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Investigation of Impact of Calcination Temperature on The Structural, Microstructural, and Functional Properties of MgO Nanoparticles using Williamson-Hall (W-H) Method

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ABSTRACT :

In this study the depletion of magnesium oxide (MgO) nanoparticles was also done by low cost and simple co-precipitation technique and further crystallization was done at two temperatures. The structure of the samples was also systematically studied using the powder X-ray diffraction (XRD) and a quantitative measure of the crystallite size along with the microstrain was measured using the Williamson Hall (W-H) size strain method. The W-H plots demonstrated a definite tendency- the higher the value of the calcification temperature, the lower the value of the microstrain, and the larger the crystallite size increased according to the thermal coarsening and the release of the crystalline defects. The specified behavior is typical of the thermodynamically preferred rearrangement of the crystal structure with the strain reduction that is imposed and the development of the growing crystallites into larger domains. Not only can the result of this work allow achieving a deeper insight into how the calcination process affects microstructural stability of the MgO nanoparticle, but it is also important that calcination is one of the matters that should be considered in the regulation of properties in an attempt to balance the nature of crystallinity, the quantity of defects, and the surface features of the nanoparticle optimally.

Keywords: magnesium oxide, calcination, co-precipitation, XRD, Williamson Hall, crystallite size, microstrain, size strain plot

1. Introduction

Magnesium oxide (MgO) is an all-purpose ceramic oxide and it has been given a fair share of the folly due to its wide variety of application and use in catalysis, adsorption, environmental remedies, refractory products, and energy storing system[1]. Its internal properties high temperature sensitive, good rich thermal, good chemical inertness, rich based surface site[2]. Together with the bulk properties, MgO has highly dependable nanoscale behavior depending on the variation of microstructural parameters (crystallite size, lattice strain and defect density) that can make it stable and react with the surroundings, as well as electronic properties. One of the factors that have been identified to influence these characteristics, as far as the determination of structural and functional characteristics of MgO nanoparticle is concerned, is the temperature of calcification. Structural imperfection and surface influences of crystallite size size are increasingly becoming determinant to nanostructured materials. A small crystal unit and lattice strains, primarily due to dislocations, vacancies and non equivalent distortions on the crystal lattice can explain to a large extent this enlargement of X-ray diffraction (XRD) peaks [3]. The Williamson Hall (W-H) tip is a less complicated though more accurate approach to size broadening in the peak, and the strain broadening in the peak, and therefore, to more-accurate size and microstrain measurements in nano materials. [4].

Higher calcination temperatures would increase diffusion and lattice reorganization, favoring defect relaxation and crystallite coarsening. Moreover, although the co-precipitation method is deemed the most cost-competitive and scale-up able technique to yield high-purity MgO nanoparticles with customizable morphology, only a few studies attempted to relate the calcination temperature to structural properties through W-H analysis. Consequently, the current study fills this research gap by synthesizing MgO nanoparticle through co-precipitation process and subsequent heat treatment at different temperatures ($450^oC, 650^oC, 700^oC,$). The XRD was used to determine the structural and microstructural changes and quantitatively investigated the crystallite size and micro strain using WilliamsonHall(W -H) method. The paper is an attempt to analyze the evolution of microstructural parameters in a systematic manner to gain further insight understanding on how calcination controls the crystallinity and the relaxation of defects in MgO nanoparticles and provide a more realistic framework of controlling their properties in catalytic, environmental as well as electrochemical applications.

2. Methodology

MgO nanoparticles were prepared by a co-precipitation technique using magnesium sulphate heptahydrate ($MgSO_4.7H_2O$) as the source. Magnesium sulphate heptahydrate was then added to ammonium hydroxide (NH_4OH) under stirring to form magnesium hydroxide precipitates. The precipitate was then filtered, comprehensively washed with deionized water in order to eliminate unadulterated ions and dried. The resulting precursor was then calcined

in air at $450^oC, 650^oC, 700^oC$ temperatures under the conditions of 24 h to obtain crystalline MgO nanoparticle. Structural characterization was performed by the X-ray diffraction (XRD) with Cu K alpha [3].

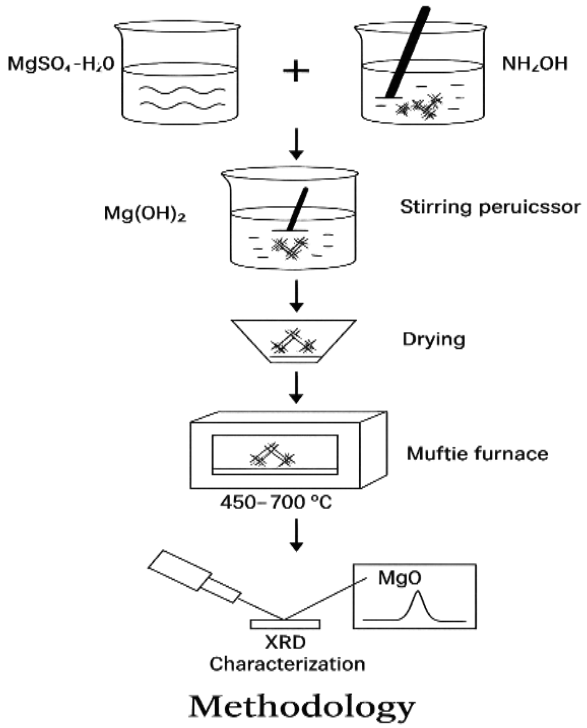

Fig. 1 - Methodology

3. Equations

The Williamson–Hall method is employed to evaluate both crystallite size (D) and microstrain (ε) contributions to peak broadening in X-ray diffraction (XRD) analysis. The squared form of the W–H equation is expressed as:

$$(\boldsymbol{\beta \sin \theta})^2 = \left(\frac{k\lambda}{D}\right)^2 + 16\varepsilon^2 sin^2\theta \quad \textbf{(1)}$$

4. Result and Discussions

The Williamson–Hall squared plots for MgO calcined at different temperatures are shown in *Figure 1*. At 450 °C, the plot exhibits the highest slope and intercept, indicating significant microstrain and smaller crystallite domains. Increasing the temperature to 650 °C reduces the slope and increases the intercept moderately, showing relaxation of strain accompanied by particle coarsening. At 700 °C, both slope and intercept values are minimized, confirming substantial crystallite growth with reduced lattice strain. These observations align with reported studies where higher calcination temperatures lead to improved crystallinity and reduced defect density in MgO nanoparticles [5].The decrease in microstrain suggests that dislocations and lattice imperfections are progressively annealed out, while the growth in crystallite size is a direct consequence of thermally activated grain coalescence [4]. Functionally, such evolution implies a trade-off: higher crystallinity benefits dielectric stability and catalytic site uniformity, whereas excessive growth may decrease surface area important for adsorption and surface-driven reactions.

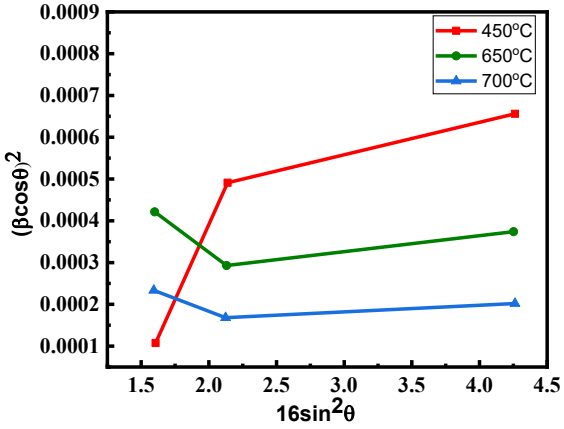

Figure 2. Williamson–Hall squared plots of MgO nanoparticles calcined at 450 °C, 650 °C, and 700 °C, demonstrating the effect of calcination on microstructural evolution.

4.1 Tables

The table highlights that increasing calcination temperature improves MgO crystallinity: 450 °C shows broad peaks with high strain, 650 °C indicates partial strain relaxation with coarsening, and 700 °C yields sharp peaks with well-crystallized, low-strain nanoparticles.

Table 1 – Effect of calcination temperature on XRD peak broadening and corresponding microstructural features of MgO nanoparticles.

Calcination Temperature (°C)	XRD Peak Broadening Trend	Microstructural Implication
450	Broad peaks	High defect density, strained lattice
650	Narrower peaks	Partial strain relaxation, coarsening begins
700	Sharpest peaks	Well-crystallized, minimal strain

5.Conclusion

This investigation systematically demonstrates the critical role of calcination temperature in defining the structural and microstructural properties of MgO nanoparticles synthesized via the co-precipitation method. XRD analysis combined with Williamson–Hall (W–H) evaluation confirmed that higher calcination temperatures progressively decreased lattice strain while enhancing crystallite size, evidencing thermally driven defect relaxation and grain growth. The observed trend highlights a balance between crystallinity and surface-related features, where lower temperatures preserve higher defect density and surface activity, while elevated temperatures improve lattice stability and reduce imperfections. Such insights establish a clear structure–property relationship, offering practical guidance for tailoring MgO nanoparticles toward specific applications in catalysis, adsorption, and energy-related technologies. Importantly, the present study underscores the effectiveness of W–H analysis as a simple yet reliable tool for decoupling strain and size effects, thereby contributing to a more rational design of functional oxide nanomaterials for industrial and environmental applications.

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