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XRD-Based Structural and Crystallite Size Analysis of Nano-MgO Prepared via Sol–gel Method

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Abstract. Nano-MgO was synthesized via a nitrate–citrate sol–gel combustion method, in which magnesium nitrate acted as the oxidizing precursor and citric acid served simultaneously as the chelating agent and organic fuel. The formation of a homogeneous Mg–citrate coordination network enabled controlled gelation, while the auto-combustion process produced a porous MgO matrix composed of ultrafine crystallites. Calcination at 800 °C promoted phase consolidation, removal of organic residues, and enhanced long-range atomic ordering. X-ray diffraction confirmed the exclusive formation of the cubic periclase phase ($Fm\bar{3}m$), with no detectable hydroxide or carbonate impurities. Crystallite size analysis yielded an average size of 16.03 nm based on the Scherrer equation, whereas the Williamson–Hall method produced a larger value of 25.44 nm with a microstrain of 5.37×10^{-4}, indicating minimal lattice distortion. The discrepancy between the two size estimates reflects the contribution of microstrain effect not considered in the Scherrer approach. Overall, the synthesized nano-MgO exhibits high crystallinity, narrow crystallite size distribution, and excellent structural stability, highlighting its potential for catalytic, gas-sensing, and high-temperature functional applications. These results demonstrate that the sol–gel combustion method is an efficient and reliable strategy for producing nanocrystalline MgO with well-controlled structural characteristics.

Keywords: *Nano-MgO, sol–gel combustion, XRD analysis, Crystallite Size, microstrain, Scherrer, Williamson–Hall*

Abstrak. Nano-MgO disintesis melalui metode *sol–gel combustion* nitrat–sitrat, di mana magnesium nitrat berfungsi sebagai prekursor pengoksidasi dan asam sitrat berperan ganda sebagai ligan pengkelat dan bahan bakar organik. Pembentukan jaringan koordinasi Mg–sitrat yang seragam memungkinkan proses gelasi yang terkontrol, sementara *auto-combustion* menghasilkan matriks MgO berpori dengan kristalit sangat halus. Kalsinasi pada 800 °C mendorong konsolidasi fasa, penghilangan residu organik, dan peningkatan keteraturan atomik jangka panjang. Difraksi sinar-X mengonfirmasi terbentuknya fasa periklase kubik ($Fm\bar{3}m$) tanpa adanya pengotor hidroksida maupun karbonat. Analisis ukuran kristalit menggunakan persamaan Scherrer menghasilkan nilai 16,03 nm, sedangkan metode Williamson–Hall memberikan ukuran 25,44 nm dengan mikroregangan $5,37 \times 10^{-4}$, yang menunjukkan distorsi kisi yang sangat rendah. Perbedaan kedua nilai tersebut mencerminkan kontribusi regangan yang tidak diperhitungkan dalam pendekatan Scherrer. Nano-MgO yang dihasilkan menunjukkan kristalinitas tinggi, distribusi ukuran kristalit yang sempit, dan stabilitas struktural yang baik, sehingga cocok untuk aplikasi katalitik, sensor gas, dan material fungsional bersuhu tinggi. Secara keseluruhan, metode *sol–gel combustion* terbukti efisien dan andal untuk menghasilkan MgO nanokristalin dengan karakteristik struktural yang terkontrol.

Kata kunci: *Nano-MgO, sol–gel combustion, analisis XRD, ukuran kristal, mikroregangan, Scherrer, Williamson–Hall*

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INTRODUCTION

Magnesium oxide (MgO) nanoparticles have garnered significant attention in materials science due to their exceptional physicochemical properties, including high thermal stability, catalytic activity, and antimicrobial performance (Gatou, Skylla, et al., 2024; Hornak, 2021; Sim, Gençaslan, &

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Merdan, 2024). Magnesium oxide stands out due to its simple cubic (rock-salt) structure, strong ionic bonding between Mg^{2+} and O^{2-} ions (Yang et al., 2024). These attributes make nano-MgO a promising candidate for applications in heterogeneous catalysis (Farouk et al., 2024; Eladeb, 2024; Gajengi, Sasaki, & Bhanage, 2017; Wang et al., 2018), environmental remediation (Cai et al., 2017; Nassar et al., 2017; Bazrafshan et al., 2023;Gatou, et al., 2024), and advanced ceramics (Chanda et al., 2022). In comparison with bulk MgO, nanoscale MgO exhibits enhanced surface area, increased defect density, and higher reactivity; therefore, precise control over particle size and crystal structure is essential for optimizing its performance in advanced technological applications (Hornak, 2021).

Various synthesis methods have been reported for the preparation of MgO nanoparticles, including co-precipitation, hydrothermal, and combustion techniques (Hornak, 2021). Although these approaches are relatively well established, they often suffer from limitations such as particle agglomeration, broad size distribution, or the requirement for high-pressure processing conditions. As a result, achieving precise control over crystallite size and structural homogeneity remains a significant challenge in MgO nanoparticles synthesis.

Among the available synthesis strategies, the sol–gel method has been widely recognized as one of the most efficient and versatile routes for fabricating metal oxide nanomaterials with high purity and homogeneous microstructures. The sol–gel process offers several advantages, including low synthesis temperature, tunable stoichiometry, molecular-level mixing, and excellent control over particle growth and morphology (Saleh, 2020; Negrescu et al., 2022). For MgO specifically, the sol–gel method enables the formation of uniform precursor gels that, upon calcination, yield nanoparticles with controllable crystallinity and crystallite size. Nassar et al. (2017) successfully synthesized nano-MgO using oxalic acid as the fuel, yielding a crystallite size of approximately 12 nm.

At the nanoscale, MgO exhibits unique crystallographic behaviors such as lattice contraction or expansion, altered microstrains, and defect-driven reactivity, which differ substantially from those of bulk materials. These nanoscale structural features directly influence surface basicity, oxygen vacancy concentration, and overall reactivity, which are crucial for the performance of MgO-based catalysts and functional materials (Hornak, 2021). Therefore, understanding the crystallographic evolution of MgO nanoparticles as a function of synthesis and heat-treatment parameters is essential for designing high-performance materials with tailored structural and surface properties (Kumari, Pakshirajan, & Pugazhenthi, 2023).

To evaluate the structural properties of the synthesized nanoparticles, X-ray diffraction (XRD) constitutes one of the most powerful and indispensable techniques. XRD analysis provides key structural information such as phase purity, lattice parameters, and crystallite size (Giannini et al., 2016). The Scherrer equation and Williamson–Hall analysis have been extensively applied to interpret the broadening and shifting of diffraction peaks, providing insights into the nanocrystalline nature and internal strain distribution (Rani et al., 2019). Recent reports indicate that MgO synthesized through sol–gel, precipitation and

hydrothermal routes predominantly exhibits the periclase ($Fm\bar{3}m$) phase, though its crystallite size and lattice distortion are highly dependent on synthesis temperature, precursor composition, and calcination duration. Bindhu et al., (2016) successfully synthesized MgO nanoparticles through a wet chemical reaction using magnesium nitrate and sodium hydroxide yielding spherical particles with an average size of 16 nm. Sim et al., (2024) successfully synthesized nano-MgO via the sol–gel method using magnesium nitrate as the precursor and a 25% ammonia solution as the pH-adjusting agent, producing an average crystallite size of approximately 18.92 nm.

Despite the extensive research on MgO nanoparticles, there remains a continuous need to systematically correlate sol–gel synthesis conditions with the resulting crystallite size and structural characteristics, particularly using XRD-based analysis. Such correlations are essential for optimizing nano-MgO for advanced catalytic, optical and environmental applications. This study focuses on examining the crystal structure, phase composition, and crystallite size of MgO nanoparticles synthesized via the sol–gel method using comprehensive XRD analysis

MATERIAL AND METHODS

Materials

Magnesium nitrate hexahydrate $Mg(NO_3)_2.6H_2O$, ≥ 99%, Sigma-Aldrich) was used as the primary precursor. Citric acid monohydrate (≥ 99%, Merck) served as a chelating agent to control the complexation of Mg2+. Ammonium hydroxide solution 25% (Merck) was employed for pH adjustment during gel formation. Deionized water was used as the solvent throughout all stages of the synthesis.

Methods

The synthesis of nano-MgO was carried out using a sol–gel combustion approach based on nitrate precursors. In a typical synthesis, 5.12 g of $Mg(NO_3)_2 \cdot 6H_2O$ was dissolved in 40 mL deionized water under continuous magnetic stirring at room temperature to obtain a clear precursor solution. Citric acid was subsequently added at a citrate-to-magnesium molar ratio of 2:1 to ensure the formation of a stable Mg–citrate complex. The mixture was heated to 70 °C and maintained at this temperature until a fully homogeneous solution was achieved.

A 25% NH_4OH solution was added dropwise to increase the pH to 10, thereby initiating gel formation. The resulting sol was heated at 70 °C until it transformed into a viscous gel. This gel was subsequently subjected to an aging process to produce a xerogel, characterized as a solid, powder-like material. The obtained xerogel was then calcined at 800 °C for 2 hours to facilitate the formation of nano-MgO metal oxide.

Crystallographic characterization was performed using a Bruker D8 Advance X-ray diffractometer equipped with Cu Kα radiation (λ = 1.5406 Å), operating over a 2θ range of 10–90° with a scanning rate of 0.02° s^{-1}. The resulting diffraction patterns were used for phase identification by referencing the Crystallography Open Database (COD), employing CIF file #1011193 corresponding to the Periclase (MgO) phase. The crystallite size was subsequently determined using the Scherrer equation, Eq. (1):

$$D = \frac{K\lambda}{\beta cos\theta}, \quad \ldots . (1)$$

where *D* denotes the crystallite size (nm), *K* represents the crystallite shape factor (0.9), *λ* is the X-ray wavelength, *β* is the full width at half maximum (FWHM) of the diffraction peak (in radians), and *θ* is the diffraction angle. Furthermore, lattice microstrain was evaluated through the Williamson–Hall method, Eq. (2):

$$\beta cos\theta = \frac{K\lambda}{D} + 4\varepsilon\, sin\theta \quad \ldots . (2)$$

in which a plot of βcosθ versus 4sinθ was generated to obtain the crystallite size from the intercept and the microstrain from the slope of the linear fit.

RESULTS AND DISCUSSION

Formation of the MgO Phase During the Sol–Gel Combustion Process

The synthesis of nano-MgO in this study was carried out via a sol–gel combustion route involving the formation of a Mg–citrate complex, followed by a spontaneous combustion reaction between magnesium nitrate (as the oxidizer) and citric acid (as the reductant/chelating agent) (Gatou, Skylla, et al., 2024;Dhal et al., 2024;Yin et al., 2024). Figure 1 shows the photographic images of the precursor system during the sol–gel synthesis of nano-MgO.

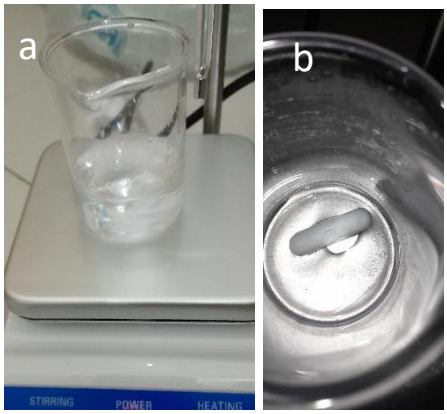

Figure 1. Images of the sol–gel synthesis stages of nano-MgO: (a) precursor sol; and (b) nano-MgO gel prior to combustion and calcination

At the initial stage (Figure 1(a), $Mg(NO_3)_2.6H_2O$ was completely dissolved in deionized water under continuous stirring, forming a clear and homogeneous sol. The absence of visible turbidity or undissolved particles indicated successful precursor dissolution and uniform dispersion of Mg^{2+} ions in the solution. Upon the addition of citric acid followed by pH adjustment, the sol gradually transformed into a viscous and homogeneous gel (Figure 1(b), confirming effective chelation between Mg^{2+} ions and citrate ligands. The overall combustion process may be represented by the generalized reaction (M. Y. Nassar et al., 2017).

$$18Mg(NO_3)_2.6H_2O(aq) + 10C_6H_8O_7 \rightarrow 18MgO(s) + 60CO_2(g) + 148H_2O(g) + 18N_2(g)$$

This exothermic reaction produces a porous gel accompanied by the release of abundant gaseous byproducts such as CO_2, H_2O vapor, and N_2, which ultimately leads to the formation of a material characterized by small crystallite dimensions and a highly porous structure (K. I. Nassar, Teixeira, & Graça, 2025).

During the initial heating stage, magnesium nitrate undergoes thermal decomposition, producing MgO along with gaseous species and water vapor. In parallel, citric acid experiences pyrolytic decomposition and combustion, yielding CO_2 and amorphous carbon. The combination of these processes generates a porous inorganic framework, which subsequently undergoes structural refinement

during the calcination step (Periyasamy, Asrafali, & Lee, 2024; Hosni, Ali, & Shama, 2024). Calcination at 800 °C promotes the enhancement of MgO crystallinity, the removal of residual carbon species, and the controlled growth of crystallites. In studies examining the influence of calcination temperature on the structure of MgO, it has been found that MgO synthesized at higher calcination temperatures exhibits greater thermal stability and improved crystallinity (Huang, Yang, & Wang, 2020).

X-ray Diffraction Analysis for Crystallographic Structure Identification

The XRD pattern of the sample is presented in Figure 2. All diffraction peaks correspond to the cubic MgO phase (periclase, space group $Fm\bar{3}m$), with no detectable reflections attributable to $Mg(OH)_2$ or $MgCO_3$. This confirms that the combustion process followed by calcination is sufficient to achieve phase purification of the MgO structure.

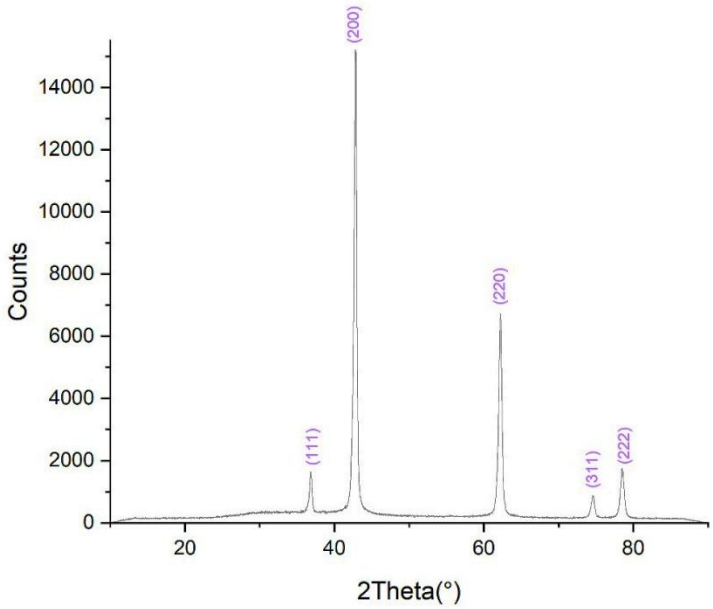

Figure 2. XRD Pattern of Nano-MgO

The diffraction pattern exhibits five major peaks located at 2θ = 37.14° (111), 43.11° (200), 62.46° (220), 74.83° (311), and 78.76° (222). All reflections match the characteristic periclase structure (COD #1011193), which is typically formed from nitrate-based precursors through the sol–gel combustion process (Amir et al., 2024; Gatou, Skylla, et al., 2024).

The strong peak intensity and the symmetrical peak profiles indicate a high degree of crystallinity, which is a well-established characteristic of MgO synthesized via citrate–gel combustion, particularly after high-temperature treatment. The (200) reflection at 43.11° exhibits the highest intensity, consistent with the typical diffraction pattern of cubic MgO.

Crystallite Size Analysis Using the Scherrer Equation

The crystallite size was calculated from the FWHM values of the diffraction peaks using the Scherrer equation. Table 1 presents the

crystallite dimensions obtained from the five principal 2θ reflections. The average Scherrer crystallite size was approximately 16.03 nm. The largest crystallite size was associated with the (111) reflection, whereas the smallest value corresponded to the higher-index (222) plane at 78.76°. A systematic decrease in crystallite size with increasing 2θ is consistent with the well-established trend of peak broadening in nanoscale oxide materials. Lower-index planes tend to exhibit preferential growth, resulting in larger crystallites along the (111) and (200) directions. The relatively small crystallite size is attributed to the intrinsic characteristics of the sol–gel combustion mechanism, in which rapid redox reactions generate ultrafine primary particles. Previous studies have reported that MgO synthesized through combustion routes typically exhibits crystallite sizes in the range of 15–30 nm (Sim et al., 2024; M. Y. Nassar et al., 2017). This value confirms that the sol–gel synthesis route effectively produced MgO nanoparticles within the lower-nanoscale regime (<20 nm), consistent with other reports on MgO obtained via wet-chemical synthesis.

Table 1. Crystallite Size Calculated from the Scherrer Formula

Plane (hkl)	2θ(°)	D (nm)
(111)	37.14	20.88
(200)	43.11	18.88
(220)	62.46	15.25
(311)	74.83	12.84
(222)	78.76	12.37

Williamson–Hall–Based Microstrain Analysis

Microstrain (ε) in the MgO nanoparticles was assessed using the Williamson–Hall (W–H) method to decouple the respective contributions of crystallite size and lattice strain to the observed peak broadening. The W–H plot (Figure 3) was generated by plotting βcosθ as a function of 4sinθ for all major diffraction reflections. Linear fitting of the data produced a slope of 5.37 × 10^{-4}, corresponding to a microstrain value of 0.0537%.

The relatively low microstrain value suggests that the sol–gel combustion synthesis route promotes uniform nucleation and progressive crystallization, thereby minimizing defect accumulation during drying and thermal decomposition. The intercept obtained from the W–H plot corresponds to a crystallite size of approximately 25.44 nm, which is larger than the value estimated using the Scherrer equation, as expected when strain-induced broadening is separated from size effects.

Due to the limited number of diffraction peaks and the absence of repeated measurements, error bars were not included in the Williamson–Hall plot. Nevertheless, the linear fitting provides a reasonable estimation of both crystallite size and lattice microstrain and the obtained values remain consistent with those previously reported for nanocrystalline metal oxides.

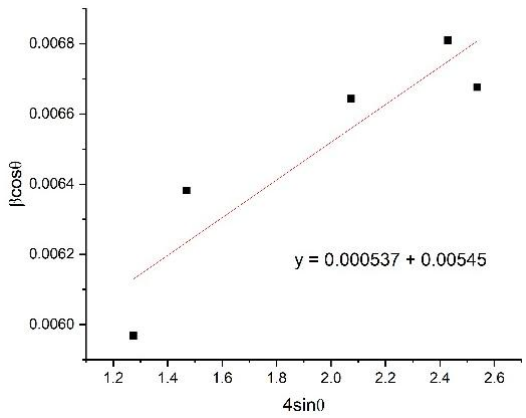

Figure 3. Williamson–Hall plot for nano-MgO

The average crystallite size calculated using the Scherrer equation was approximately 16.03 nm, whereas the Williamson–Hall

method yielded a slightly larger value of 25.44 nm, as expected due to the separation of strain effects. This discrepancy is consistent with the well-established tendency of the Scherrer equation to underestimate crystallite size because it does not account for strain-induced peak broadening. In-depth crystallographic structure and optoelectrical studies of nanomaterials by Munna et al., (2025) reported 'crystallite size, estimated through various models, ranged from 31.85 nm (Scherrer) to 61.08 nm (Williamson–Hall) in ZnO nanoparticles, clearly indicating that the W–H size is larger.

A key insight from this study is the inverse relationship between crystallite size and microstrain, which aligns with classical models of nanocrystal growth. However, the microstrain observed in this work remains relatively low despite the small crystallite size. This indicates that the nucleation process was highly efficient, with minimal defect formation, well-controlled calcination temperatures, and uniform drying of the gel matrix. These findings highlight the effectiveness of the sol–gel route in producing MgO nanoparticles with high crystallinity even at the nanoscale.

The crystallite size and lattice microstrain of the synthesized nano-MgO were systematically evaluated using the Scherrer and Williamson–Hall methods based on XRD peak broadening. The relatively low microstrain value indicates minimal lattice distortion and reflects the high crystallographic quality achieved through sol–gel combustion followed by calcination at 800 °C. These results confirm that XRD-based analysis provides reliable insight into the structural characteristics of nano-MgO synthesized via this route. Nevertheless, further characterization using SEM or TEM is considered as future work to provide direct morphological evidence and particle size distribution of the synthesized nano-MgO. In addition, particle size analysis (PSA) may be employed to complement the crystallite size information obtained from XRD measurements.

Correlation of Sol–Gel Combustion Synthesis with Structural Features and Functional Properties

Magnesium nitrate functions as a critical oxidizing precursor in the synthesis process, as it readily supports auto-combustion, accelerates the formation of the MgO phase, and promotes the generation of ultrafine primary particles. Nitrate-based precursors are well known to produce more homogeneous nanocrystalline MgO compared to chloride- or acetate-based salts due to their superior redox balance and decomposition behavior (Hornak, 2021).

Within the sol–gel system, citric acid plays a dual functional role. As a chelating agent, it stabilizes Mg^{2+} ions and facilitates the formation of a homogeneous gel network. Simultaneously, it acts as a fuel that undergoes redox reactions with $Mg(NO_3)_2$, triggering spontaneous combustion. This dual behavior promotes a narrow crystallite size distribution and yields uniformly shaped particles(Gatou, Skylla, et al., 2024; Sharma, Khan, Khanuja, & Mishra, 2025).

Calcination at 800 °C was selected in this experiment because it is highly effective in removing residual organic species and carbon, thereby enhancing structural stability, increasing the degree of crystallinity, and reducing lattice strain in MgO. Although this temperature is sufficiently high to promote the development of a well-defined crystalline phase, it remains moderate enough to prevent

excessive crystallite growth (>40 nm), ensuring that the nanoscale characteristics of the material are optimally preserved.

Implications for Material Functionality and Application Potential

The structural characteristics of the MgO produced in this study indicate strong potential for a wide range of functional applications. In catalytic systems, including transesterification and pollutant degradation, crystallite sizes in the range of 20–25 nm provide high surface basicity, as reported by Mahala et al., (2023) and Gatou, Skylla, et al. (2024). For gas-sensing applications, the well-ordered periclase structure improves signal stability and sensitivity, consistent with the findings Rahmawati Wibowo & Primary Putri (2022). Furthermore, the demonstrated structural stability at 800 °C suggests that the synthesized MgO is also well suited for ceramic and refractory applications that require high thermal resistance.

CONCLUSION

Nano-MgO was successfully synthesized through a sol–gel combustion method utilizing magnesium nitrate as the inorganic precursor and citric acid as both the chelating agent and organic fuel. XRD analysis confirmed the formation of phase-pure cubic periclase MgO ($Fm\bar{3}m$) without detectable secondary phases, indicating effective phase purification through combustion followed by calcination at 800 °C. The crystallite sizes estimated by the Scherrer and Williamson–Hall methods were 16.03 nm and 25.44 nm, respectively, with a low microstrain value (5.37×10^{-4}), suggesting minimal lattice distortion. These results demonstrate that the sol–gel combustion route enables controlled crystallite growth and high structural quality without additional post-treatment. Overall, this method represents a simple, cost-effective, and reproducible approach for producing nanocrystalline MgO with potential applications in catalysis and functional oxide materials.

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