(a)) corresponds to MgO nanoparticles synthesized via a green synthesis method using *Nigella sativa* seeds. The spectrum reveals several distinct absorption bands indicative of various functional groups and molecular vibrations associated with the MgO nanoparticles. The broad absorption peak at 3438.31 cm^{-1} corresponds to the stretching vibrations of hydroxyl (-OH) groups, signifying the existence of adsorbed water molecules on the MgO-NPs. The peak observed at 1635.55 cm^{-1} is attributed to the bending vibrations of water molecules (H-O-H), further confirming the presence of surface moisture. The absorption band at 1446.20 cm^{-1} may be associated with C-H bending vibrations, likely originating from residual organic compounds from the plant extract used in the synthesis process. The peak at 1080.25 cm^{-1} is typically assigned to C-O stretching vibrations, possibly due to biomolecules from NSSE involved in the reduction and stabilization of the MgO-NPs. The absorption bands observed at 881.73 cm^{-1}, 650.72 cm^{-1}, and 608.81 cm^{-1} indicate Mg-O stretching vibrations, which confirm the synthesis of MgO nanoparticles[8]. Additionally, the peaks at 498.88 cm^{-1} and 434.43 cm^{-1} are characteristic of metal-oxygen (Mg-O) bonding, further corroborating the presence of magnesium oxide nanoparticles[19,20].

Overall, the FT-IR analysis confirms the successful synthesis of MgO-NPs, with evidence of surface hydroxylation and biomolecular interactions contributing to their stability and functional properties.

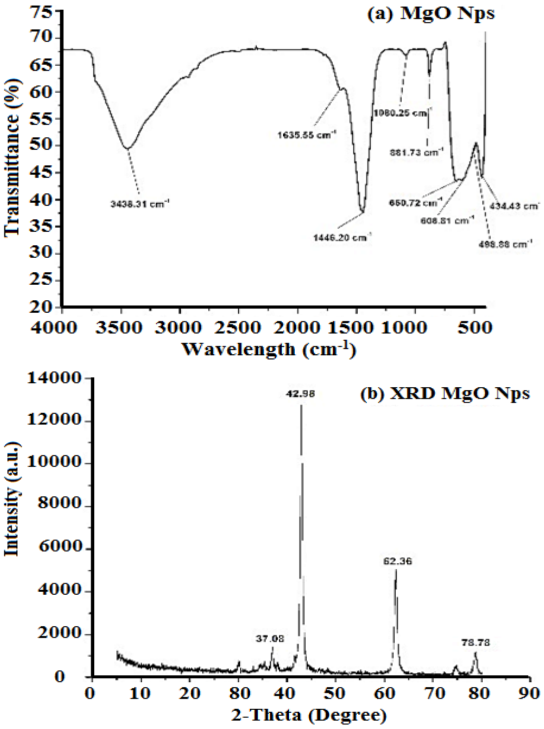

Fig. 1(a). FT-IR spectra and (b) XRD spectra of MgO-Nps synthesized from NSSE

XRD

The X-ray diffraction pattern presented corresponds to MgO-Nps synthesized via a green synthesis approach using NSSE (Fig. 1(b)). The diffraction peaks observed at 37.08°, 42.98°, 62.36°, and 78.78° (2θ values) indicate the crystalline nature of the synthesized MgO nanoparticles. The utmost intense peak at 42.98° corresponds to the (200) plane, a characteristic reflection of MgO's face-centered cubic (FCC) structure. Additional diffraction peaks at 37.08°, 62.36°, and 78.78° are indexed to the (111), (220), and (311) planes, respectively, confirming the cubic structure of MgO nanoparticles. Thus, the XRD analysis confirms the formation of highly crystalline, pure MgO nanoparticles with a cubic structure. The green synthesis method using NSSE proves to be an effective and eco-friendly route for nanoparticle fabrication.

UV-Visible Spectrum

The UV-Visible absorption spectrum (Fig. 2(a)) presented corresponds to magnesium oxide MgO-Nps synthesized by using NSSE. The spectrum features a strong absorption peak at 224 nm, characteristic of MgO nanoparticles. This peak is attributed to electronic transitions within the MgO lattice, primarily involving charge transfer transitions between oxygen and magnesium ions. The pronounced absorption indicates an efficient quantum confinement effect in the synthesized MgO nanoparticles, commonly observed in nano-scale materials due to their reduced size and increased surface-to-volume ratio.

The results from UV-Visible spectroscopy confirm the successful synthesis of MgO-NPs, showcasing a strong quantum confinement effect and high crystallinity. The observed blue shift in absorption, when compared to bulk MgO, signifies a reduced particle size and enhanced optical properties. The eco-friendly synthesis approach utilizing *Nigella sativa* extract produces nanoscale MgO, which has potential applications in various fields.

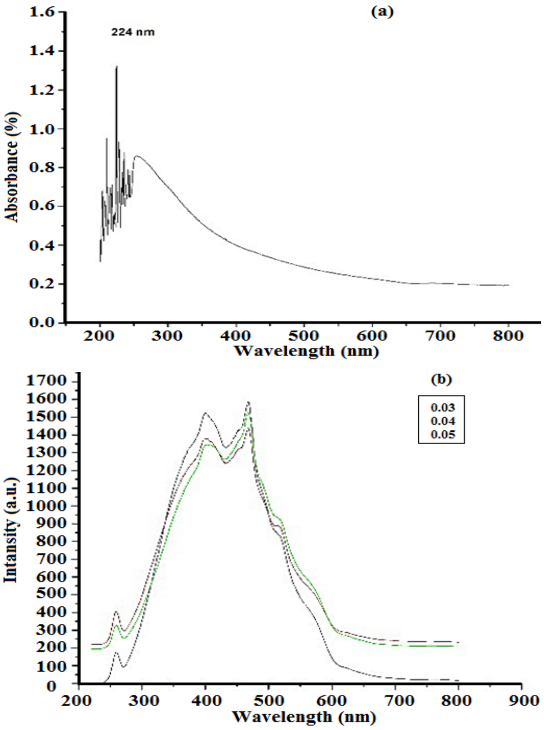

Fig. 2(a). UV-Visible spectra and (b) PL spectra of MgO-NPs synthesized from NSSE

PL studies

The provided photoluminescence (PL) spectra correspond to MgO-NPs synthesized using a green synthesis approach with NSSE at varying precursor concentrations (0.03, 0.04, and 0.05 M) (Fig. 2(b)). PL spectroscopy is a crucial analytical technique to investigate the optical and electronic properties of semiconductor and nanomaterial systems by examining their emission characteristics upon excitation. The sample synthesized at 0.03 M shows the highest emission intensity, suggesting higher oxygen vacancies and defect site density. As the concentration increases (0.04 M and 0.05 M), the intensity decreases slightly, which may indicate a reduction in defect-related emissions due to improved crystallinity. The PL spectra of green-synthesized MgO nanoparticles demonstrate strong defect-related emissions, particularly oxygen vacancy states in the blue-green spectral range. The variation in PL intensity with precursor concentration suggests that lower concentrations (0.03 M) lead to higher defect density. In contrast, higher concentrations (0.04 M and 0.05 M) improve crystallinity and reduce defect-related emissions.

Scanning electron microscopy (SEM)

The SEM image provides a detailed visualization of the morphology of MgO-NPs synthesized using NSSE. The micrograph, captured at a magnification of 30,000, reveals that the MgO nanoparticles exhibit a predominantly quasi-spherical shape with a tendency to form agglomerates. This agglomeration is owing to the high surface energy and strong interparticle interactions, which are common characteristics of nanoparticles. The observed nanostructured morphology suggests a uniform particle size distribution with some degree of porosity. The average particle size of the MgO nanoparticles in the given SEM image is approximately 6.5 nm. Overall, the SEM analysis confirms the successful biosynthesis of MgO nanoparticles with a well-defined nanostructure.

Energy Dispersive X-ray (EDX)

The Energy Dispersive X-ray (EDX) spectrum corresponds to magnesium oxide (MgO) nanoparticles synthesized using *Nigella sativa* seed extract. The spectrum prominently features characteristic peaks of magnesium (Mg) and oxygen (O), confirming the composition of the synthesized nanoparticles. The Mg K peak appears around 1.34 keV, while the OK peak is near 0.67 keV. These findings indicate the presence of MgO, confirming the successful synthesis of the nanoparticles.

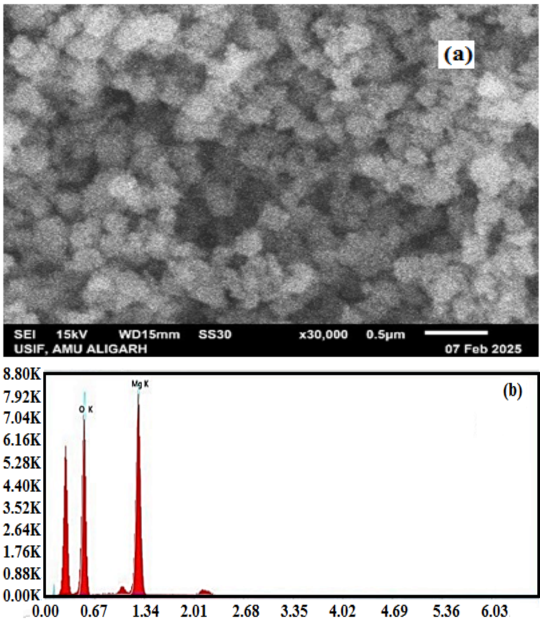

Fig. 3(a). SEM image and (b) EDX spectra of MgO-NPs synthesized from NSSE

Elemental Mapping Analysis of Green-Synthesized MgO Nanoparticles

The given energy dispersive X-ray spectroscopy (EDS) elemental mapping images correspond to MgO nanoparticles synthesized via green synthesis using *Nigella sativa* seed extract. Elemental mapping is an essential technique used in scanning electron microscopy (SEM-EDS) to analyze the spatial distribution of elements within a sample, confirming the composition and homogeneity of the synthesized material.

The figure provides a morphological overview of the MgO Nanoparticle. The Nanoparticle exhibit an aggregated structure with irregularly shaped clusters, which is common in MgO synthesized through green methods. The bright regions correspond to high atomic number elements (likely MgO clusters), while the darker regions represent lower-density areas.

Oxygen (O-K) Mapping

The green-coloured elemental map represents the distribution of oxygen (O-K edge) in the sample. Oxygen is evenly distributed, indicating the presence of MgO rather than pure Mg clusters. The high intensity of oxygen signals confirms that the synthesized nanoparticles contain MgO rather than metallic Mg.

Magnesium (Mg-K) Mapping

The red-coloured elemental map corresponds to the magnesium (Mg-K edge) distribution. The Mg signal matches well with the oxygen distribution, confirming the formation of magnesium oxide (MgO) rather than separate Mg or Mg (OH) phases. The uniform spread of Mg signals across the sample supports the hypothesis that MgO is evenly distributed and homogeneously synthesized. The use of NSSE as a green reducing and stabilizing agent has likely influenced the morphology and elemental dispersion of the MgO-NPs. The SEM-EDS elemental mapping confirms the successful synthesis of MgO-NPs, with homogeneous magnesium and oxygen distribution. The green synthesis technique using *Nigella sativa* extract has effectively produced MgO without major impurities, making the material suitable for applications in catalysis, biomedical sciences, and environmental remediation.

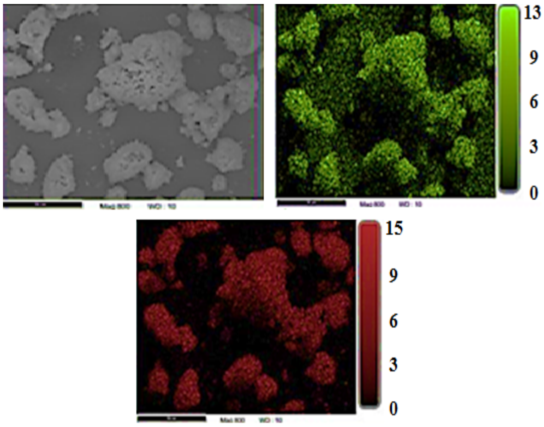

Fig. 4. Elemental Mapping Analysis of Green-Synthesized MgO Nanoparticles showing Oxygen (O-K) Mapping and Magnesium (Mg-K) Mapping

High-Resolution Transmission Electron Microscopy Analysis of MgO Nanoparticles

The HR-TEM image presents the morphological characteristics and structural details of MgO-NPs synthesized using NSSE. The nanoparticles exhibit an agglomerated structure with a quasi-spherical shape, indicative of their high surface energy and potential interactions among particles. The scale bar of 100 nm provides a reference for estimating the nanoparticle dimensions, indicating that the particles are in the nanoscale range. The observed morphology and particle distribution suggest that the green synthesis approach successfully produces MgO nanoparticles with controlled size and shape.

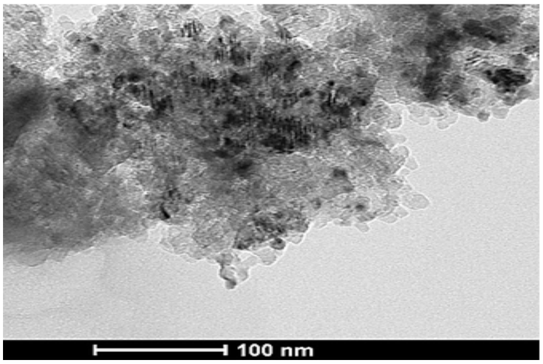

Fig. 5. HR-TEM image of Green-Synthesized MgO Nanoparticles

CONCLUSION

The eco-friendly biosynthesis of MgO-NPs using the plant extracts involves the reduction of metal ions to nanoparticles. NSSE have been successfully extracted and utilized for the synthesis of NPs since it contains a wide range of alkaloids, flavonoids, phenolics, and essential oils.The development of dependable and environmentally sustainable methods for producing metallic nanoparticles is an important advancement in the field of applied nanotechnology. The long-term chemical and physical characteristics of biosynthesized nanoparticles need to be better understood, especially in light of the potential applications of nanotechnology in daily life.

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Conflicts of Interest

The authors declare no competing interests.

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