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Structural, Morphological and Elemental Characterization of MgO Nanoparticles Synthesized by the Sol–Gel Method

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Abstract:

Magnesium oxide (MgO) nanoparticles were successfully synthesized using a sol–gel method and systematically investigated to evaluate their structural, morphological and elemental characteristics. The synthesized MgO nanoparticles were characterized using X-ray diffraction (XRD), scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX). The XRD pattern revealed characteristic diffraction peaks corresponding to the (111), (200), (220), (311) and (222) planes, confirming the formation of a cubic face-centered cubic MgO structure. The observed reflections were in good agreement with the standard JCPDS Card No. 45-0946. The average crystallite size calculated using the Debye–Scherrer equation was found to be 54.36 nm, confirming the nanocrystalline nature of the synthesized material. SEM analysis revealed a fine, irregular and granular morphology with considerable agglomeration of the nanoparticles, which is attributed to the high surface energy of nanosized MgO particles. The EDX spectrum confirmed the presence of Mg and O as the major constituent elements, with no significant impurity elements detected. The elemental mapping further demonstrated the distribution of Mg and O throughout the analyzed region. The combined XRD, SEM and EDX results confirm the successful synthesis of crystalline and predominantly phase-pure MgO nanoparticles by the sol–gel method, indicating their potential suitability for surface-dependent applications such as catalysis, sensing, adsorption and other advanced functional applications.

Keywords: Magnesium oxide nanoparticles; MgO; Sol–gel method; X-ray diffraction; SEM; EDX; Crystallite size; Nanomaterials.

1. Introduction

Nanotechnology has emerged as an important area of research because materials exhibit significant changes in their structural, surface, chemical, optical, electrical and catalytic properties when their dimensions are reduced to the nanometer scale [1, 2]. In particular, metal oxide nanoparticles have attracted considerable attention because of their tunable physicochemical properties, high surface-to-volume ratio, thermal stability and chemical versatility. Among the various metal oxides, magnesium oxide (MgO) is an important alkaline-earth metal oxide that has received considerable research interest in recent years. MgO consists of Mg^{2+} and O^{2-} ions arranged in a stable ionic crystal lattice and generally crystallizes in the cubic face-centered cubic (FCC) periclase structure. At the nanoscale, MgO exhibits properties that can differ significantly from those of bulk MgO because of its increased surface area, large fraction of surface atoms and size-dependent structural characteristics [3, 4]. These features make MgO nanoparticles attractive for applications in catalysis, adsorption, sensors, ceramics, environmental remediation, energy-related materials and biomedical technologies. MgO is particularly attractive as a functional nanomaterial because of its high thermal stability, chemical stability, dielectric properties, basic surface characteristics and relatively low toxicity. The surface of MgO contains highly active sites that can interact with gases, organic molecules and other chemical species, making nanosized MgO useful in heterogeneous catalysis and sensing-related applications [3-6]. The reduction of particle size to the nanometer range substantially increases the specific surface area and consequently the number of surface-active sites. Therefore, controlling the crystallite size, particle morphology, degree of agglomeration and crystalline phase is essential for obtaining MgO nanoparticles with desirable properties. Previous studies have demonstrated that nanostructured MgO can be employed in sensor technology, polymeric materials, battery-related systems and other functional applications because of its distinctive electrical and surface properties [7-9].

In the present work, MgO nanoparticles were synthesized by a simple sol–gel method and systematically characterized to investigate their structural, morphological and elemental properties. The crystalline structure and phase formation of the synthesized MgO nanoparticles were examined using X-ray diffraction (XRD), while the surface morphology, particle distribution, and agglomeration behavior were investigated using scanning electron microscopy (SEM). The elemental composition and spatial distribution of the constituent elements were analyzed using energy-dispersive X-ray spectroscopy (EDX) and elemental mapping.

2. Material and methods

2.1 Synthesis of MgO nanoparticles by sol–gel method

MgO nanoparticles were synthesized by a sol–gel method using magnesium acetate tetrahydrate [$Mg(CH_3COO)_2 \cdot 4H_2O$] as the magnesium precursor. Analytical-grade magnesium acetate tetrahydrate was accurately weighed and dissolved in distilled water under continuous magnetic stirring to obtain a homogeneous precursor solution. Citric acid was used as a complexing agent, and the citric acid-to-metal ion molar ratio was maintained at approximately 1:1. The resulting solution was continuously stirred at approximately 60–70 °C for 2 h to facilitate complex formation and obtain a clear and homogeneous sol [11-14]. The pH of the solution was adjusted to approximately 9–10 by the controlled addition of dilute ammonia solution under continuous stirring. With increasing pH, hydrolysis and condensation reactions occurred, resulting in the formation of a stable sol, which gradually transformed into a viscous gel. The obtained gel was aged for approximately 24 h at room temperature to promote further condensation and network formation. The aged gel was then dried at approximately 100 °C for 12 h to remove the solvent and residual moisture, producing a dry precursor powder [15, 16]. The dried precursor was gently ground using an agate mortar to obtain a homogeneous powder and subsequently subjected to calcination at 500 °C for 3 h in air. During calcination, the organic constituents and acetate groups were decomposed and removed, while the Mg–O network was developed and crystallized to form MgO nanoparticles.

3. Result and Discussion

Figure 1(a–d) presents the scanning electron microscopy (SEM) micrographs of the synthesized MgO nanoparticles at different magnifications, namely 1000×, 3000×, 5000× and 10,000× respectively. The SEM observations provide important information regarding the surface morphology, particle distribution, agglomeration and approximate particle dimensions of the synthesized MgO nanoparticles. In Figure 1(a), recorded at 1000× magnification with a 10 µm scale bar, the sample exhibits an irregular and highly aggregated surface morphology. Several relatively large, rounded and irregularly shaped agglomerates are observed, which are composed of numerous smaller MgO particles [16, 17]. The formation of such agglomerates can be attributed to the high surface energy of nanosized MgO particles, which promotes particle–particle interactions during drying and subsequent thermal treatment. At 3000× magnification [Figure 1(b)], with a 5 µm scale bar, the finer particulate structure becomes more clearly visible. The micrograph reveals a heterogeneous distribution of small MgO particles along with larger secondary agglomerates, indicating that the synthesized material possesses a nanoscale primary-particle structure with some degree of aggregation. In Figure 1(c), obtained at 5000× magnification, the morphology of the fine particles is more clearly resolved, showing a densely packed granular structure with interconnected particles [17, 18]. The increased magnification indicates that the large structures observed at lower magnification are actually assemblies of much smaller particles. Figure 1(d), recorded at 10,000× magnification with a 1 µm scale bar, provides the highest-resolution morphological observation and clearly shows numerous fine, irregular to quasi-rounded MgO nanoparticles distributed throughout the surface.

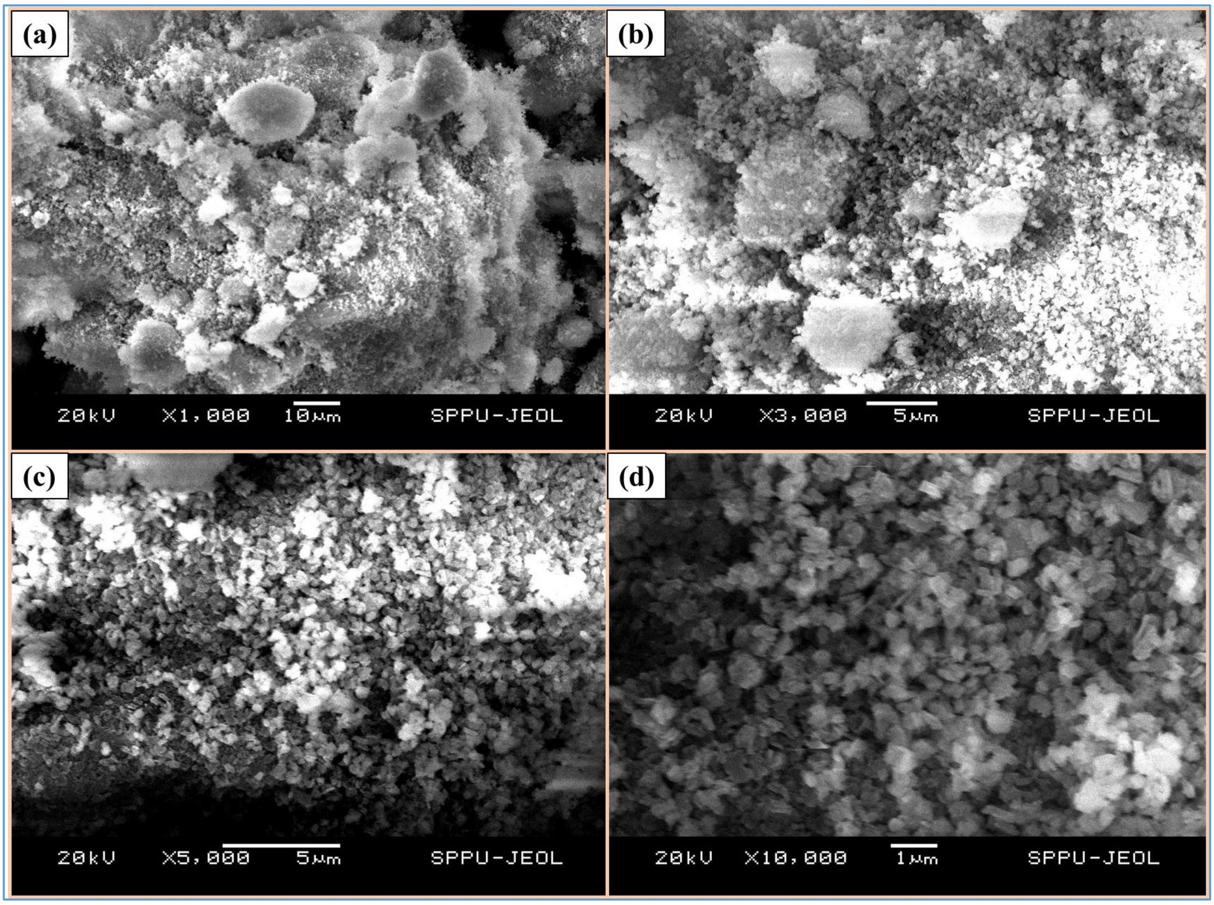

Figure 1 (a-d). SEM micrograph of MgO nanoparticles at different magnification

The particles appear strongly interconnected and partially agglomerated, while the presence of numerous interparticle voids suggests a relatively porous and rough surface. Such a morphology can provide a comparatively large active surface area, which is advantageous for applications involving surface-dependent processes such as gas sensing, catalysis and adsorption. SEM results confirm the successful formation of a fine-grained MgO nanoparticulate structure by the sol–gel method, although some agglomeration is evident [19, 20]. The progressive improvement in morphological resolution from Figure 1(a) to Figure 1(d) demonstrates that the observed microscale agglomerates consist of considerably smaller primary particles, supporting the nanoscale nature of the synthesized MgO material [19, 21].

Figure 2(a) shows the energy-dispersive X-ray spectroscopy (EDX) spectrum of the synthesized MgO nanoparticles. Figure 2(b) presents the corresponding SEM elemental mapping. The EDX spectrum exhibits two prominent characteristic peaks corresponding to oxygen (O) and magnesium (Mg), confirming the presence of the principal constituent elements of MgO. The relatively intense Mg and O signals indicate the successful formation of the MgO phase through the sol–gel synthesis process [22, 23].

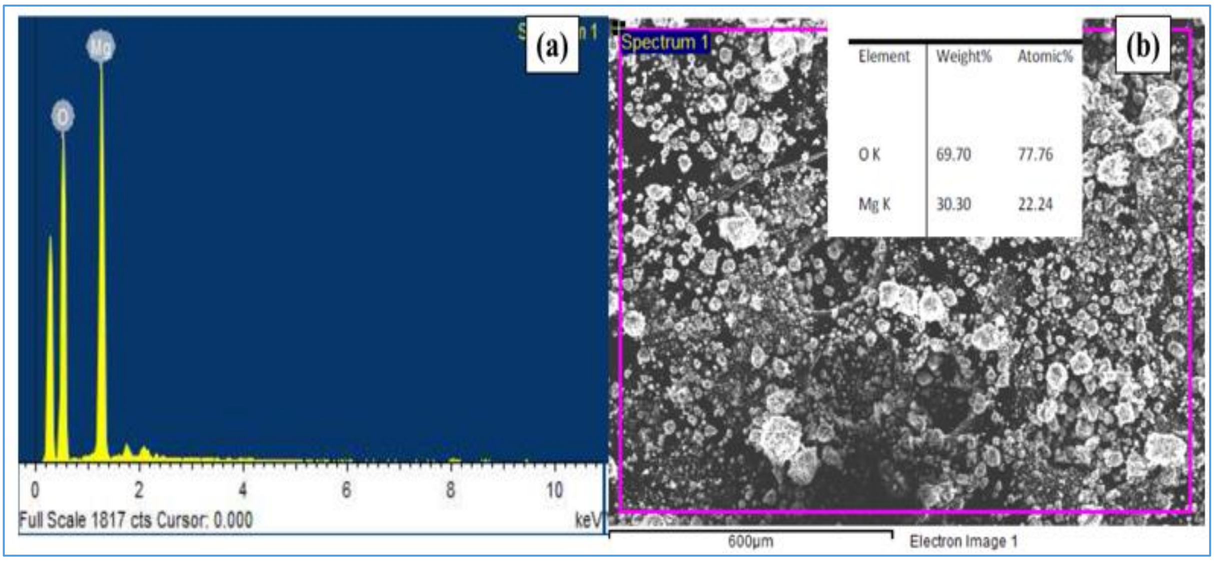

Figure 2. (a) EDX spectrum & (b) Elemental mapping of MgO nanoparticles

The quantitative EDX analysis shows that oxygen constitutes approximately 69.70 wt.% (77.76 at.%), whereas magnesium accounts for 30.30 wt.% (22.24 at.%). The observed deviation from the ideal stoichiometric MgO composition can be associated with several factors, including surface oxygen adsorption, the interaction of the electron beam with the sample, particle morphology, and the limitations of quantitative EDX analysis for light elements such as oxygen [24, 25]. No significant peaks corresponding to foreign or impurity elements are observed, indicating good chemical purity of the synthesized material. The elemental mapping shown in Figure 2(b) demonstrates the spatial distribution of Mg and O throughout the analyzed region. The Mg and O elements are distributed over the particle/agglomerate surface, supporting the homogeneous formation of the MgO nanoparticulate material. The combined EDX spectrum and elemental mapping therefore provide strong evidence for the successful synthesis of MgO nanoparticles with good elemental purity and relatively uniform elemental distribution by the sol–gel method [25-27].

Figure 3 shows the X-ray diffraction pattern of the synthesized MgO nanoparticles. The diffraction pattern was analyzed using Cu Kα radiation with a wavelength of approximately 1.5406 Å. The characteristic reflections of MgO can be indexed to the cubic, face-centered cubic (FCC) periclase structure with space group Fm-3m [28, 29].

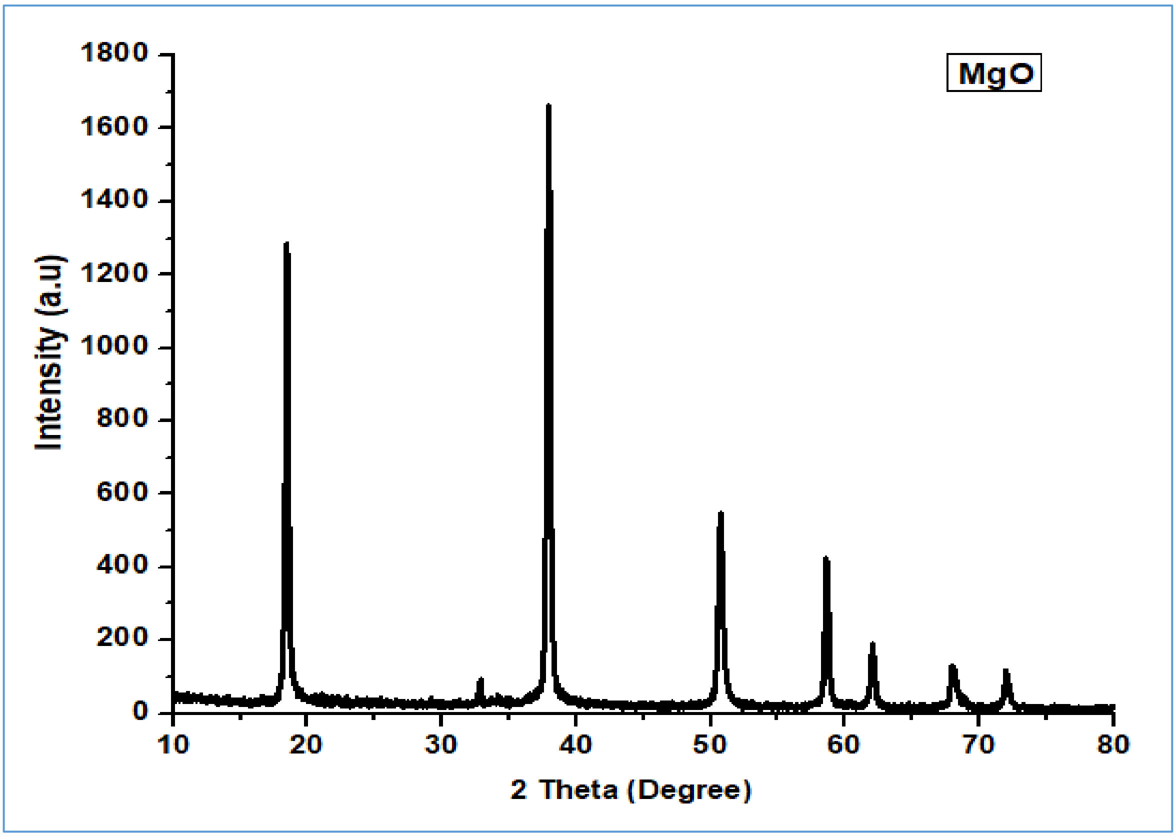

Figure 3. XRD pattern of MgO nanoparticles

The observed diffraction peaks are consistent with the standard JCPDS/PDF Card No. 45-0946, which is widely reported for cubic MgO. The principal MgO reflections occur at approximately $2\theta = 36.9°$, 42.9°, 62.3°, 74.6–74.7° and 78.6° corresponding to the (111), (200), (220), (311) and (222) crystallographic planes respectively. The presence of these characteristic reflections confirms the formation of crystalline MgO nanoparticles [29, 30]. The intense reflections around the (111) and (200) planes indicate good crystallinity of the synthesized material, while the additional higher-order reflections at (220), (311) and (222) further confirm the cubic MgO phase. No prominent additional crystalline peaks attributable to secondary Mg-containing phases are evident in the principal MgO region, suggesting good phase purity. The average crystallite size was estimated from the broadening of the diffraction peaks using the Debye–Scherrer equation (Eq. 1) [30, 31].

$$D = K\lambda/\beta\cos\theta \qquad \text{(Eq. 1).}$$

Where,

D represents the crystallite size, K is the shape factor (0.9), λ is the wavelength of Cu Kα radiation (1.5406 Å), β is the full width at half maximum (FWHM) of the diffraction peak, and θ is the Bragg angle.

The calculated average crystallite size of the MgO nanoparticles is 54.36 nm, confirming their nanocrystalline nature. The relatively sharp diffraction peaks together with the calculated nanoscale crystallite size indicate that the sol–gel method is effective for producing well-crystallized MgO nanoparticles [31].

4. Conclusions

In the present work, MgO nanoparticles were successfully synthesized by the sol–gel method and their structural, morphological and elemental properties were systematically investigated using XRD, SEM and EDX techniques. The XRD analysis confirmed the formation of cubic face-centered cubic MgO, with characteristic diffraction reflections corresponding to the (111), (200), (220), (311) and (222) planes, in good agreement with JCPDS Card No. 45-0946. The average crystallite size calculated using the Debye–Scherrer equation was found to be 54.36 nm, confirming the nanocrystalline nature of the synthesized material. SEM observations revealed a fine granular morphology consisting of interconnected nanoparticles with some degree of agglomeration, which can be attributed to the high surface energy of nanosized MgO particles. EDX analysis confirmed the presence of magnesium and oxygen as the major constituent elements, while elemental mapping demonstrated their distribution throughout the analyzed region. The combined characterization results establish that the sol–gel method is an effective route for the preparation of crystalline MgO nanoparticles with nanoscale crystallite dimensions and good elemental composition. The obtained structural and morphological characteristics make the synthesized MgO nanoparticles promising candidates for further investigation in various functional applications.

5. Future scope

The synthesized MgO nanoparticles also be explored for gas sensing, adsorption, catalytic, environmental and antibacterial applications. The fabrication of MgO-based thin films and nanocomposites with other metal oxides or polymers may provide improved functional properties and broaden their applications in sensing, electronic, energy and biomedical technologies.

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