

Microwave-Assisted Green Combustion Synthesis of Phase-Pure MgFe_2O_4 Nanoparticles using *Tinospora cordifolia*: Structural Characterization and Electrical Relaxation Behaviour

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Phase-pure MgFe_2O_4 nanoparticles were successfully synthesized via a rapid microwave-assisted green combustion route using *Tinospora cordifolia* (giloy) extract as a natural phytochemical combustion fuel without post-combustion calcination of the synthesized powder. The crystal structure and microstructure of the synthesized nanoparticles and the electrical properties of sintered pellets prepared from the synthesized powder were systematically investigated using X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy, scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS) and temperature-dependent dielectric measurements. XRD confirmed the formation of a single-phase cubic spinel structure (space group $Fd\bar{3}m$) without detectable secondary phases. The average crystallite sizes estimated by the Debye–Scherrer, Williamson–Hall (UDM) and size–strain plot (SSP) methods were 15.10, 18.36 and 18.26 nm, respectively. The close agreement among these values confirms the nanocrystalline nature of MgFe_2O_4 , with relatively low lattice strain. FTIR spectra exhibited the characteristic tetrahedral and octahedral metal-oxygen vibrational bands of the spinel lattice, while SEM and EDS analyses revealed a porous agglomerated morphology and confirmed the presence of Mg, Fe and O. Dielectric measurements performed over the frequency range of 1 Hz–100 kHz and the temperature range from room temperature to 400 °C showed pronounced temperature- and frequency-dependent dielectric behaviour consistent with Maxwell-Wagner interfacial polarization and thermally activated electron hopping processes. The AC conductivity exhibited thermally activated semiconducting behaviour, whereas electric modulus analysis revealed broad relaxation features characteristic of non-Debye dielectric relaxation. The relaxation times derived from the consistently selected high-temperature M'' relaxation features followed an approximately Arrhenius-type dependence, with an activation energy of 0.758 eV.

Keywords: MgFe_2O_4 , Microwave, Green synthesis, Electric modulus, Electrical relaxation.

INTRODUCTION

Spinel ferrites with the general formula MFe_2O_4 , where M is a divalent metal ion, have attracted considerable interest due to their tunable structural, electrical, magnetic, catalytic and optical properties. These materials generally crystallize in the cubic spinel structure with space group $Fd\bar{3}m$, in which oxygen ions form a close-packed framework and metal cations occupy tetrahedral (A) and octahedral (B) sites. Cation distribution between these sublattices strongly affects charge transport, dielectric polarization and magnetic interactions, supporting applications in sensors, catalysis, electromagnetic components, energy-storage systems and high-frequency

devices [1-5]. Among them, magnesium ferrite (MgFe_2O_4) is attractive due to its partially inverse spinel structure, chemical stability, relatively high electrical resistivity, moderate dielectric response and environmentally benign composition. Its properties are sensitive to cation distribution, crystallite size, lattice strain, grain boundaries, porosity and defects, which are strongly influenced by the synthesis route and thermal treatment [6-9].

MgFe_2O_4 nanoparticles have been prepared by solid-state, sol-gel, co-precipitation, hydrothermal, citrate-gel combustion and microwave-assisted methods. Several of these methods require prolonged processing, repeated grinding, high calcination temperatures or considerable energy input. Conventional

combustion methods may further involve synthetic organic fuels that generate undesirable gaseous products [10,11]. Plant-mediated synthesis offers a more sustainable alternative, as phytochemicals such as polyphenols, carbohydrates, flavonoids, amino acids and organic acids can assist precursor complexation and act as reducing or combustion agents. Coupling this approach with microwave irradiation enables rapid volumetric heating and efficient energy transfer, promoting fast nucleation and crystallization with reduced post-synthesis thermal treatment [12,13].

Despite growing interest in green synthesis of spinel ferrites, reports on *Tinospora cordifolia*-mediated microwave assisted preparation of MgFe_2O_4 remain limited. Previous studies have reported microwave-assisted MgFe_2O_4 synthesis using citrus lemon extract, yielding an average particle size of 20 ± 2 nm without further high-temperature calcination [14]. MgFe_2O_4 prepared by co-precipitation, sol-gel and auto-combustion routes showed Scherrer crystallite sizes of 22.06, 50.07 and 33.35 nm, respectively [9]. Green lemon juice-assisted MgFe_2O_4 has been reported with a crystallite size of about 32.9 nm and frequency-dependent dielectric dispersion [15], while hydrothermal MgFe_2O_4 exhibited a crystallite size of 19.5 nm with grain and grain-boundary contributions to the dielectric response [16]. Plant-mediated synthesis using *Cajanus cajan* leaf extract has also produced MgFe_2O_4 nanoparticles of approximately 12–20 nm size [17]. The present study combines *T. cordifolia*-mediated microwave combustion with Debye–Scherrer, Williamson–Hall (W-H) and size-strain plot (SSP) analyses, together with temperature-dependent dielectric, conductivity, electric modulus and Arrhenius relaxation studies. The focus is on correlating this rapid synthesis route with the electrical relaxation behaviour of MgFe_2O_4 rather than claiming novelty for plant-mediated microwave synthesis itself. Since no direct energy-consumption or life-cycle assessment was performed, claims regarding energy efficiency and environmental sustainability are kept qualitative.

In this work, phase-pure MgFe_2O_4 nanoparticles were synthesized through a rapid microwave-assisted combustion route using *T. cordifolia* extract as a natural phytochemical fuel and complexing medium. The product was characterized by X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS). Crystallite size and lattice strain were evaluated using the Debye–Scherrer, W-H and SSP methods. The dielectric constant (ϵ'), dielectric loss (ϵ''), loss tangent ($\tan \delta$), AC conductivity (σ_{ac}) and complex electric modulus (M' and M'') were examined over 1 Hz–100 kHz from room temperature to 400 °C. Electrical relaxation and activation energy were further evaluated using imaginary electric modulus and Arrhenius analyses to establish relationships between the structural characteristics and temperature- and frequency-dependent electrical response of MgFe_2O_4 .

EXPERIMENTAL

All the chemicals and solvents were used as received without further purification. Analytical reagent grade magnesium nitrate hexahydrate [$\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$] and ferric nitrate

nonahydrate [$\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$] were used as the magnesium and iron precursors for the synthesis of MgFe_2O_4 nanoparticles. Deionized water was used throughout the experimental work.

Preparation of *T. cordifolia* extract: Fresh *T. cordifolia* stems were thoroughly washed with tap water followed by deionized water to remove surface impurities. The cleaned stems were dried at room temperature for 1 week and ground into a fine powder. An aqueous extract was prepared by dispersing 10 g of the powdered stem in 100 mL of deionized water (1:10, w/v). The mixture was heated at 60 °C under continuous magnetic stirring (600 rpm) for 40 min. After cooling to room temperature, the suspension was filtered through Whatman No. 1 filter paper to obtain a clear extract. The filtrate was stored at 4 °C until further use. The extraction procedure was adapted with minor modifications from previously reported green synthesis methods for ferrite nanoparticles [12,18].

Microwave-assisted green synthesis of MgFe_2O_4 nanoparticles: Stoichiometric amounts of $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ corresponding to the MgFe_2O_4 composition were dissolved in the freshly prepared *T. cordifolia* extract under continuous magnetic stirring to obtain a homogeneous precursor solution. The solution was heated at 60 °C until a viscous gel was formed. The gel was transferred to a borosilicate glass beaker and irradiated in a domestic microwave oven operating at 600 W for approximately 5 min, initiating a rapid self-sustained combustion reaction. The combustion process yielded a fluffy porous brown powder, which was gently ground using an agate mortar to obtain fine MgFe_2O_4 nanoparticles. No post-combustion calcination of the synthesized powder was performed.

Characterization: The crystal structure and phase purity of the synthesized MgFe_2O_4 nanoparticles were examined using a Rigaku Ultima IV XRD (Rigaku Corporation, Japan) with $\text{CuK}\alpha$ radiation ($\lambda = 1.5406$ Å). Diffraction patterns were recorded over the 2θ range of 10° – 90° with a step size of 0.02° . The average crystallite size was estimated using the Debye–Scherrer equation, while the W-H and SSP methods were employed to evaluate the crystallite size and lattice strain. The lattice parameter, unit-cell volume, dislocation density, X-ray density, bulk density and apparent porosity were calculated using standard crystallographic relations. FTIR spectroscopy was employed to identify the characteristic metal-oxygen vibrations of the spinel structure. The surface morphology was investigated by SEM, whereas the elemental composition was analyzed using EDS.

Pellet preparation and electrical measurements: The synthesized MgFe_2O_4 nanopowder was compacted into cylindrical pellets of 13 mm diameter and 1.30 mm thickness using a stainless-steel die under a uniaxial load of approximately 5 tons. The pellets were sintered in air at 600 °C for 4 h and allowed to cool naturally to room temperature inside the furnace. Prior to electrical measurements, both pellet surfaces were coated with conductive silver paste to ensure good electrical contact.

The dielectric properties were measured using a Hioki LCR meter equipped with a Hioki 9140-10 test fixture over the frequency range of 1 Hz–100 kHz and the temperature range from room temperature to 400 °C at intervals of 5 °C.

Representative results are presented for 100 Hz, 1 kHz, 10 kHz and 100 kHz. The ϵ' , ϵ'' , $\tan \delta$, σ_{ac} , M' and M'' were calculated using the following relations:

$$\epsilon' = \frac{C_p t}{\epsilon_0 A}$$

$$\epsilon'' = \epsilon' \tan \delta$$

$$\sigma_{ac} = 2\pi f \epsilon_0 \epsilon''$$

$$M' = \frac{\epsilon'}{\epsilon'^2 + \epsilon''^2}$$

$$M'' = \frac{\epsilon''}{\epsilon'^2 + \epsilon''^2}$$

where C_p is the measured capacitance, t is the pellet thickness, A is the electrode area, ϵ_0 is the permittivity of free space and f is the applied frequency.

The relaxation time corresponding to the selected M'' relaxation features was determined using:

$$\tau = \frac{1}{2\pi f_{\max}}$$

and the activation energy was obtained from the Arrhenius equation:

$$\tau = \tau_0 \exp \frac{E_a}{k_B T}$$

where τ_0 is the pre-exponential factor, E_a is the activation energy and k_B is the Boltzmann constant. The bulk density of the pellets was calculated from the measured pellet mass and dimensions, while the apparent porosity was estimated using the theoretical X-ray density obtained from XRD analysis.

RESULTS AND DISCUSSION

Phase formation and crystal structure analysis: The phase formation and crystal structure of the microwave assisted *T. cordifolia*-derived MgFe_2O_4 nanoparticles were investigated by XRD. The diffraction pattern shown in Fig. 1 exhibits well-defined reflections at $2\theta = 30.26^\circ$, 35.65° , 43.28° , 57.22° and 62.77° , which are indexed to the (220), (311), (400), (511) and (440) crystallographic planes, respectively. These reflections agreed well with the standard diffraction pattern of cubic spinel MgFe_2O_4 (ICDD Pdf No. 88-1935) and confirm crystallization in the $Fd\bar{3}m$ space group. The observed diffraction pattern is consistent with previous reports on MgFe_2O_4 nanoparticles synthesized by combustion, sol-gel and green synthesis methods [8,9,11,13].

No additional diffraction peaks corresponding to crystalline MgO , $\alpha\text{-Fe}_2\text{O}_3$, Fe_3O_4 or other impurity phases were detected within the instrumental resolution, demonstrating the successful formation of phase-pure MgFe_2O_4 through the microwave-assisted green combustion process. The absence of secondary phases indicates that the heat generated during microwave-assisted combustion was sufficient to promote direct crystallization of the spinel phase without requiring post combustion calcination of the synthesized powder. The phytochemical constituents present in the *T. cordifolia* extract

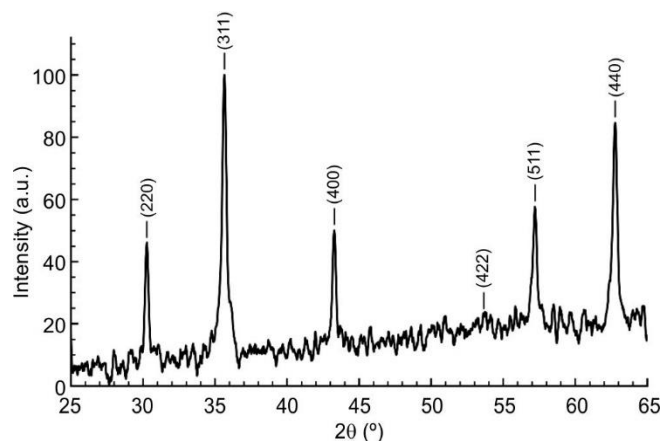


Fig. 1. XRD pattern of the as-synthesized MgFe_2O_4 nanoparticles

likely facilitated homogeneous precursor complexation and provided an efficient combustion environment, promoting uniform nucleation and rapid phase formation.

The diffraction peaks are relatively sharp with moderate broadening reflects the good crystallinity and nanocrystalline character of the synthesized MgFe_2O_4 . In nanostructured ferrites, peak broadening arises from both finite crystallite size and lattice microstrain. Since, the Debye–Scherrer method does not distinguish between these contributions, the diffraction data were further examined using the Williamson–Hall (W-H), Uniform Deformation Model (UDM) and size–strain plot (SSP) methods.

Crystallite size and microstructural analysis: The Debye–Scherrer method assumes that peak broadening arises solely from the finite crystallite size, whereas the W-H and SSP methods consider the combined contributions of crystallite size and lattice strain [8,9]. The crystallite size estimated using the Debye–Scherrer equation was 15.10 nm, confirming the nanocrystalline nature of the synthesized MgFe_2O_4 . Since this method neglects strain-induced broadening, it generally provides the apparent coherent domain size derived from the diffraction peak widths.

To separate the size and strain contributions, the W-H (UDM) was employed according to:

$$\beta \cos \theta = \frac{K\lambda}{D} + 4\epsilon \sin \theta$$

where β is the full width at half maximum (FWHM), K is the Scherrer constant, λ is the X-ray wavelength, D is the crystallite size and ϵ is the lattice strain. The corresponding W-H plot is shown in Fig. 2a. A linear relationship was obtained from the $\beta \cos \theta$ versus $4\sin \theta$ plot. The corresponding crystallite size and lattice strain were calculated as 18.36 nm and 0.816×10^{-3} , respectively. The relatively small strain value reflects the limited degree of lattice distortion in the microwave combusted MgFe_2O_4 .

The crystallite size was further estimated using the SSP method, which accounts for both size- and strain-induced broadening while exhibiting higher statistical weight to reflections with higher diffraction intensity. As shown in Fig. 2b, the SSP analysis yielded an average crystallite size of 18.26 nm, closely matching the value obtained from the W-H method. The small difference between the two values points to a mod-

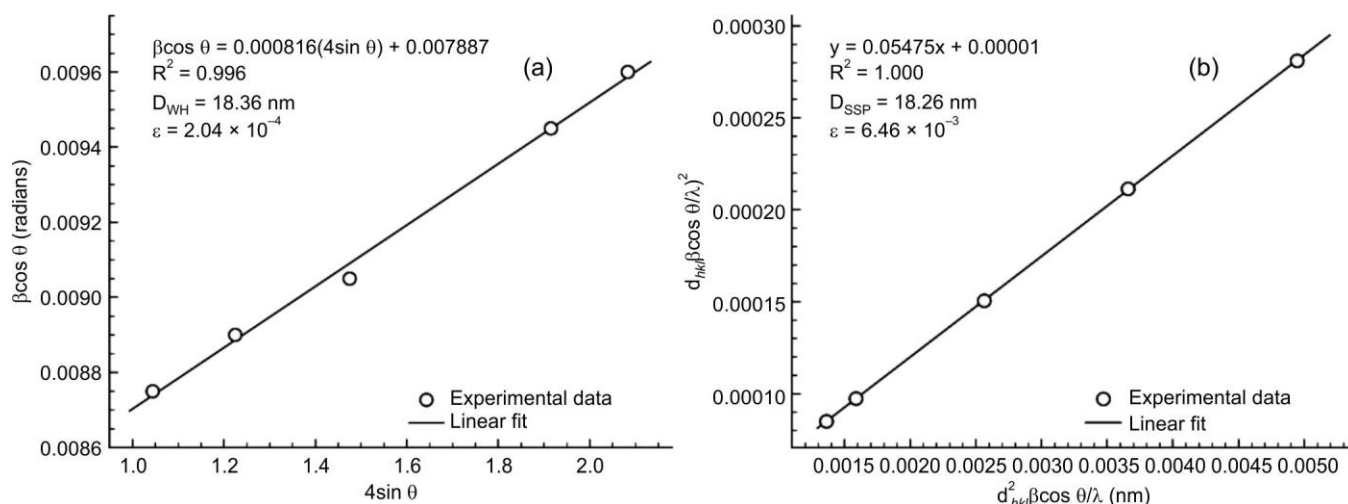


Fig. 2. Plots of (a) Williamson-Hall (UDM) and (b) size-strain (SSP) analyses of synthesized MgFe_2O_4 nanoparticles for estimating crystallite size and lattice strain

erate contribution of strain to the observed peak broadening and supports the consistency of the line-broadening analysis..

A comparison of the crystallite sizes obtained using the three methods is shown in Fig. 3. The Debye-Scherrer, W-H and SSP methods yielded average crystallite sizes of 15.10, 18.36 and 18.26 nm, respectively. The comparatively smaller value obtained from the Debye-Scherrer method is expected since it does not account for lattice strain, whereas the W-H and SSP methods evaluate both crystallite size and strain simultaneously. The close agreement between the latter two methods demonstrates the consistency of the structural analysis and confirms that the synthesized MgFe_2O_4 consists of well-crystallized nanometer-sized domains with relatively low lattice distortion. The differences among these values are attributed to the different assumptions and treatments of size- and strain-induced peak broadening in the respective models; therefore, these values should be regarded as model-dependent estimates of XRD crystallite size rather than exact particle sizes.

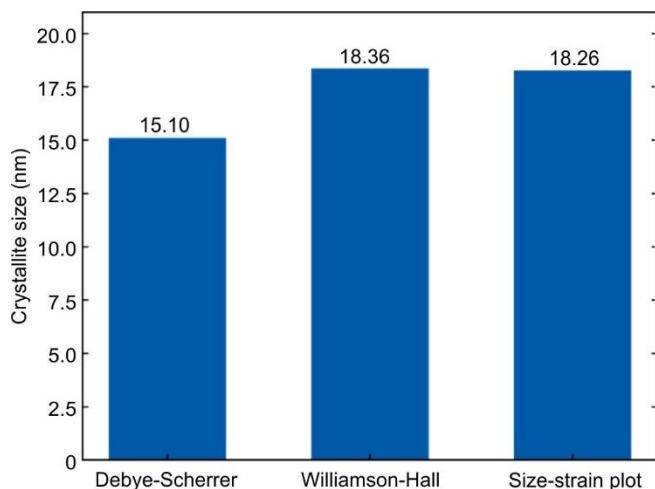


Fig. 3. Comparison of the crystallite sizes of MgFe_2O_4 nanoparticles estimated by Debye-Scherrer, W-H (UDM) and SSP methods

The calculated lattice parameter varied slightly from 8.345 to 8.367 Å, with unit-cell volumes of 581.08–585.68

Å³. The corresponding theoretical X-ray density was 4.538–4.574 g cm⁻³, while the experimental bulk density of the sintered pellet was approximately 4.057 g cm⁻³. The resulting apparent porosity was 10.6–11.3%. The lower experimental density can be related to residual pores formed during combustion and retained following sintering. This level of porosity is typical of combustion-derived ferrites and may contribute to the interfacial polarization responsible for part of the dielectric response [6,7].

FTIR analysis: The FTIR spectrum recorded in the range of 4000–400 cm⁻¹ is shown in Fig. 4. A broad absorption band centered at ~3404 cm⁻¹ is attributed to the O–H stretching vibration of surface-adsorbed water molecules and hydroxyl groups. The weak band observed near 1620 cm⁻¹ corresponds to the H–O–H bending vibration of adsorbed moisture. Another weak absorption around 1420 cm⁻¹ may be associated with surface carbonate species or residual organic functional groups originating from atmospheric adsorption during sample handling. Similar absorption bands have been reported for combustion- and plant-mediated synthesized spinel ferrites [19–21].

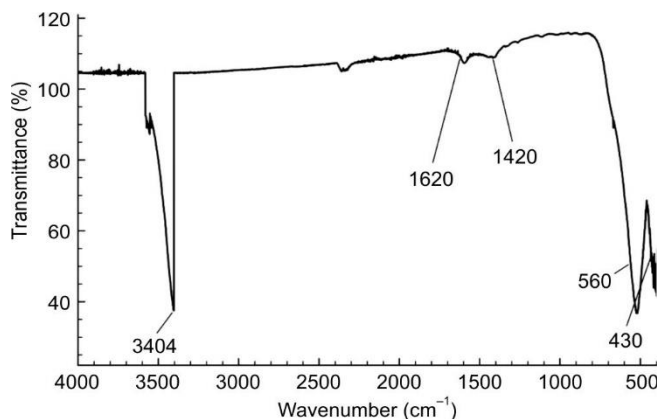


Fig. 4. FTIR spectrum of the as-synthesized MgFe_2O_4 nanoparticles

The most prominent features of the spectrum are the two characteristic absorption bands observed at approximately

560 cm^{-1} and 430 cm^{-1} , which are the characteristic vibrational modes of the spinel ferrite structure. The higher-frequency band at 560 cm^{-1} is assigned to the intrinsic stretching vibration of the metal-oxygen bond at the tetrahedral (A) site, whereas the lower-frequency band at 430 cm^{-1} corresponds to the stretching vibration of metal-oxygen bonds at the octahedral (B) site. The difference in the band positions arises from the distinct bond lengths and coordination environments of the tetrahedral and octahedral cation sites within the cubic spinel lattice.

The observation of these two characteristic vibrational bands provides spectroscopic evidence for the successful formation of the MgFe_2O_4 spinel structure. The FTIR findings are consistent with the XRD results and support the formation of crystalline spinel MgFe_2O_4 through the microwave-assisted *T. cordifolia*-mediated green combustion route. Similar FTIR characteristics have been reported for MgFe_2O_4 nanoparticles prepared by combustion, sol-gel and other green synthesis methods [8,9,13].

Surface morphology and elemental analysis: The surface morphology of the synthesized MgFe_2O_4 powder was examined by SEM as shown in Fig. 5a. The micrograph displays an irregular, porous and highly agglomerated structure composed of interconnected clusters of finer particles. At the $10\text{ }\mu\text{m}$ scale, the image mainly reflects the size and distribution of these agglomerates rather than individual nanoparticles; hence, a quantitative particle-size value was not derived from this micrograph.

The pronounced agglomeration can be associated with the high surface energy of fine ferrite particles and attractive interactions between magnetic domains. The porous structure may arise from rapid gas evolution during the self-propagating combustion process. Comparable porous and agglomerated morphologies have been reported for MgFe_2O_4 and related spinel ferrites prepared by combustion and plant mediated routes [8,11,13]. The pores and interfaces between agglomerates may influence the heterogeneous electrical response of the compacted pellets, although their individual contribution cannot be established from the SEM image alone.

The elemental composition was examined by EDS with the corresponding spectrum as shown in Fig. 5b. The spectrum contains the characteristic signals of Mg, Fe and O. The quantitative analysis gave 20.61 wt.% O, 9.35 wt.% Mg and 70.04 wt.% Fe. On normalization, these values correspond to approximately 44.01 at.% O, 13.14 at.% Mg and 42.85 at.% Fe, compared with 57.14 at.% O, 14.29 at.% Mg and 28.57 at.% Fe for ideal MgFe_2O_4 . The deviation, particularly in the Fe and O contents, means that the EDS data should be treated as localized, semi-quantitative compositional information rather than a direct representation of bulk stoichiometry. Sampling volume, surface roughness, matrix effects and the relatively low sensitivity of EDS to oxygen can contribute to such differences. The EDS results therefore confirm the presence of Mg, Fe and O and the absence of detectable foreign metallic elements, while phase purity is primarily established from the XRD results.

Dielectric properties

Dielectric constant (ϵ'): The temperature dependence of the dielectric constant (ϵ') of the sintered MgFe_2O_4 pellets is shown in Fig. 6a. The observed frequency dispersion can be described by the Maxwell–Wagner interfacial polarization mechanism and Koops’ two-layer model, in which relatively conducting grains are separated by more resistive grain boundaries. At low frequencies, charge carriers can accumulate at the grain boundaries, producing space-charge polarization and higher ϵ' values. As the frequency increases, the electric field reverses more rapidly, giving charge carriers less time to accumulate at grain boundaries and leading to a decrease in ϵ' .

The increase in ϵ' with temperature can be associated with thermally activated electron hopping between Fe^{2+} and Fe^{3+} ions at neighbouring octahedral (B) sites. Higher temperatures enhance the mobility of localized charge carriers, strengthening dipolar and interfacial polarization. Similar dielectric behaviour has been reported for MgFe_2O_4 and related spinel ferrites prepared through combustion and green synthesis routes [6,13,22]. No abrupt discontinuity was obser-

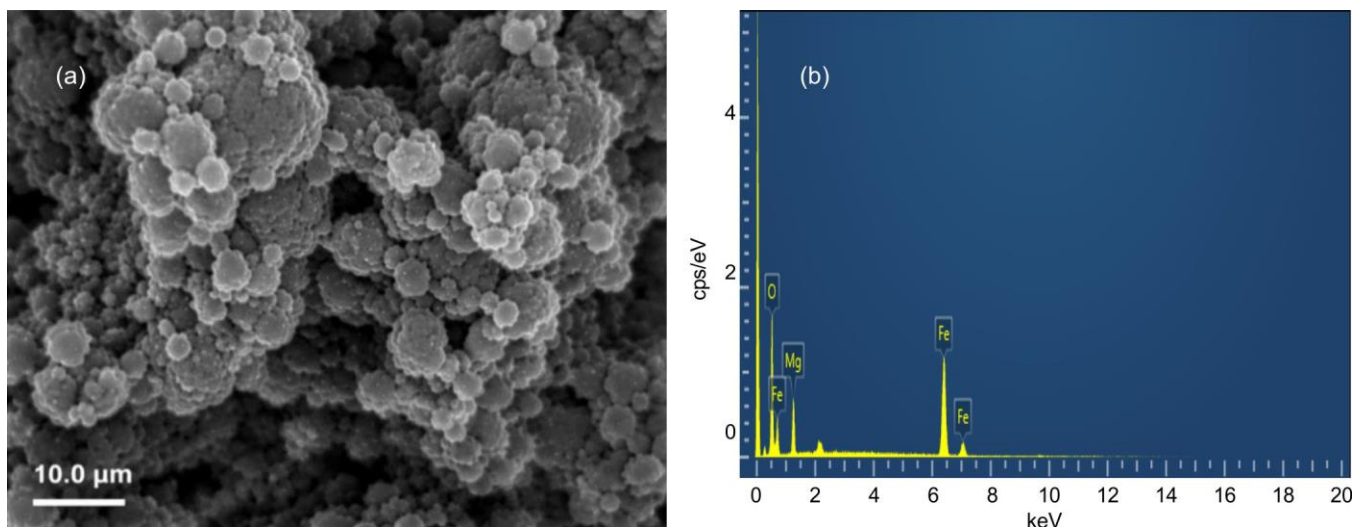


Fig. 5. SEM micrograph of as-synthesized MgFe_2O_4 powder prepared by the microwave-assisted *T. cordifolia*-mediated green combustion route and (b) corresponding EDS spectrum with the elemental composition of the sample

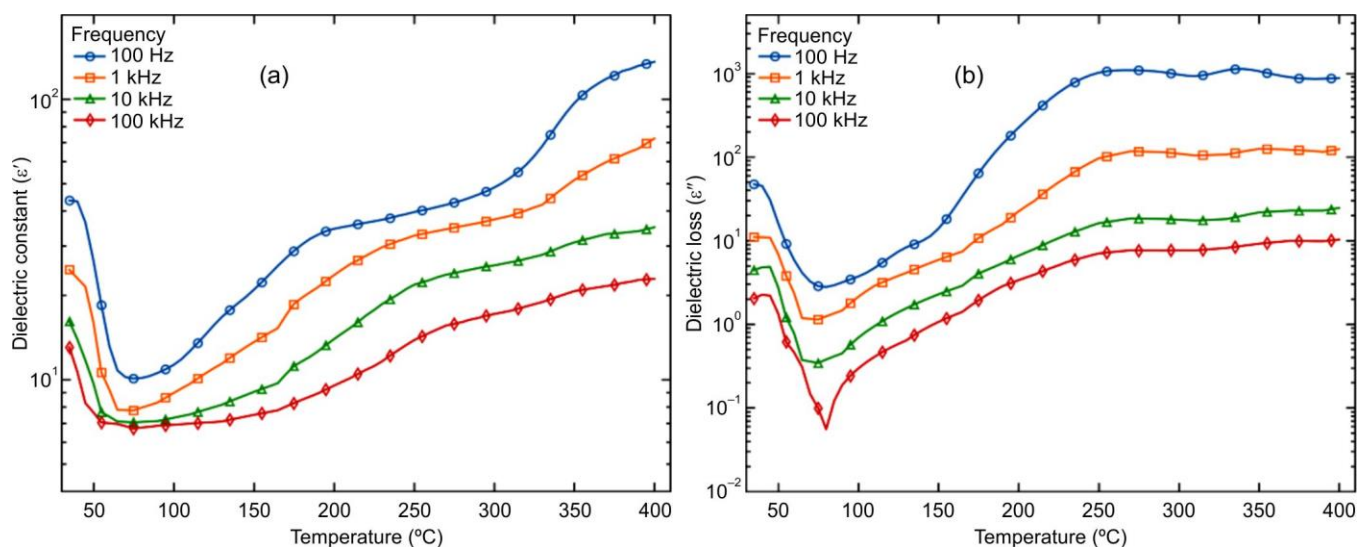


Fig. 6. (a) Temperature dependence of ϵ' (b) Temperature dependence of ϵ'' of the sintered MgFe₂O₄ nanoparticles prepared from the synthesized powder at selected frequencies (100 Hz, 1 kHz, 10 kHz and 100 kHz)

ved in the dielectric response over the investigated temperature range. The gradual variation is consistent with a continuous thermally activated polarization process under the present conditions. The dielectric behaviour of the synthesized MgFe₂O₄ can therefore be described by the combined contribution of grain-boundary polarization and localized electron hopping.

Dielectric loss (ϵ''): The variation of ϵ'' with temperature at representative frequencies is shown in Fig. 6b. Similar to the dielectric constant, the dielectric loss exhibits a pronounced dependence on both temperature and frequency. At lower temperatures, ϵ'' remains relatively low since the mobility of localized charge carriers is limited, which result in the reduced dielectric energy dissipation. With increasing temperature, ϵ'' rises across the measured frequency range, with a more pronounced increase at lower frequencies. This behaviour can be associated with thermally activated hopping between Fe²⁺/Fe³⁺ centres in the spinel lattice, which enhances charge-carrier mobility and polarization losses. The frequency dependence arises from the ability of charge carriers to respond to the alternating electric field. At low frequencies, the charge carriers have more time to move and accumulate around the grain boundaries, which leads to higher dielectric losses. At higher frequencies, rapid field reversal restricts carrier movement and reduces energy dissipation, which lead to the lower ϵ'' values. Similar dielectric behaviour has been reported for MgFe₂O₄ and related spinel ferrites prepared by the combustion and green synthesis routes [6,7,20,22].

The dielectric loss gradually increases over the investigated temperature range, which can be attributed to thermally activated hopping of charge carriers rather than any abrupt change in the material. The broad variation of ϵ'' with temperature may also arise from relaxation processes associated with grains, grain boundaries and localized defect states.

Loss tangent ($\tan \delta$): The temperature dependence of $\tan \delta$ for the synthesized MgFe₂O₄ nanoparticles is shown in Fig. 7. The loss tangent varies with both temperature and frequency, which reflect the energy dissipated during polarization

under an alternating electric field. At lower temperatures, $\tan \delta$ remains relatively low due to the limited mobility of charge carriers. As the temperature increases, broad relaxation features appear, particularly at lower frequencies, as thermally activated carriers become more mobile and contribute more strongly to dielectric loss.

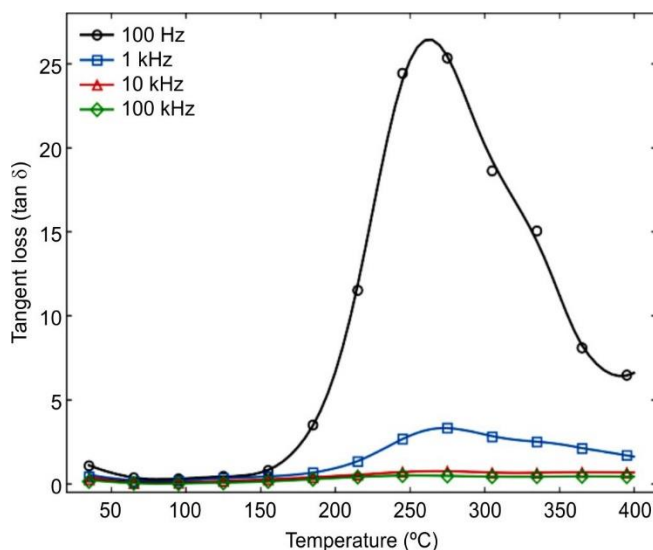


Fig. 7. Temperature dependence of $\tan \delta$ for synthesized MgFe₂O₄ nanoparticles at selected frequencies (100 Hz, 1 kHz, 10 kHz and 100 kHz)

The broad relaxation features indicate that the material does not have a single relaxation time. Instead, the response involves a range of relaxation times associated with grains, grain boundaries and localized defect states [6,23]. The loss tangent decreases gradually with increasing frequency. The relatively slow field variation at lower frequencies facilitates carrier movement toward grain boundaries, where the charge accumulation contributes to higher dielectric loss. At higher frequencies, the electric field changes too rapidly for the hopping carriers to respond fully, which reduces dielectric loss and

gives lower $\tan \delta$ values. This behaviour agrees with the thermally activated hopping process proposed for the dielectric response of MgFe_2O_4 nanoparticles. The $\tan \delta$ results provide useful information on the relaxation behaviour, however, the overlapping features make it difficult to obtain reliable quantitative relaxation parameters from these plots alone. The complex electric modulus formalism was therefore used for a more detailed analysis.

Electrical transport and relaxation behaviour

AC conductivity (σ_{ac}): The temperature-dependent σ_{ac} of the sintered pellets prepared from the synthesized powder is shown in Fig. 8 as $\log_{10}\sigma_{ac}$ versus $1000/T$.

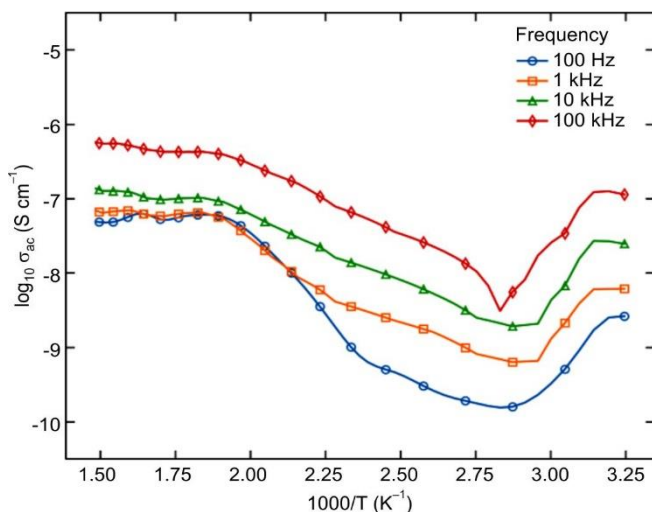


Fig. 8. Variation of $\log_{10}\sigma_{ac}$ with reciprocal temperature ($1000/T$) for synthesized MgFe_2O_4 nanoparticles at selected frequencies (100 Hz, 1 kHz, 10 kHz and 100 kHz)

At the lower-temperature end, the σ_{ac} varies only weakly and shows a small non-monotonic region. With further heating, σ_{ac} generally increases and the rise becomes more pronounced

above approximately 200°C . This behaviour confirms that electrical transport in the MgFe_2O_4 pellet is predominantly thermally activated. The absence of an abrupt conductivity discontinuity indicates that the observed change arises from progressive activation of localized charge carriers rather than from a sharp electrical transition. The increase in conductivity with frequency shows that localized charge carriers can undergo short-range hopping more effectively under a rapidly alternating electric field. At lower frequencies, the electrical response is influenced more strongly by resistive grain boundaries, whereas at higher frequencies the contribution of the comparatively conducting grain interiors becomes increasingly significant. Such frequency dispersion is characteristic of electrically heterogeneous polycrystalline ferrites containing grains and grain-boundary regions with different resistive characteristics [6,7]. The thermally activated increase in conductivity is consistent with localized electron hopping involving $\text{Fe}^{2+}/\text{Fe}^{3+}$ centres at neighbouring octahedral (B) sites in the spinel lattice. Increasing temperature supplies the energy required for localized electrons to overcome potential barriers and hop between mixed-valence iron sites, thereby enhancing charge transport. Similar hopping-controlled conductivity has been reported for MgFe_2O_4 and related spinel ferrites [8,13,24]. The conductivity results are consistent with the dielectric response, in which enhanced the charge-carrier mobility at elevated temperatures produces increased polarization and dielectric loss.

Electric modulus analysis: The complex electric modulus formalism was employed to investigate the electrical relaxation behaviour of the synthesized MgFe_2O_4 nanoparticles. In contrast to dielectric permittivity, the electric modulus representation suppresses the influence of electrode polarization and emphasizes the bulk electrical response of the material, thereby providing a more reliable description of relaxation processes occurring within the ferrite grains and grain boundaries [6]. The variation of the M' with reciprocal temperature is shown in Fig. 9a. The relatively low M' values at lower

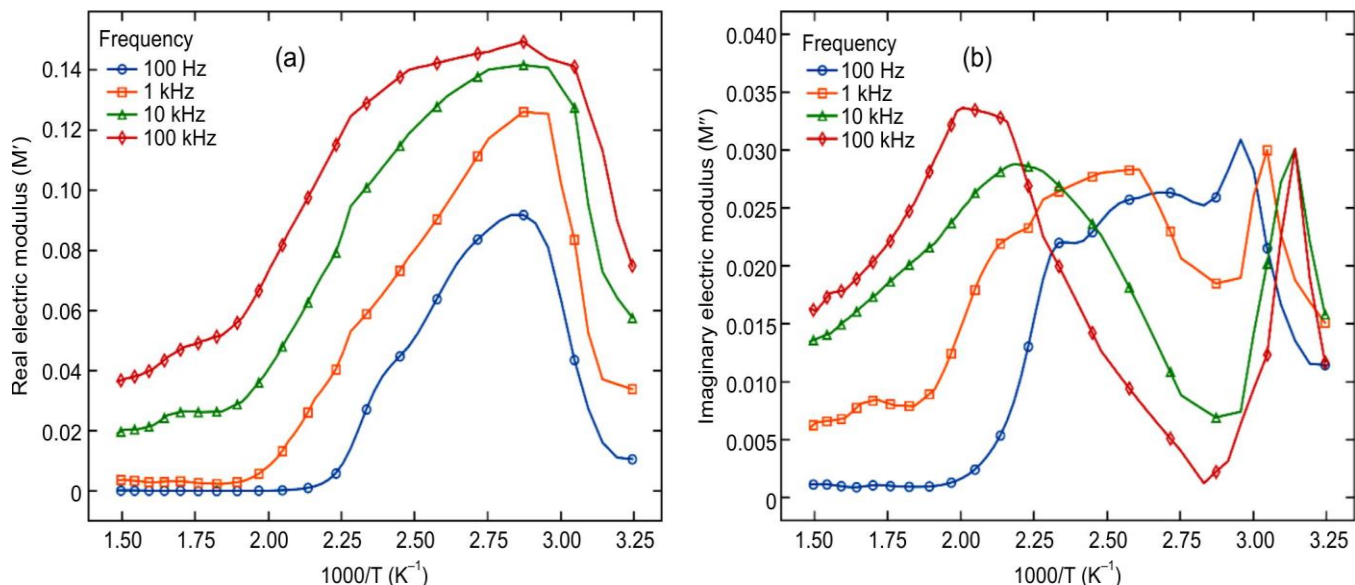


Fig. 9. (a) Temperature dependence of the real electric modulus (M') and (b) imaginary electric modulus (M'') of the synthesized MgFe_2O_4 nanoparticles at selected frequencies (100 Hz, 1 kHz, 10 kHz and 100 kHz)

temperatures are associated with the limited charge-carrier mobility. As the temperature rises, M' increases gradually and tends to become nearly constant at higher temperatures. This behaviour reflects the reduced contribution of space-charge polarization and the higher influence of bulk relaxation. M' also increases with frequency, as the rapid variation of the applied field limits interfacial polarization and makes the bulk response more evident.

The temperature dependence of the M'' is shown in Fig. 9b. The relaxation response remains broad over the investigated temperature range and is associated with localized charge carrier movement. Rather than a single relaxation time, the material exhibits a range of relaxation times, which is characteristic of heterogeneous polycrystalline ferrites. The electrical response may arise from the combined contributions of grains, grain boundaries and localized defect states [6,23]. The modulus representation confirms that the observed electrical relaxation originates predominantly from the bulk material rather than from electrode polarization. The relaxation process is consistent with thermally activated electron hopping between neighbouring $\text{Fe}^{2+}/\text{Fe}^{3+}$ centres at octahedral (B) sites of the spinel lattice. Since the M'' spectra exhibit overlapping relaxation features, the higher-temperature relaxation feature was selected consistently at each frequency for quantitative analysis.

Arrhenius analysis: To quantify the electrical relaxation process, relaxation times were obtained from the selected high-temperature M'' relaxation features at different frequencies. Since the M'' spectra showed overlapping features, the same high-temperature feature was consistently selected for all frequencies to maintain a common relaxation process for the Arrhenius analysis. The Arrhenius plot in Fig. 10 shows an approximately linear variation of $\ln \tau$ with $1000/T_p$. The linear fit gave an activation energy of 0.758 eV, a pre-exponential factor of 4.50×10^{-14} s and $R^2 = 0.933$.

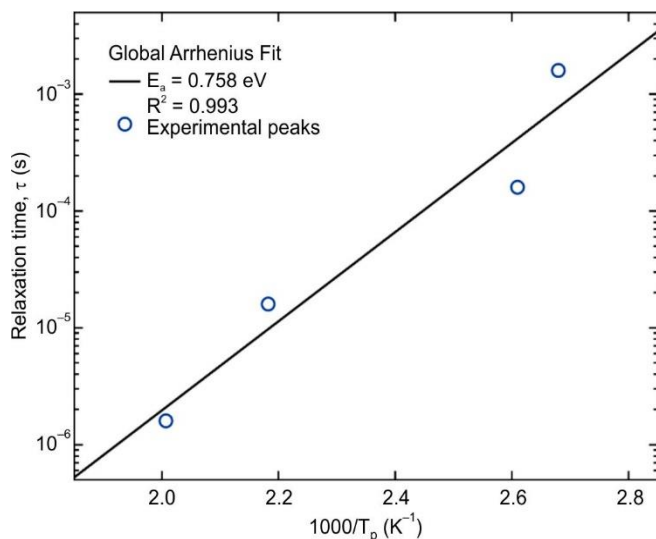


Fig. 10. Arrhenius plot of relaxation time for sintered MgFe_2O_4 pellets prepared from the synthesized powder

The activation energy is comparable to reported values for MgFe_2O_4 and related spinel ferrites, where relaxation has been linked to localized electron hopping between neigh-

bouring $\text{Fe}^{2+}/\text{Fe}^{3+}$ ions at octahedral (B) sites [6,8,24]. The reasonable linearity of the Arrhenius plot supports a thermally activated relaxation process over the investigated temperature range. This result agrees with the dielectric, AC conductivity and electric modulus measurements, which point to localized charge-carrier hopping as the main contributor to the electrical relaxation of MgFe_2O_4 nanoparticles.

Electrical relaxation mechanism: The electrical transport behaviour of the synthesized MgFe_2O_4 nanoparticles can be interpreted from the combined dielectric, AC conductivity and electric modulus results. The increase in dielectric constant, dielectric loss and AC conductivity with temperature points to a thermally activated charge-transport process. At lower temperatures, restricted carrier mobility results in weaker polarization and lower conductivity. With increasing temperature, the localized electron hopping involving $\text{Fe}^{2+}/\text{Fe}^{3+}$ centres at octahedral (B) sites becomes more favourable, which enhances polarization and electrical conduction. Similar hopping related transport has been reported for MgFe_2O_4 and other spinel ferrites [6,8,13].

The porous and agglomerated microstructure observed by SEM provides conducting regions separated by relatively resistive grain boundaries. The accumulation of charge at grain boundaries contributes to the dielectric response at lower frequencies, which is consistent with the Maxwell–Wagner and Koops models [6,23]. At higher frequencies, rapid field reversal limits carrier movement and weakens interfacial polarization. The electric modulus results provide further evidence that the observed relaxation is mainly associated with the bulk response rather than electrode polarization. The broad M'' features point to a distribution of relaxation times, characteristic of non-Debye relaxation. Such behaviour is commonly associated with polycrystalline ferrites, where grains, grain boundaries and localized defect states can contribute to the electrical response [23,24].

Conclusion

Phase-pure MgFe_2O_4 nanoparticles were synthesized by microwave-assisted green combustion using *T. cordifolia* extract as a natural phytochemical fuel, without post-combustion calcination. XRD confirmed a single-phase cubic spinel structure ($Fd\bar{3}m$) with crystallite sizes of 15.10, 18.36 and 18.26 nm from the Debye–Scherrer, W-H and SSP methods, respectively, along with low lattice strain. FTIR confirmed the characteristic metal–oxygen vibrations of the spinel lattice, while SEM/EDS revealed a porous, agglomerated morphology with Mg, Fe and O as the major elements. The dielectric and AC conductivity results showed clear temperature- and frequency-dependent behaviour associated with thermally activated charge transport. Arrhenius analysis of the selected high-temperature M'' relaxation features gave an activation energy of 0.758 eV, a pre-exponential factor of 4.50×10^{-14} s and $R^2 = 0.933$. The dielectric, conductivity and modulus results are consistent with localized charge hopping and non-Debye relaxation in the nanocrystalline ferrite. The observed structural and electrical characteristics support the possible use of the synthesized MgFe_2O_4 in the ferrite-based dielectric and electronic applications within the investigated conditions.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

DECLARATION OF AI-ASSISTED TECHNOLOGIES

During the preparation of this manuscript, the authors used an AI-assisted tool(s) to improve the language and no data, results or scientific conclusions were generated by AI. All analyses, interpretations and conclusions are the authors' own. The authors reviewed and edited the content and take full responsibility for the published work.

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