



Investigating the Effect of Calcination on Physical Properties of Magnesium Oxide Nanoparticles

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ABSTRACT

In this concise study Magnesium oxide (MgO) nanoparticles are ideal for a variety of uses in adsorption, refractory materials, and catalysis because of their exceptional qualities, which include large surface area, thermal stability, and catalytic activity. One of the key processes involved in the calcination heat treatment process of magnesium oxide nanoparticles includes the setting up of crystal structure, surface area, particle size as well as thermal stability of the final product. This paper focuses on how the process of calcination influences MgO nanoparticles and highlights the manner in which it influences a number of characteristics which are rather vital for various commercial and environmental uses. Calcination of MgO nanoparticles favors their use in high-temperature and catalytic applications by enhancing shape and size distribution, as well as the crystalline nature and thermal stability of the particles. In addition, calcination can change the mechanical featured and adsorption capacity of magnesium oxide nanoparticles, increasing its versatility.

Key words; Calcination; Magnesium oxide nano particles; Thermal treatment; Crystallite size; Surface area.

Introduction :

Magnesium oxide (MgO) nanoparticles are of recent interest among materials scientists and engineers as a consequence of their physical and chemical characteristics that enable them to be used as catalysts [1, 2, 3], refractory materials and absorption materials. The particles described here are on the nanoscale [4], which endows them with large surface areas and increased reactivity relative to their large-scale counterparts[5]. Since the use of MgO nanoparticles in the mentioned applications depends on the properties of the nanoparticles which need to be defined and controlled, it is highly important to understand and control the properties of MgO nanoparticles [1]. In the present work, calcination [6, 7, 8], the thermal treatment method, has been found to highly influence the features of MgO nanoparticles. This process can be done in air or oxygen and it aims at decomposing the precursors and converting the material to the oxide by heating it to a particular temperature. The manner in which calcination is done depending on the temperature and time that is used, influences some of the properties of the nanoparticles such as the crystallite size, surface area and even the shape. These changes in the physical properties are important as the change in the reactivity and the functionality of the material is affected [9, 10, 11]. Despite the established importance of calcination, there is still a need for a systematic investigation into how varying calcination conditions affect the physical properties of MgO nanoparticles. While some studies have explored these aspects, comprehensive research that correlates specific calcination parameters with the resulting structural and morphological characteristics of the nanoparticles is limited. Such a study would provide valuable insights for tailoring MgO nanoparticles for specific industrial applications. The purpose of this study is to establish the impact that calcination has on some properties of MgO nanoparticles which include crystallite size, surface area and shape[12]. It is specifically in this context that the present work has attempted the systematic exploration of the available thermal treatment conditions namely calcination temperature and time, so as to arrive at a more accurate correlation between the thermal treatment conditions and the properties of the derived nanoparticles. This study's results should help in revealing the thermal properties of MgO nanoparticles and provide recommendations on their synthesis. Therefore, the outcomes of this research will be of huge impact in the preparation and usage of MgO nanoparticles. Through a systematic study of the effects of calcination in this research, it will be possible to build a bench-marking for the controlled synthesis of MgO nanoparticles of desired characteristics, thus improving their efficiency in their intended uses. More importantly, these findings could be applied to other metal oxide nanoparticles making it possible to share generality in the science and engineering of nanomaterials.

Experimental Procedure

MgO Synthesis

During a typical synthesis, 100 milliliter's of deionized water, 0.1M of magnesium sulphate heptahydrate (MgSO₄·7H₂O) was made [13, 14, 15]. The mixture was then refluxed at the same temperature for approximately 12 hours after 0.25 to 0.5 M of ammonia solution (NH₄OH) was added dropwise and forcefully to the aforesaid solution at 100C. The resulting white product after complete reaction was then separated through washings with deionized water as this removed all the by-products and lastly the solid was further dried in air at 120 °C for next 12 h. MgO nanoparticles with a particle size in the range of 50–60 nm were obtained by calcining the as-synthesized products at 450°C and 600°C for two hours in air. In this case, MgO nanoparticles designated "A" and "B" were produced for concentrations of 0.25 M and 0.5 M NH₄OH, respectively.



Figure 1. Preparation of MgO nanoparticles

Characterization :

X-Ray Diffraction Analysis. We find that calcination temperature has a profound effect on both crystallinity and crystallite size of MgO nanoparticles by analyzing the X-ray diffraction pattern of MgO nanoparticles calcined at 450°C, 600°C, and 700°C. The diffraction pattern recorded at 450°C indicates a lower crystallite size, averaging between 10 and 20 nm, and lower crystallinity as indicated by broader peaks; major peaks correspond to the (200), (220), and (222) planes of the cubic MgO phase. When calcination temperature increases to 600°C the peaks become more intense and narrow indicating better crystallinity and a little-enhanced crystallite size of 20-30 nm, phase purity is excellent with no signs of impurities. Among all the sintering temperatures, it has been found that the peaks of MgO are most intense at 700°C signifying that there are well developed crystalline structures and further, an increased crystallite size of MgO (30–40 nm) as sintering progresses at higher temperatures. Figure 2 shows XRD results for the successful formation of the MgO nanoparticles. The detected primary reflections at $2\theta = 42.80^\circ$, 62.08° , and 78.45° , which correspond to the (2 0 0) and (2 2 0) planes of MgO with face-centered cubic structure, respectively, are in good agreement with the standard JCPDS card (No: 78-0430). The synthetic material's strong crystallinity is shown by the XRD pattern's high intensity (2 0 0) orientation peak. The XRD pattern showed no distinctive Mg (OH)₂ or Mg or other impurity peaks, demonstrating the high quality of the produced products Using (2 0 0) reflection. In line with this, the outcomes presented here confirm that calcination temperature is a determinant of the physical characteristics of MgO nanoparticles. An increase in temperature leads to improved crystallinity and increased crystallite size and the best crystalline structure was found at 700 °C. Nevertheless, it is possible to obtain materials with increased porosity or undesirable grain growth or sintering processes, which may- change the properties of nanoparticles at higher temperature. This work has validated that MgO nanoparticle properties can be tailored based on specific application requirements through fine-tuning of calcination conditions.

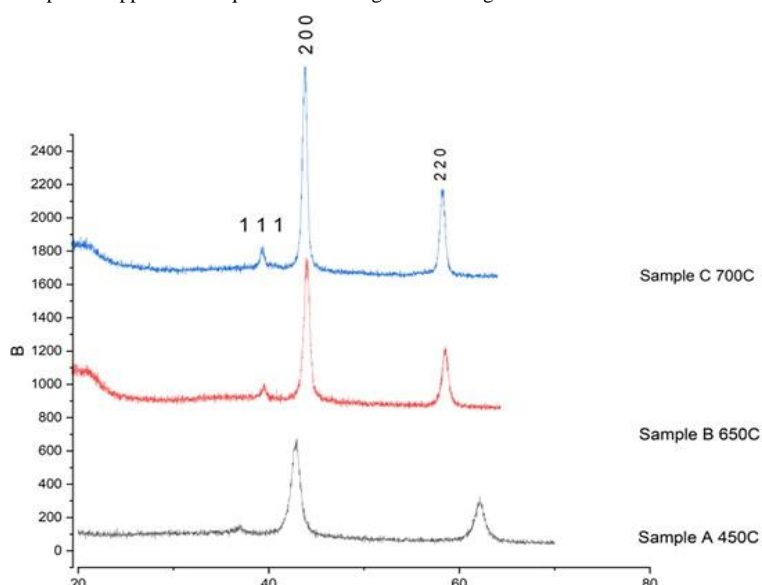


FIGURE 2. XRD graph for MgO nanoparticles

Fourier Transform infra-red Analysis. Fourier Transform Infrared (FTIR) analysis of MgO nanoparticles calcined at 450°C, 600°C, and 700°C helps to identify functional groups and verify phase changes resulting from different calcination temperatures. Across all temperatures, FTIR spectra typically show a broad absorption band around 3430 cm⁻¹ due to O-H stretching, attributed to adsorbed water or surface hydroxyl groups. This band's intensity generally decreases as temperature increases, indicating effective removal of surface hydroxyls with higher calcination. The characteristic Mg–O stretching vibrations appear around 500–600

cm⁻¹ and become more pronounced as calcination temperature rises, particularly at 700°C, reflecting enhanced MgO crystallinity and phase purity. Higher temperatures yield clearer Mg–O peaks due to the reduced presence of impurities or unreacted precursors, confirming that calcination at elevated temperatures supports the formation of high-purity MgO with minimal surface contamination. Those presented by sample A (gray) have the lowest value of transmittance and thus can be associated with either the lower degree of crystallinity or larger number of surface imperfections, such as impurities. The FTIR spectra of three MgO nanoparticle samples (A, B, and C) subjected to varying calcination conditions are presented in the figure. The x-axis denotes the wavenumber (cm⁻¹), while the y-axis represents transmittance %.

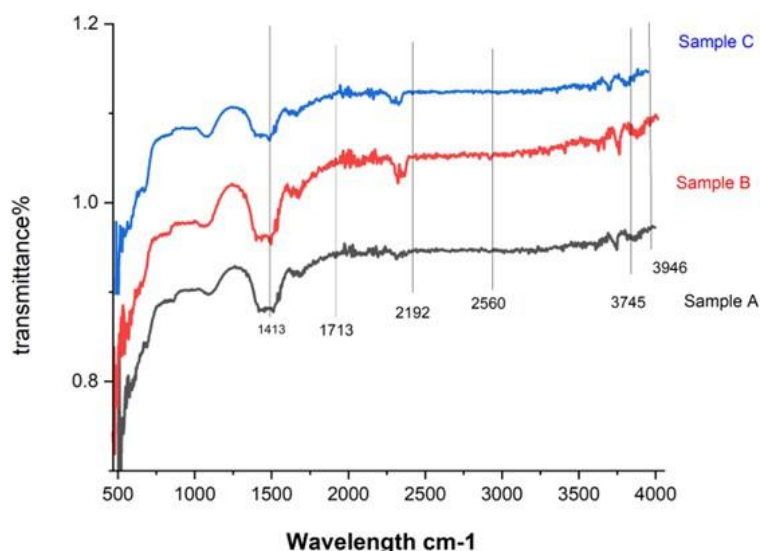


FIGURE 3. Ftir Spectra of MgO nanoparticles

Sample B (red) exhibits intermediate transmittance and can be assigned to the presence of certain surface groups or adsorbents, as indicated by absorption bands at 1413 cm⁻¹ and 1713 cm⁻¹. Sample C (blue) shows the highest transmittance, likely due to increased crystallinity or purity. Sample A (gray) has the lowest transmittance, suggesting a lower degree of crystallinity or higher surface impurity levels. Key peaks at 2192 cm⁻¹, 2560 cm⁻¹, 3745 cm⁻¹, and 3946 cm⁻¹ correspond to specific structural characteristics influenced by calcination. Among Samples C (blue), the one with the highest transmittance at most wavelengths could be attributed to higher crystallinity or purity resulting from its calcination process.

Conclusion :

Consequently, coupled with results of analyze of causation of calcination on MgO nanoparticles physical properties at 450°C, 600°C and 700°C it is founded that there are changes in the aspect of crystalline structure, size of crystallites and phase purity. The increase of calcination temperature led to a significant improvement of the crystalline structure of MgO nanoparticles as well as its size, and the most suitable one at 700°C where it exhibits well-defined peaks of nanocrystal line MgO in XRD patterns and confirmed high purity of synthesized MgO by FTIR analysis. Decreasing the concentration of hydroxyl groups and the appearance of clear Mg–O stretching vibrations at higher temperatures also imply efficient elimination of impurities that lend evidence to the generation of high quality MgO nanoparticles. These results suggest that calcination temperature should be strictly regulated when using MgO nanoparticles for catalysis, electronics and environmental applications, as their physical characteristics depend on this parameter.

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