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Hibiscus rosa-sinensis Plant Extract Assisted Microwave Synthesis of Magnesium Ferrite Nanoparticles

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ABSTRACT

The magnetic and structural properties of magnesium ferrite (MgFe_2O_4) nanoparticles (NPs) have fascinated the attention of researchers. MgFe_2O_4 NPs were prepared by *Hibiscus rosa-sinensis* plant extract assisted microwave combustion technique. The as prepared MgFe_2O_4 NPs were subjected to structural, magnetic, and structural properties using XRD, FT-IR, SEM and VSM analysis. Cubic phase was observed through XRD studies and the calculated lattice constant value is 3.845\AA . SEM technique confirmed the sphere-shaped nanostructured morphology of the MgFe_2O_4 ferrites. VSM studies were used to calculate the magnetization, coercivity and retentivity of the MgFe_2O_4 . M-H loop revealed the magnetic behavior of the prepared MgFe_2O_4 ferrite with superparamagnetic. The photocatalytic degradation of the synthesized spinel ferrites with methylene blue (MB) as organic pollutant was also studied.

Key words: Cubic MgFe_2O_4 , *Hibiscus rosa-sinensis*, Plant extract, Photocatalytic degradation, Superparamagnetic

Hazardous pollutants manifesting in water bodies represent a threat to individuals and the environment [1]. To minimize these dangerous pollutants, procedures such as activated carbon adsorption, chlorination, ultrafiltration, and ozonation treatment have been applied. Nonetheless, none of the treatments listed above are cost-effective [2]. The development of an environmentally acceptable, cost-effective approach for the degradation of dyes and pollutants utilizing semiconducting catalyst has so piqued academics' interest [3]. Ferrites are well-known for their remarkable uses in opto-magnetic and photoelectronic materials, allowing low-energy photons to display optical absorption and presenting a perfect electronic structure for photocatalytic applications [4]. Bacterial infections are a serious threat to humans, and spinel ferrite nanoparticles have the ability to kill hazardous germs. The quest for new materials that can be used as antibacterial agents, as well as the evaluation of their properties, has become a promising research area [5- 6].

Magnesium ferrite [7–10], a semiconductor spinel ferrite with higher chemical stability and more catalytic sites, is a promising candidate for photocatalysis. Calcium ferrite is used in biomedicine, drug administration, magnetic resonance imaging, and magnetic hyperthermia, among other things [11-15]. In addition, transition metal ions were used to change the characteristics of calcium ferrites, resulting in doped calcium

ferrites. Spinel ferrite nanoparticles can be prepared using a variety of procedures, including hydrothermal, ceramic, auto-combustion, mechanical milling, sol-gel, and coprecipitation. Because of the high yield, homogeneity, and small particle size, the coprecipitation approach provides the most advantages [16-20]. Herein we propose to synthesize MgFe_2O_4 NPs by *Hibiscus rosa-sinensis* plant extract assisted microwave combustion technique and studied their structural, morphological, and magnetic electrical properties. The as prepared nano spinel ferrites were tested for its photo catalytic degradation over methylene blue dye as organic pollutant.

MATERIALS AND METHODS

Iron (III) nitrate, magnesium (II) nitrate (Sigma-Aldrich, 99%), used microwave assisted using *Hibiscus rosa-sinensis* plant extract green synthesis of MgFe_2O_4 NPs. Deionized water was utilized in all stages of the synthesis. Malachite (MG) was purchased for the photocatalytic degradation. The *Hibiscus rosa-sinensis* plant extract was washed with deionized water. Plant moisture was removed at 50°C . To each gram of watercress powder, 10 ml of deionized water was added and shaken at 50°C , overnight. Finally, the extract was separated with Whatman paper and centrifuged. Iron (III) nitrate, magnesium (II) nitrate solution was added to 50 ml of aqueous extract. The mixture was kept in a microwave oven. The final product was washed well with DI water and ethanol twice finally dried at 70°C and used for further characterizations.

Characterization techniques

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The structural characterization of MgFe_2O_4 NPs was performed using Rigaku Ultima X-ray diffractometer equipped with $\text{Cu-K}\alpha$ radiation ($\lambda = 1.5418 \text{ \AA}$). The surface functional groups were analyzed by Perkin Elmer FT-IR spectrometer. Morphological studies and energy dispersive X-ray analysis (EDX) of MgFe_2O_4 NPs have been performed with a Jeol JSM6360 high resolution scanning electron microscopy (HR-SEM). Magnetic measurements were carried out at room temperature using a PMC MicroMag 3900 model vibrating sample magnetometer equipped with 1 Tesla magnet.

RESULTS AND DISCUSSION

Powder - XRD diffraction studies

The structural properties of MgFe_2O_4 NPs were studied from the diffraction pattern (Fig 1) obtained from XRD

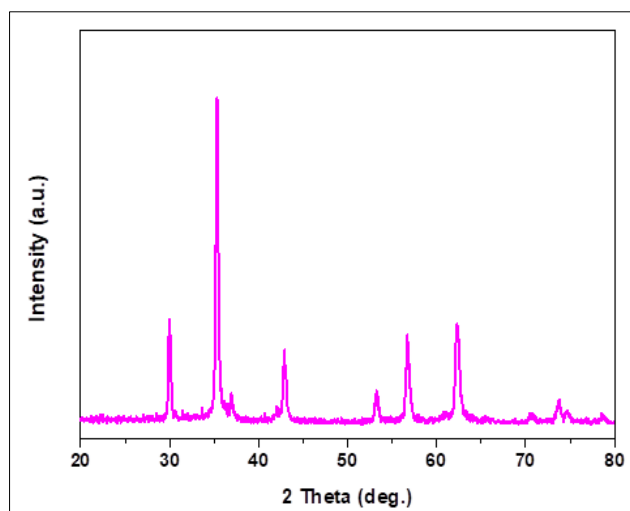


Fig 1 Powder XRD pattern of MgFe_2O_4 NPs

SEM analysis

SEM images (Fig 3) presented the surface morphology of the prepared spinel MgFe_2O_4 NPs. SEM images specified that the materials were smaller particle size of the material

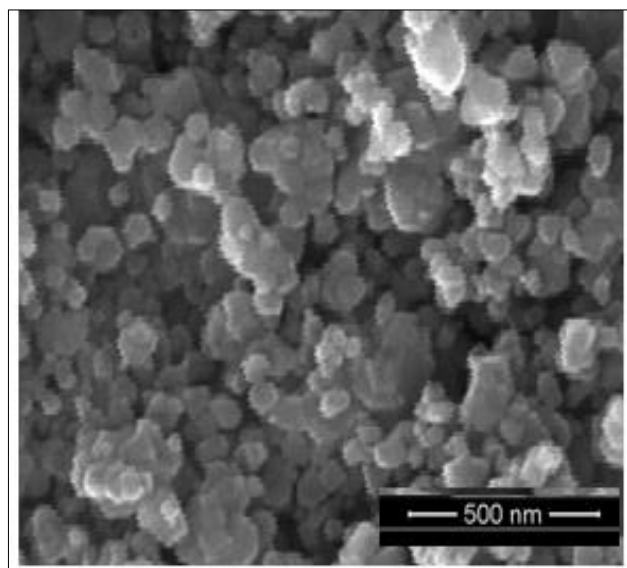


Fig 3 SEM image of MgFe_2O_4 NPs

technique [21]. The existence of the lattice planes in the XRD patterns elucidates the structure of single-phase cubic structure of MgFe_2O_4 NPs with space group $Fd3m$. The average crystallite size D was calculated from Scherrer formula and obtained 18.26 nm.

FT-IR analysis

The FT-IR spectra of the synthesized MgFe_2O_4 NPs were studied between $400\text{--}4000 \text{ cm}^{-1}$. The characteristic spinel absorption band at 450 and 600 cm^{-1} were accredited to the stretching vibrations due to the interaction of oxygen and cations in Mg-O and Fe-O bond linkages. The bands at 700 and 800 cm^{-1} were due to O-Fe-O and Fe-OH linkages and the band at 455 cm^{-1} related to Fe-O linkage. The absorption band at 642 cm^{-1} was attributed to Fe-O bond of MgFe_2O_4 NPs skeleton. The broad bands at 3420 and 1622 cm^{-1} were assigned to the O-H broadening due to the compaction of KBr pellets [22].

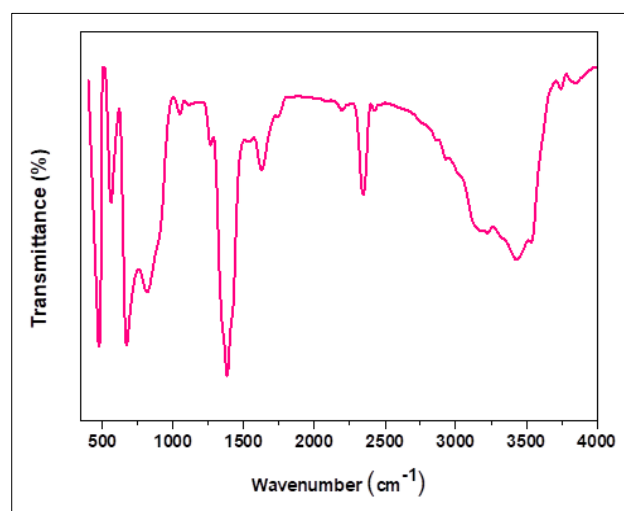


Fig 2 FT-IR spectra of MgFe_2O_4 NPs

ranges between $12\text{--}20 \text{ nm}$. The images indicated the aggregation of the material with cubical and regular morphology of MgFe_2O_4 NPs. High diameter pictures depicted agglomerations resulting in the formation of heterogeneous surface.

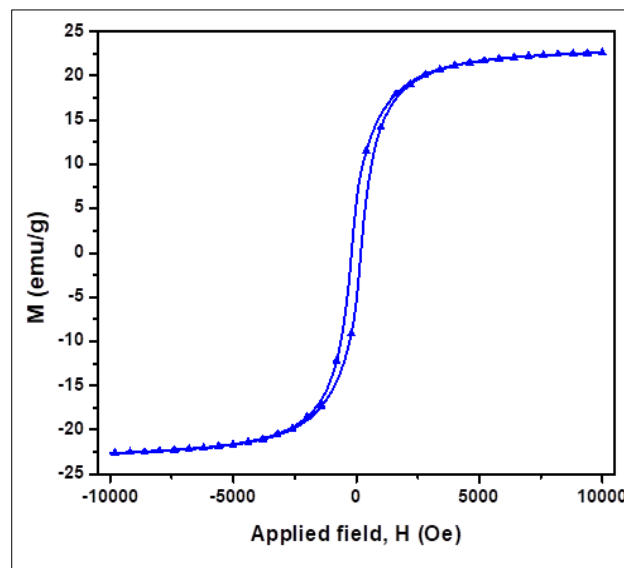


Fig 4 VSM results of MgFe_2O_4 NPs

VSM studies

The hysteresis curve (Fig 4) of the synthesized MgFe_2O_4 NPs showed superparamagnetic behaviour, indicating the synthesized MgFe_2O_4 NPs as soft ferrites [23]. The coercivity was found to be 246.24 Oe. The M_s , and M_r , value of the MgFe_2O_4 NPs is 23.58 emu/g and 52.85 emu/g respectively. Lower value H_c of the synthesized MgFe_2O_4 NPs showed the magnetically soft ferrite and distortion of spin happens on the surface due to the magneto-crystalline anisotropy.

Photocatalytic degradation studies

The photocatalytic experiment was done by using MgFe_2O_4 NPs and 100 mL of 25 ppm organic methylene blue dye solution. The photocatalytic reaction media was left for stirring for 30 minutes to get adsorption equilibrium before the illumination of UV- lamp. The photo catalytic studies were performed for 180 minutes under the irradiation of UV-lamp and the solutions were drawn every 30 minutes and measured with UV-Visible spectra (Fig 5) and showed 96.25% efficiency of dye degradation for MgFe_2O_4 NPs in 180 minutes attributed to the smaller crystallite size [24].

CONCLUSION

The as prepared MgFe_2O_4 NPs were subjected to structural, magnetic, and structural properties using XRD, FT-IR, SEM and VSM analysis. MgFe_2O_4 NPs were prepared by *Hibiscus rosa-sinensis* plant extract assisted microwave combustion technique. Cubic phase was observed through XRD studies and the calculated lattice constant value is 3.845 Å. SEM technique confirmed the sphere-shaped nanostructured morphology of the MgFe_2O_4 ferrites. VSM studies were used to calculate the magnetization, coercivity and retentivity of the MgFe_2O_4 . M-H loop revealed the magnetic behavior of the prepared MgFe_2O_4 ferrite with superparamagnetic. VSM analysis indicated that the as prepared MgFe_2O_4 NPs were soft ferrites. MgFe_2O_4 NPs exhibited 96.25% degradation in 180 minutes with methylene blue dye.

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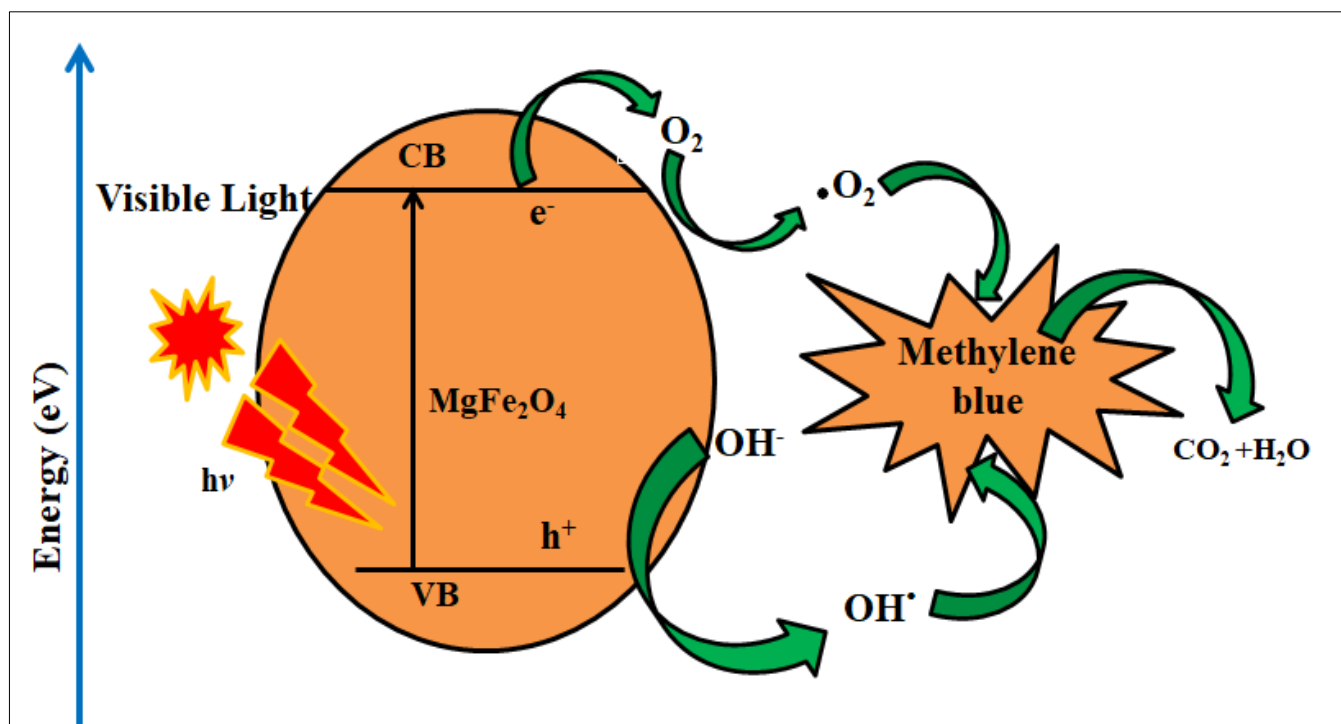


Fig 5 Photocatalytic dye degradation using MgFe_2O_4 NPs

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