

Synthesis and Characterization of MgTiO_3 Nanoparticles by Sol-Gel Method

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ABSTRACT

This study is on one-pot synthesis of magnesium titanate (MgTiO_3) nanoparticles via sol-gel route. The morphology, particles size, crystal structure, phase identification and functional groups of the produced nanomaterials were studied using high resolution scanning electron microscopy (HRSEM), X-ray diffraction (XRD) and Fourier transform infrared (FTIR) techniques. HRSEM analysis revealed characteristic homogeneous spherical shape for TiO_2 nanoparticles, while MgO and MgTiO_3 exhibited aggregated morphologies with irregular shapes with particle size 13.42 nm for TiO_2 , 14.65 nm for MgO and 10.47 nm for MgTiO_3 . XRD analysis established anatase phase of TiO_2 , periclase phase for MgO , and mixture of the two phases for MgTiO_3 nanoparticles. Crystallite sizes calculated using Debye-Scherrer equation were 19.10 nm, 39.30 nm, and 17.51 nm for TiO_2 , MgO and MgTiO_3 respectively. FTIR spectral profiles of TiO_2 , MgO and MgTiO_3 show peaks for surface hydroxyl groups, molecular water, and metal-oxygen stretching. TiO_2 exhibits bands at 3300 cm^{-1} (O-H) and 670 cm^{-1} (Ti-O-Ti), MgO exhibited bands at 3400 cm^{-1} (O-H) and 603 cm^{-1} (Mg-O), while MgTiO_3 exhibited band at 3400 cm^{-1} (O-H) and at 614 cm^{-1} and 456 cm^{-1} (Ti-O-Mg-O). The resultant MgTiO_3 nanocomposite exhibited the desired characteristics, proving the sol-gel process to be a simple method for material preparation with promising applications in different fields.

Keywords: MgTiO_3 , Sol-Gel, HRSEM, XRD, FTIR

INTRODUCTION

Nanotechnology has been area of extensive research for the past four decades owing to the application of materials unique physical, chemical, and biological properties in different fields [1, 2]. Nanoparticles are central to this area, and they are very tiny particles with sizes ranging from

1 to 100 nanometers [3]. These nanoparticles are fundamental building blocks of nanotechnology and have been found to possess wide-ranging applications in varied areas such as sensors, electronics, water treatment, cosmetics, biomedical implants, pharmaceuticals, environmental remediation, catalysis, and material science [4]. The unique characteristics of nanomaterials such as their smaller size, crystallinity, shape, high surface area-to-volume ratio, quantum confinement effects, and modified electronic structures greatly impact their catalytic, magnetic, electrical, and optical properties [5,6].

Titanium dioxide (TiO₂) nanoparticles have attracted much attention owing to its high chemical stability and multi-functional nature, making it an important ingredient in many commercial products. The anti-corrosive, photocatalytic, and optoelectronic characteristics of TiO₂ nanoparticle are accountable for its extensive application in plastics, paints, varnishes, paper, inks, personal care products, sunscreens, photovoltaic cells, wastewater treatment, and bactericidal applications [7]. The medical use of TiO₂ nanoparticle extends to photodynamic cancer treatment, drug delivery systems, cell imaging, biosensors for biological assays, and gene editing, demonstrating further its potential in contemporary technology.

Similarly, magnesium oxide (MgO) nanoparticles have attracted much attention due to their outstanding thermal stability, surface reactivity, heat resistance, and chemical inertness [8]. As an inorganic compound with a wide band gap of about 7.8 eV, MgO nanoparticle plays a significant role in catalysis and antimicrobial functions, particularly against foodborne pathogens. Due to their strong ionic nature, fixed stoichiometry, unique crystal structure, and defects in the surface structure, MgO nanoparticles displays great adsorption properties and therefore, are highly effective in wastewater treatment and poisonous gas removal from the environment [9]. Furthermore, they are biocompatible and non-toxic and hence are fit for biomedical applications and environmental cleanup.

Current developments in nanotechnology have led to the synthesis of various ceramic nanomaterials called titanates such as PbTiO₃, SrTiO₃, BaTiO₃, FeTiO₃, CaTiO₃, and MgTiO₃ which are used in a wide range of technical applications such as dielectric, pyroelectric, ferroelectric, piezoelectric, and optoelectronic.

Titanates are compound containing the titanate ion, TiO₃²⁻ which has excellent photocatalytic properties such as chemical stability, thermal stability, high charge separation and increased efficiency of pollutant degradation. Magnesium titanate (MgTiO₃) is a member of

perovskite nanostructures where Ti⁴⁺ ions are substituted with Mg²⁺, introducing oxygen vacancies for charge neutrality. They are known to have unique properties such as increase surface area and quantum size effects as well as thermal and chemical stability, modified electronic and layered structure that facilitates greater charge separation [10, 11]. These properties make them effective for application in electronics, optics, photocatalysis, catalysis and in recent time in wastewater treatment.

Synthesis of nanomaterials employs diverse techniques, including chemical precipitation, thermal plasma, chemical vapour deposition, hydrothermal treatment, laser ablation, spray pyrolysis, ball-milling, sol-gel processes, and vacuum arc deposition. Of these, the sol-gel technique stands out with its low cost, ease of operation, and good control of nanoparticle size and shape [12]. Sol-gel approach entail steps such as hydrolysis, polymerization, evaporation, and condensation of metal salt precursors, leading to the creation of stable colloidal suspensions. One pot synthesis combines all reactant in a single reaction vessel to produce desired compound, reducing processing step and energy consumption.

In light of the importance of such nanomaterials, the current work seeks to synthesize TiO₂, MgO, and MgTiO₃ nanoparticles using one-pot sol-gel method and characterize the prepared nanomaterials using various analytical techniques

MATERIALS AND METHODS

Materials

Titanium (IV) isopropoxide (Ti{OCH(CH₃)₂}₄, 97%), magnesium nitrate hexahydrate (Mg(NO₃)₂.6H₂O, 99%) were purchased from Sigma Aldrich, Germany, sodium hydroxide (NaOH, 96%) and polyethylene glycol (PEG) were purchased from Lobie Chem. India. All chemicals used were of analytical grade, and all reagents were prepared in doubly distilled water.

Synthesis of TiO₂ Nanoparticles

TiO₂ nanoparticle was synthesized via sol gel method using Ti{OCH(CH₃)₂}₄ as precursor. In a typical synthesis, 16 cm³ of Ti{OCH(CH₃)₂}₄ was added to 100 cm³ of distilled water in a 250 cm³ beaker. Exactly 10 cm³ of 10 % (w/v) polyethylene glycol (PEG) was added and 0.5 mol/dm³ NaOH was added dropwise to attain pH of 12. The mixture was stirred at 250 rpm for 1 h with heating at 60 °C until a gel was formed which was left overnight for ageing. After the ageing process, it was washed to a pH of 7 with distilled water and then oven dried for 8 h at 70

°C. The powdered sample was calcined in a muffle furnace at a temperature of 450 °C for 3 h to obtain TiO₂ nanoparticles [13]

Synthesis of MgO Nanoparticles

MgO nanoparticle was synthesized via sol gel method using Mg(NO₃)₂.6H₂O as precursor. In a typical synthesis, 7.7 g of Mg(NO₃)₂.6H₂O was added to 100 cm³ of distilled water. Exactly 10 cm³ of 10 % (w/v) polyethylene glycol was added and 0.5 mol/dm³ NaOH was added dropwise to attain pH of 12. The mixture was stirred at 250 rpm for 1 h with heating at 60 °C until a gel was formed which was left overnight for ageing. After the ageing process, it was washed to a pH of 7 with distilled water and then oven dried for 8 h at 70 °C. The obtained powdered sample was calcined in a muffle furnace at a temperature of 450 °C for 3 h to obtain MgO nanoparticles [13].

One-Pot Synthesis of MgTiO₃ Nanoparticles

The one pot synthesis of MgTiO₃ nanoparticle was carried out using Mg(NO₃)₂.6H₂O and Ti{OCH(CH₃)₂}₄, as precursors via sol-gel process. In a typical procedure, 7.7 g of Mg(NO₃)₂.6H₂O was weighed and added to 100 cm³ of distilled water in a 250 cm³ beaker followed by adding 16 cm³ of Ti{OCH(CH₃)₂}₄. 10 cm³ of 10 % (w/v) polyethylene glycol (PEG) was added as a stabilizing agent and 0.5 mol/dm³ NaOH was added dropwise to adjust the pH to 12. The mixture was stirred at 250 rpm for 1 h with heating at 60 °C until a gel was formed which was left overnight for ageing. After the ageing process, it was washed to a pH of 7 with distilled water and then oven dried for 8 h at 70 °C. The obtained powder sample was calcined in a muffle furnace at a temperature of 450 °C for 3 h to obtain MgTiO₃ nanoparticles [13]

Characterization of the Synthesized Nanomaterials

The structure and morphology of the synthesized nanomaterials were examined with Zeiss Auriga high-resolution scanning electron microscopy (HRSEM). The crystal phase was studied with powder X-ray diffraction (XRD) Bruker AXS D8 Advance X-ray Diffractometer (Cu K α radiation, wavelength 1.5418 Å), and the functional groups of the synthesized material were analyzed using Fourier transform infrared (FTIR) spectroscopy, and the spectra were recorded on a Shimadzu FTIR-spectrophotometer at the range of 400-5000 cm⁻¹.

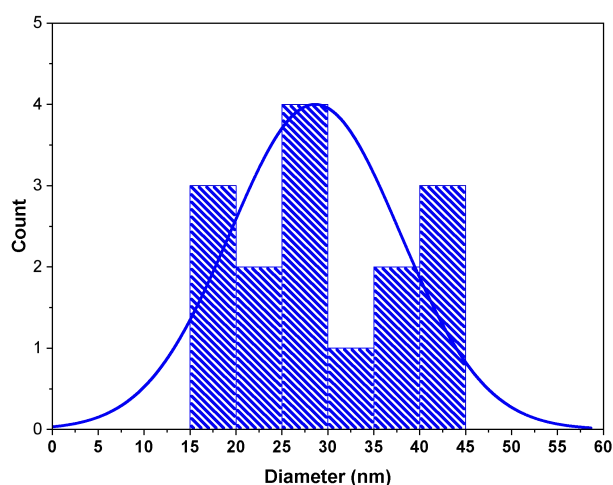
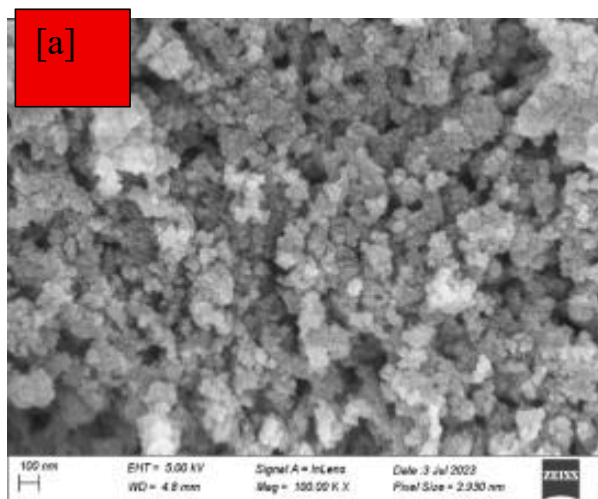
RESULTS AND DISCUSSION

HRSEM

HRSEM micrograph of TiO_2 nanoparticle in Figure 1(a) shows a spherical particle with little agglomeration indicating that the nucleation and growth methodology of TiO_2 nanoparticles controlled the formation of the grain size and morphology [14]. The average particle size is found to be 13.42 nm.

Figure 1(b) shows the HRSEM micrograph of MgO nanoparticles. They are irregular spherical particles with slight agglomeration having average particle size of 14.65 nm. Salman et al [15] reported the synthesis of MgO nanoparticle prepared by the microwave and sol-gel approaches to have average grain size of approximately 50 nm and 72 nm indicating that, synthesis methods applied to MgO nanoparticles greatly determine their shape and size. Similarly, Abinaya and Kavitha, [16], pointed the effect of different method of synthesis on the morphology of MgO nanoparticle which is due to the surface energy and size.

The HRSEM micrograph of MgTiO_3 nanoparticles shown in Figure 1(c), indicates a uniformly distributed spherical particles with little agglomeration with a particle size of 10.47 nm. This agglomeration may be due to strong interparticle interactions such as van der Waals forces and electrostatic attractions and also variations in ionic radii and diffusion rates of the constituent elements [17].



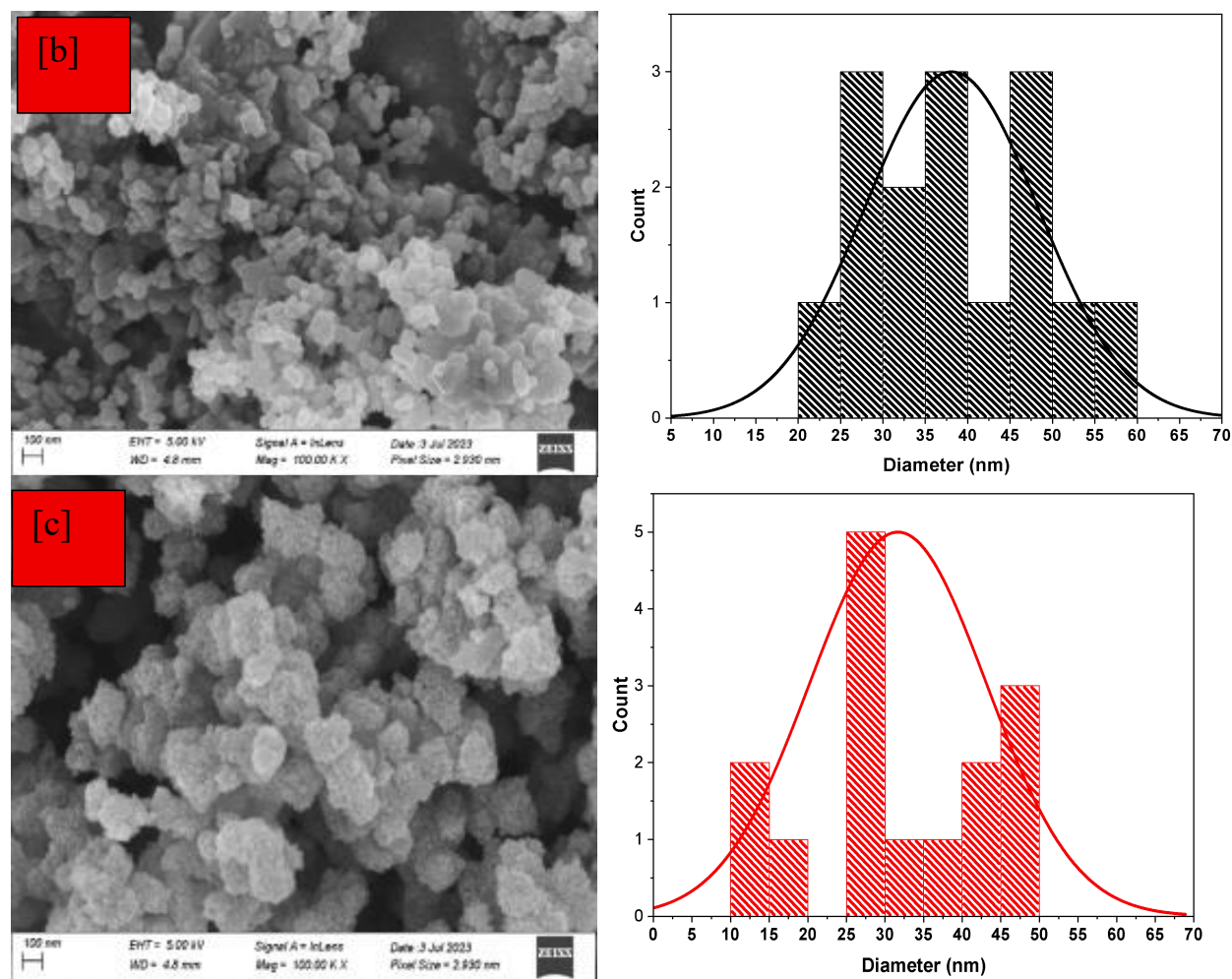


Figure 1: HRSEM Micrograph (a) TiO_2 (b) MgO (c) MgTiO_3 Nanoparticles

XRD

Figure 2 shows the XRD spectra of TiO_2 , MgO , and MgTiO_3 nanoparticles reflecting their distinct crystalline phases.

The diffraction pattern of TiO_2 nanoparticles has distinct peaks of the anatase phase, with 2θ values of 25.45° , 37.77° , 47.57° , 54.79° and 62.44° which are attributed to the crystal planes of (101), (004), (200), (211) and (204), according to the Joint Committee on Powder Diffraction Standards (JCPDS) card number 21-1272.

MgO nanoparticles diffraction pattern contain characteristic peaks appearing at 2θ positions of 6.9° , 42.9° , 62.3° , 74.7° , and 78.6° corresponding to 111, 200, 220, 311, and 222 planes, respectively which are in accordance with JCPDS card number 65-0476, and this indicates the presence of periclase MgO nanoparticle, while the XRD pattern of MgTiO_3 shows

peaks at 2θ values of 27° , 38° , 41° , 54° , and 56° for the 214, 309, 232, 223, and 321 crystal planes, respectively which correspond to the JCPDS card number 02-0874 indicating the presence of mixture of two phases for MgTiO₃. The residual peaks observe in the XRD pattern of MgTiO₃ may be due to the presence of MgO and TiO₂ phases in the sample pointing towards the presence of both phases in the sample. The crystallite sizes were calculated using the Debye-Scherrer equation (Equation 1) to be 19.10 nm, 39.30 nm and 17.51 nm for TiO₂, MgO and MgTiO₃. Comparing the observed crystallite sizes to other works, Mustapha et al [18] synthesized TiO₂ nanoparticles by the sol-gel process from titanium (IV) isopropoxide and sodium hydroxide and the crystallite size was estimated to be 12.34 nm, indicating that strain effects contribute little to broadening the diffraction peaks consistent with the anatase phase of TiO₂. Similarly, Sim et al [19] used the sol gel method for the synthesis of MgO nanoparticle and the crystallite size was found to be 18.92 nm. In the present work, it is evident that crystallite size in MgTiO₃ decreased when compared to that of TiO₂ and MgO. This may be due to lattice strain and defects and also due to Mg-Ti ion interaction which may affect nucleation and growth kinetics, resulting in smaller crystallite sizes. Furthermore, the structural complexity of MgTiO₃ over TiO₂ and MgO could also lead to retarded grain development and increased structural disorder, further restricting crystallite size.

$$D = \frac{k\lambda}{\beta \cos \theta} \quad (1)$$

where D is the crystallite size (nm), k is a constant (0.94), λ is the wavelength of the X-ray radiation ($\text{CuK}\alpha = 0.1541 \text{ nm}$), β is the full width at half maximum (FWHM) of the intense and broad peaks and θ is the Bragg's or diffraction angle.

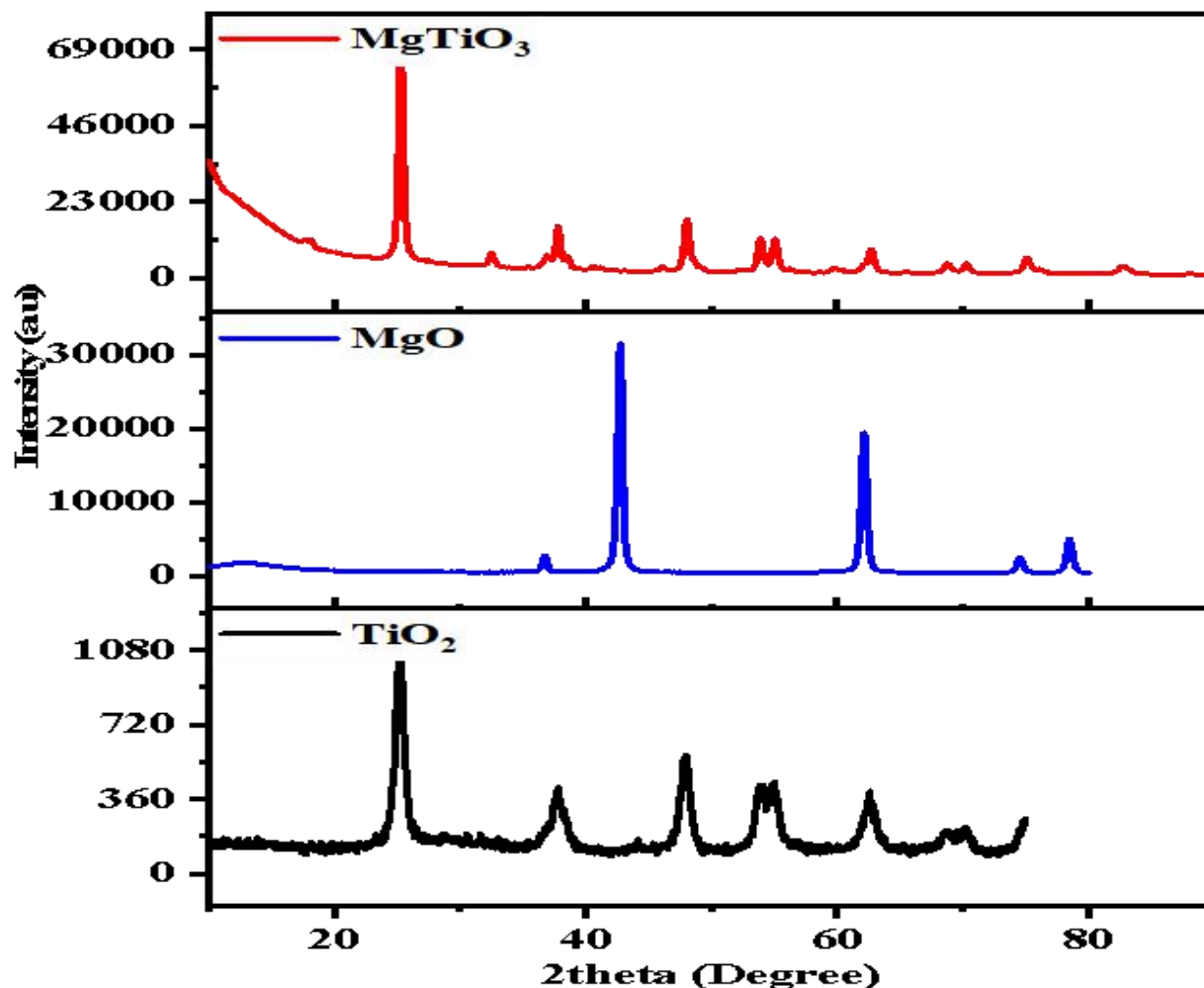


Figure 2: XRD Pattern of (a) TiO_2 (b) MgO (c) MgTiO_3

FTIR

The FTIR spectra of MgO , TiO_2 , and MgTiO_3 nanoparticles are shown in Figure 3. TiO_2 shows characteristic absorption bands at 3400 cm^{-1} , 1630 cm^{-1} , 603 cm^{-1} , and 489 cm^{-1} . The broad band at 3400 cm^{-1} is attributed to the O–H stretching vibration of surface hydroxyl groups due to adsorbed water molecule. Ghoniem et al [20], also reported an intense band at 3438 cm^{-1} attributed to the TiO_2 nanorods O–H group stretching vibration, which is in good agreement with the 3400 cm^{-1} band reported in this work. In addition, [21] noticed a wide absorption band in the range of $3300\text{--}3600\text{ cm}^{-1}$, showing the presence of hydroxyl groups generated during TiO_2 synthesis. This suggests that the presence of hydroxyl groups and water adsorbed is a feature of TiO_2 nanomaterials as also reported in previous studies [22, 23]

The bending vibration of H causes the band at 1630 cm⁻¹–O–H of molecular water. The peaks at 603 cm⁻¹ and 489 cm⁻¹ are attributed to the Ti–O stretching vibrations, which are characteristic of TiO₂ lattice vibrations. MgO reveals absorption bands at 3300 cm⁻¹, 1650 cm⁻¹, 1430 cm⁻¹, 670 cm⁻¹, and 580 cm⁻¹. The bands at 3300 cm⁻¹ and 1650 cm⁻¹ are attributed to O–H stretching and bending vibrations of adsorbed water, respectively. The band at 670 cm⁻¹ and 580 cm⁻¹ correspond to Mg–O stretching vibrations, typical of the Mg–O crystal lattice. MgTiO₃ shows absorption bands at 3400 cm⁻¹, 1456 cm⁻¹, 1138 cm⁻¹, 614 cm⁻¹, and 456 cm⁻¹. The peak at 3400 cm⁻¹ is due to O–H stretching of water adsorbed, and the band at 1456 cm⁻¹ is attributable to C–H bending vibration. The bands at 614 cm⁻¹ and 456 cm⁻¹ are due to metal-oxygen stretching (Ti–O–Mg–O).

The presence of hydroxyl group on the surface of the synthesized nanomaterials maybe attributed to the high surface area and reactivity of nanoparticles resulting in surface hydroxylation

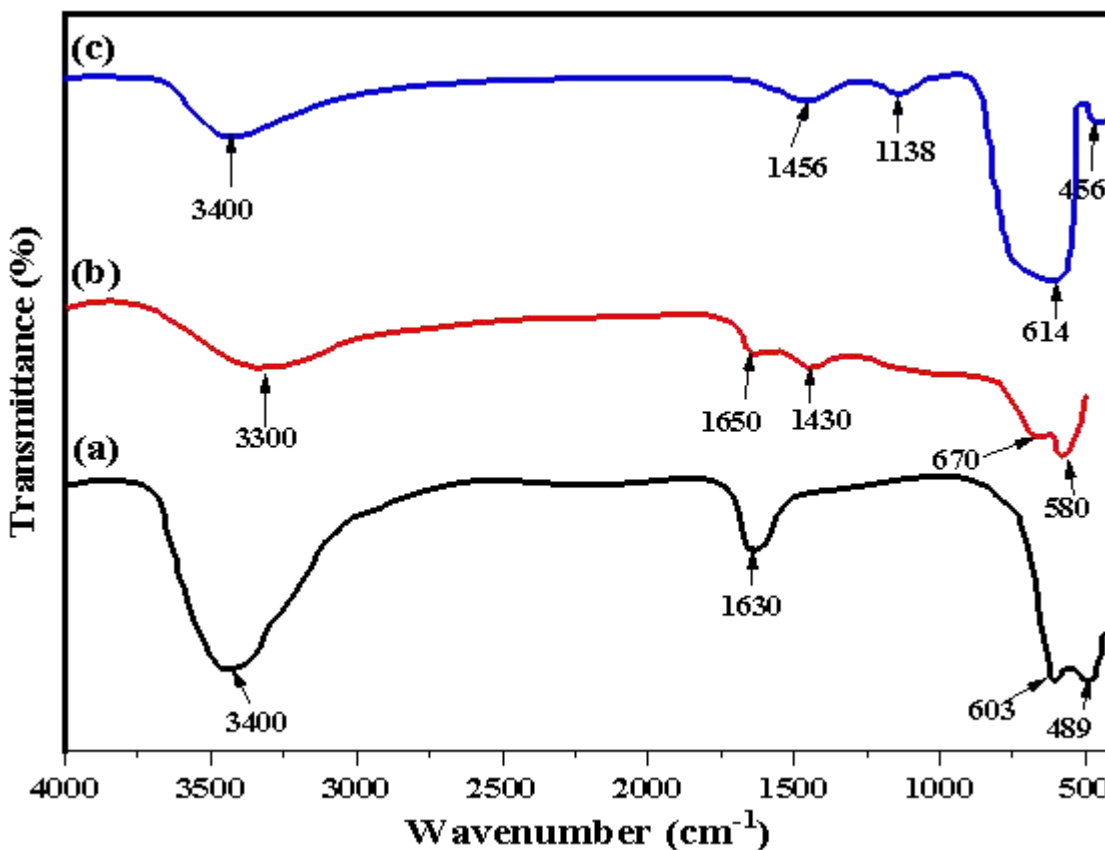


Figure 3: FTIR Spectra (a) TiO₂ (b) MgO (c) MgTiO₃ nanoparticles

CONCLUSION

The one-pot synthesis of MgTiO₃ particle was conducted via sol gel method and was compared with TiO₂ and MgO. The nanomaterials were characterized using HRSEM, XRD and FTIR. The HRSEM study revealed the morphological features of TiO₂, MgO, and MgTiO₃ nanoparticles, driven by synthesis conditions and processing parameters. TiO₂ is homogeneously nanospherical in morphology, while MgO and MgTiO₃ showed irregular morphologies with little agglomeration. Particle shape and size variations are related to parameters such as calcination time, precursor concentration, and nucleation processes. XRD analysis confirmed the crystalline phases of TiO₂, MgO, and MgTiO₃ with diffraction patterns in agreement with JCPDS standards. Crystallite size calculations indicated lower crystallite size for MgTiO₃ compared to TiO₂ and MgO which may be due to structural complexity, lattice strain, defects, and ion-ion interactions that affect nucleation and growth kinetics. The existence of residual MgO and TiO₂ peaks in the MgTiO₃ XRD pattern, indicates incomplete phase transformation. FTIR measurement identified the characteristic functional groups in TiO₂, MgO and MgTiO₃. Hydroxyl groups and adsorbed water molecules were present in all the materials, represented by broad O–H stretching bands. Individual metal-oxygen vibrations were observed in TiO₂, MgO but MgTiO₃ has mixed metal-oxygen interactions. The findings emphasize the contribution of structural complexity on the crystallite size and grain evolution, offering insights into the phase composition and material characteristics of MgTiO₃ nanoparticle that can influence its applications across diverse areas.

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