Synthesis and Characterization of Nanocrystalline $MgFe_2O_4$ for Multifaceted Applications

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Abstract

A polycrystalline $MgFe_2O_4$ ferrite sample was successfully synthesized using the sol-gel process. X-ray diffraction measurements and analysis confirmed the single-phase formation of a nanocrystalline cubic spinel structure without impurity phases. The lattice parameters, refined via the Rietveld method, was determined to be 8.38 Å, and the crystallite size was found to be approximately 12nm using the Debye-Scherrer method. FTIR measurement revealed a prominent peak at 3710 cm^{-1} corresponding to the O–H vibrational mode, alongside a weak shoulder at 3200–3270 cm^{-1} attributed to O–H elongating vibrations of adsorbed H_2O molecules. A weak peak at 2364 cm^{-1} was assigned to nitrate groups. These results confirm the successful synthesis and nanoscale characterization of $MgFe_2O_4$ with distinct structural and vibrational properties.

Keywords: Ferrite, XRD, FTIR, Rietveld, Sol-Gel.
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Introduction

Spinel ferrites have drawn significant interest owing to their versatile chemical and physical properties, which make them suitable for numerous technological applications. Magnesium ferrite ($MgFe_2O_4$), a prominent member of the general formula of spinel ferrite family MFe_2O_4 (where M represents divalent cations like Mg^{2+}, Mn^{2+}, Zn^{2+}, Ni^{2+}, Co^{2+}, or Cd^{2+}), exhibits an inverse spinel cubic structure.

Achieving the synthesis of ferrite nanoparticles with precise control over particle size distribution, uniform morphology, and reduced agglomeration remains a significant challenge to meet the needs of advanced applications. Importantly, ferrite nanoparticles exhibit enhanced electrical and magnetic properties compared to their microscale counterparts. In recent years, magnesium ferrite nanoparticles have gained significant interest for their potential applications in semiconductors, low-magnetic materials, hyperthermia therapy, and gas sensors.

Synthesis Method

The polycrystalline $MgFe_2O_4$ ferrite sample was synthesized via the sol-gel method, a commonly utilized and efficient approach for nanoparticle fabrication [1-5]. Analytical-grade reagents were employed, with $[Mg(NO_3)_2 \cdot 6H_2O]$ and $[Fe(NO_3)_3 \cdot 9H_2O]$ serving as metal ion precursors. Citric acid was used as a chelating agent to enhance metal ion complexation and improve solution stability. The nitrates and citric acid were measured in stoichiometric ratios and dissolved in a solvent within a beaker. The solution was stirred continuously for 30 minutes using a magnetic stirrer. To achieve a pH of approximately 10, ammonia was added dropwise, with the pH maintained within the range of 10–12 to promote particle size reduction. The addition of ammonia induced a color change, signifying the initiation of gel formation during constant heating and stirring. The resulting gel was subsequently dried, and the obtained nano powder was finely ground using a mortar and pestle. The complete process for $MgFe_2O_4$ ferrite is depicted in Figure 1.

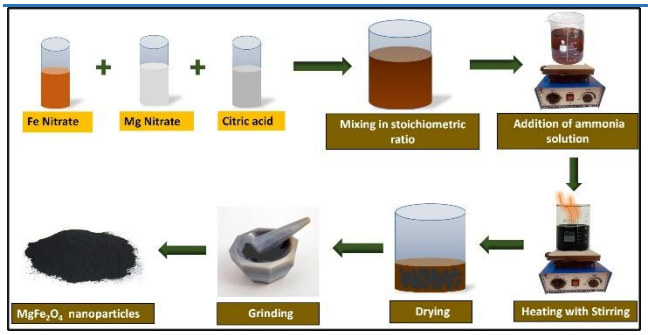

Figure 1: Synthesis procedure of $MgFe_2O_4$ nanoparticles.

Results and discussion

To determine the crystal structure and size, the XRD technique was used. Structural analysis of the synthesized $MgFe_2O_4$ sample was performed using an XRD (Bruker D8 Advance) used with a CuKα source (λ = 1.5406 Å). X-ray data were measured at 300K temperature over a 2θ range of 20°–70°. The diffraction peaks established the formation of a cubic spinel single-phase structure.

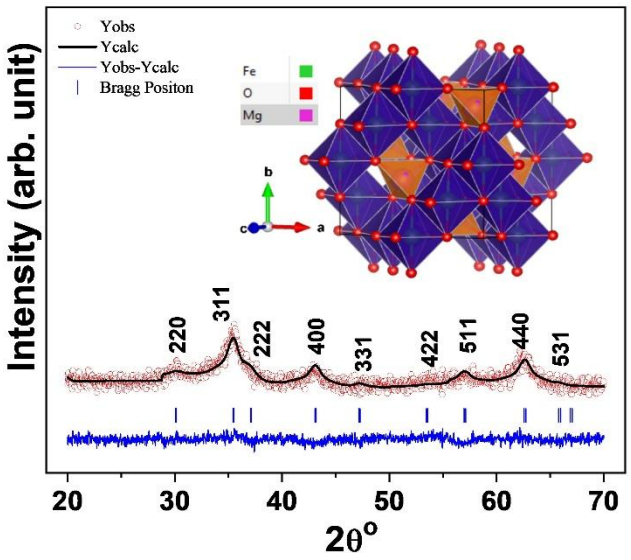

Figure 2: XRD pattern with crystal structure of $MgFe_2O_4$.

The lattice constant, refined using the Rietveld method, was determined to be 8.38 Å, as shown in Figure 2. Investigation of the XRD pattern also established the nanocrystalline nature of the sample, with no additional or impurity peaks observed. the Debye-Scherrer method was used for the crystallite size, based on the FWHM of XRD peaks, and was found to be approximately 12 nm. The broad peaks in the diffraction patterns further validate the nanoscale dimensions of the particles. The FTIR spectrum shown in Figure 3 reveals a prominent peak at 3710 cm^{-1}, corresponding to the O–H vibrational mode. A weak shoulder at 3200–3270 cm^{-1} is associated with the O–H vibrations of adsorbed H_2O molecules. Vibrational modes of adsorbed, free, or corresponding anions, along with twisting vibrations of actually interlayer and adsorbed H_2O molecules, are detected in the 2500–1000 cm^{-1} range.

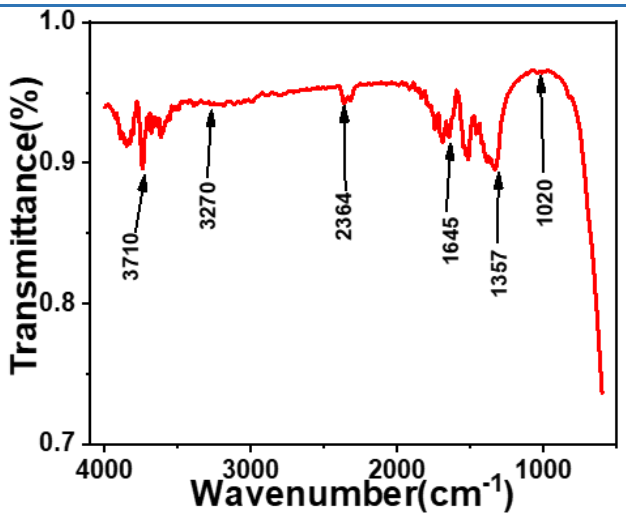

Figure 3: FTIR Spectrum of nanocrystalline $MgFe_2O_4$ Ferrite.

A twisting vibration of H_2O molecules appears at 1645 cm^{-1}, while a weak peak at 2364 cm^{-1} is accredited to nitrate groups. Additionally, a berm at 1357 cm^{-1} (ν_3 irregular stretching) and feeble peaks at 1020 cm^{-1} (ν_1 proportional stretching), 830 cm^{-1} (ν_2 out-of-plane bending), and 690–696 cm^{-1} (ν_4 scissoring distortion) correspond to the fundamental vibrational modes of NO_3^- ions derived from the initial nitrate salts.

Conclusion

The polycrystalline $MgFe_2O_4$ ferrite sample was successfully produced via the sol-gel procedure. X- ray investigation confirmed the creation of a nanocrystalline cubic spinel construction, with no other impurity phases in the sample. The lattice constant, refined using the Rietveld method, was found to be 8.38 Å, and the crystallite size was assessed to be around 12 nm using the Debye-Scherrer method. FTIR analysis showed a prominent peak at 3710 cm^{-1}, corresponding to the O–H vibrational mode, along with a weak shoulder at 3200–3270 cm^{-1}, associated with the O–H elongating vibrations of adsorbed and H_2O molecules. Additionally, a weak peak at 2364 cm^{-1} was attributed to nitrate groups. These findings confirm the successful synthesis and characterization of nanoscale $MgFe_2O_4$ with distinctive vibrational properties.

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