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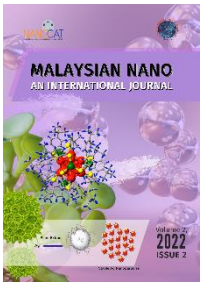

Research Article

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Synthesis and characterization of magnesium hydroxide nanoparticles via sol-gel

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Abstract

The magnesium hydroxide (Mg $(OH)_2$ nanoparticles (NPs) were prepared using the sol-gel method and presented its results in this work. Various characterization methods have been used to confirm the successful preparation of Mg $(OH)_2$ NPs. The XRD analysis exhibited the hexagonal structure of the sample. FT-IR spectrum revealed the presence of metal-oxygen stretching of prepared magnesium hydroxide NPs. The SEM analysis shows that the synthesized Mg $(OH)_2$ NPs were clustered with a rough stacked surface. From the absorption peak obtained through UV-Vis spectroscopy, the bandgap energy of Mg $(OH)_2$ NPs was calculated as 2.70 eV.

Keywords*:* Magnesium hydroxide; Sol-gel synthesis; Nanoparticles; Optical properties

1. Introduction

Magnesium hydroxide ($Mg(OH)_2$) has thermal stability with a non-toxic and non-corrosive nature and environmentally usable friendly flame retardant that suppresses fumes during a fire by going through endothermic dehydration [1]. Moreover, compared to the most popular aluminium trihydrate fillers, magnesium hydroxide can be processed at higher temperatures [1, 2]. Magnesium hydroxide powders have gained popularity over the past ten years as additives for the production of flame-retardant thermoplastics [1-3]. Due to their numerous applications, magnesium hydroxide nanoparticles have generated considerable interest [2-5]. Additionally, it is utilized in drugs as an inactive substance [6], in waster water treatment to make the acid chemically neutral and in gases with high amounts of sulfuric oxides [7]. Also, porous $Mg(OH)_2$ nanoparticles can act as a capable catalytic reagent [8]. Additionally, the production of magnesium oxide is frequently started with magnesium hydroxide as a precursor [9].

Numerous processes, including the hydrothermal route [10], the solvothermal reaction [11], precipitation [12], water-in-oil microemulsions [13], electrolysis of an aqueous magnesium salt solution [14], sol–gel technique [15], and microwave-assisted synthesis [16] can be used to create magnesium hydroxide nanostructures. The sol-gel process is used in the synthesis of ceramic oxides due to its flexibility, fast process, and affordable among many other techniques. This method is a versatile wet chemical method for synthesizing various materials, specifically metal oxides. Therefore, in this work, Mg $(OH)_2$) NPs have formed by the sol-gel method, and their optical, structural, morphological, and vibrational characteristics were carefully examined.

2. Experimental

2.1 Synthesis of magnesium hydroxide nanoparticles

The magnesium nitrate hexahydrate of wt 5.21 gm (0.2M) was dissolved in 200 ml deionized water by constant stirring for 30 min. The 0.8 gm (0.2M) of NaOH solution was added dropwise to Mg $(NO_3)2.6\ H_2O$ solution using a glass rod. Subsequently, the solution was magnetically stirred for 2h and the solution was undisturbed for the precipitate formation in 2h. The final product was obtained by filtering and washing the precipitate with deionized water and ethanol. The final product was kept in a vacuum oven at 80 $^{\circ}C$ to dry the products and remove moisture. The dried powders were then crushed using a mortar and made into a very fine powder.

2.2 Characterization

The sample was analyzed by x-ray diffraction (XRD, Rigaku D/max-2500, Cu Ka radiation, λ=0.154056 nm) to identify the crystal structure. The morphology and microstructure were determined

using a JEOL JEM-7800 scanning electron microscope (SEM). The functional modes and the vibrational frequencies present in the sample were obtained through Fourier-transform infrared spectra using Perkin Elmer (spectrum two) in the range 400 cm-1 to 4000 cm-1. UV-Visible absorbance spectra were observed in the region of 200 nm to 800 nm on a Shimadzu UV-1800 spectrometer.

3. Results and discussion

The crystalline phase of the magnesium hydroxide was determined by X-ray diffraction patterns. Figure 1 indicates the XRD pattern of Mg $(OH)_2$ NPs. The observed diffraction pattern for the synthesized sample corresponds to the hexagonal structure with the P-3m1 space group. The diffraction peaks are (011) (100) (101) (102) (110) (111) (103) (200) and (201) are perfectly in accordance with (JPDF#0-0239) and no diffraction peaks of other impurity were found. The sharp and narrow peaks with high intensity illustrate the crystalline nature of the Mg $(OH)_2$ NPs. It is worth noting that no additional peaks are seen. This demonstrated that the synthesized sample was of high purity.

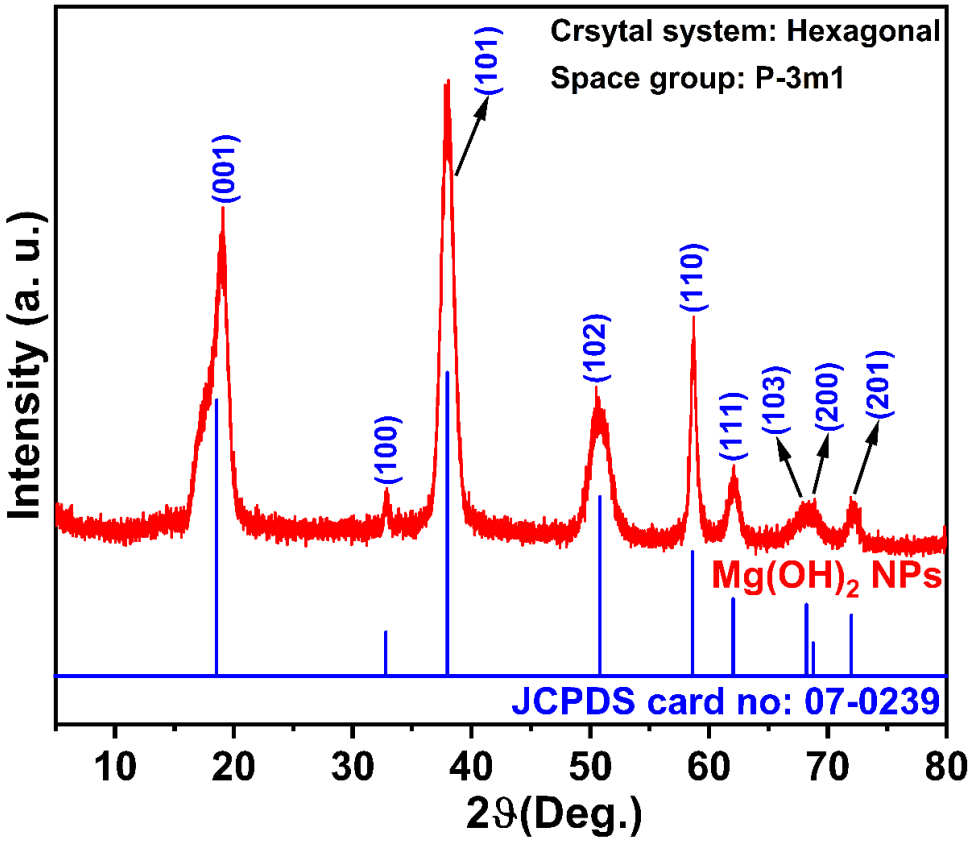

Figure 1: Powder XRD pattern of Mg $(OH)_2$ NPs

The FTIR spectrum of the pristine Mg $(OH)_2$ NPs was examined in the range of 400–4000 cm^{-1}, as shown in Figure 3. The formation of metal oxides was supported by the FT-IR spectrum, and the vibrational modes of the as-synthesized Mg $(OH)_2$ NPs were investigated. The wide-ranging peak at 3446 cm^{-1} corresponds to the O – H stretching vibration of water molecules. The absorption peaks at 1490 cm^{-1} and 1425 cm^{-1} are attributed to C =O carbonyl stretching and H – O – H bending vibrations, respectively. The smaller peak at 1372 cm^{-1} is due to N – O stretching. The peak at 501.49 cm^{-1} demonstrated the formation of metal-oxide bonds, which affirmed the formation of Mg-O-H.

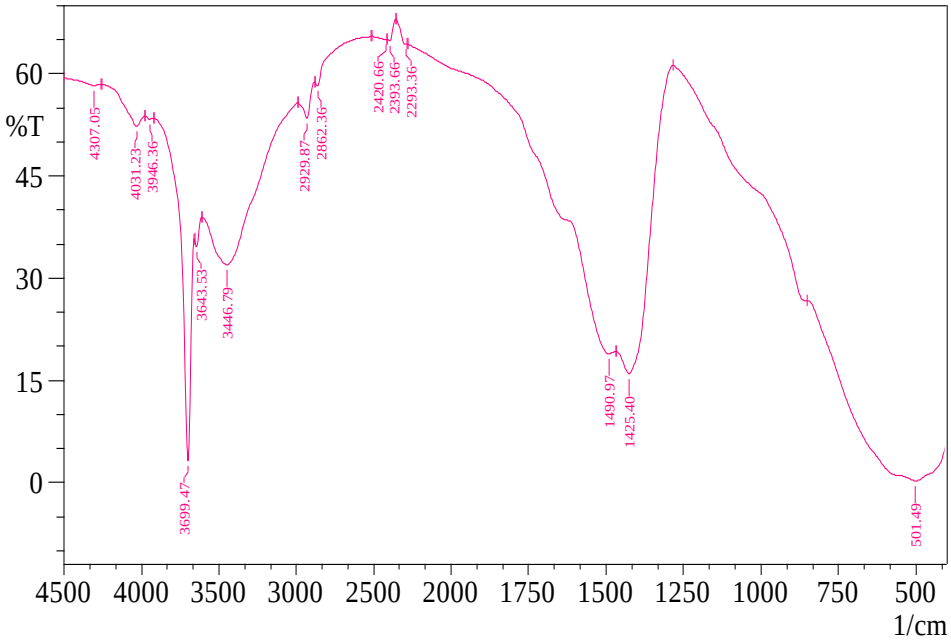

Figure 2: FTIR spectrum of Mg $(OH)_2$ NPs

Figure 3 (a-d) shows the SEM images of the as-prepared Mg $(OH)_2$ NPs at different magnifications in the range of 2 µm-30 nm. This shows that there is the aggregation or overlapping of smaller-sized Mg $(OH)_2$ particles to generate larger particles and also the grains are observed as distributed randomly and have rough stacked surface shapes.

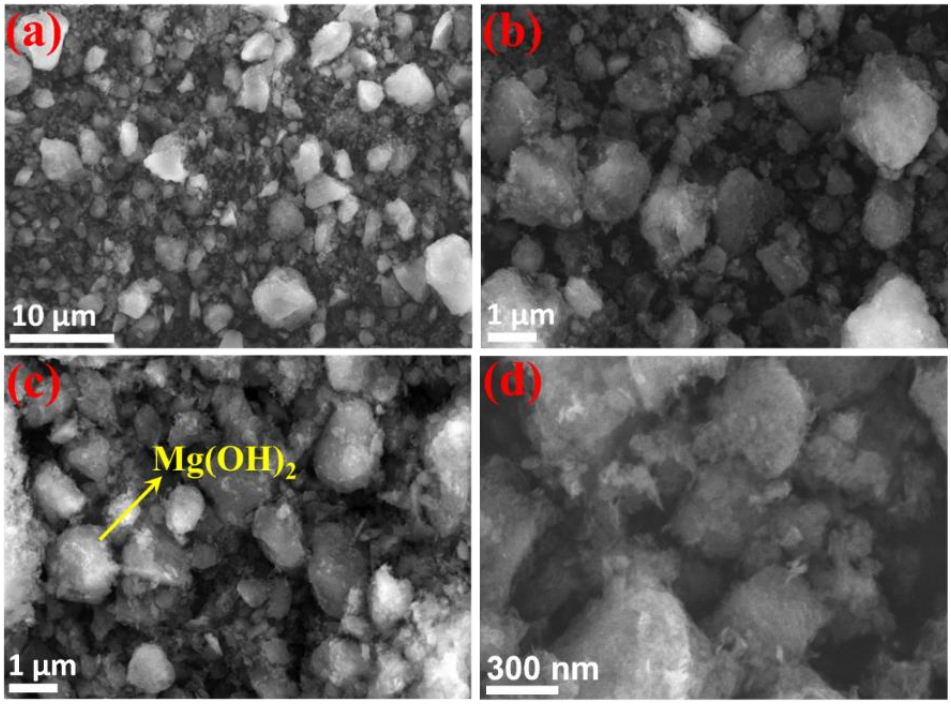

Figure 3: SEM images of Mg $(OH)_2$ NPs

The UV-visible absorption spectra of the Mg $(OH)_2$ NPs are displayed in Figure 4. The strong absorption spectra were analyzed in the range of 200 – 400 nm. The absorption peaks of the as-synthesized Mg $(OH)_2$ NPs observed at 215 nm are in the ultraviolet region. The plot of a graph between $(\alpha h\upsilon)1/2$ and $h\upsilon$, and then extrapolating the line drawn tangent to the resultant curve gives the value of the band gap energy, as shown in the insert of Figure 4. The bandgap of the magnesium hydroxide was estimated to be 2.70 eV.

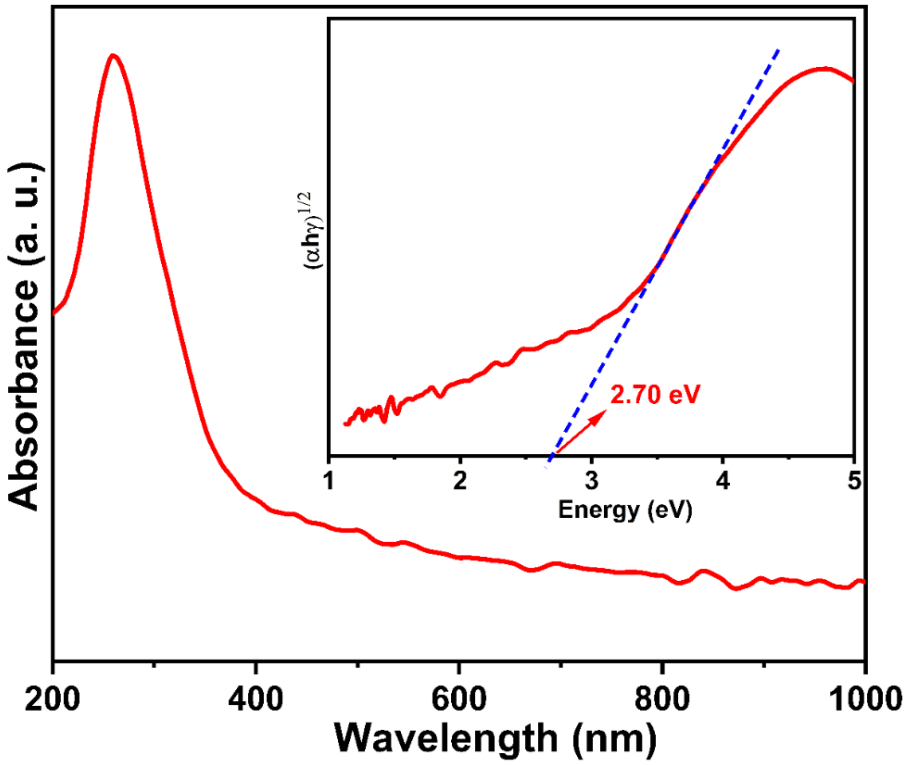

Figure 4: UV spectrum of Mg $(OH)_2$ NPs (Insert. Tauc plot)

4. Conclusion

In summary, magnesium hydroxide ([Mg $(OH)_2$] nanoparticles (NPs) were prepared by a sol-gel route and characterized thoroughly to study their crystalline structure, structure and surface morphology, and optical properties. Powder XRD analysis showed that the Mg $(OH)_2$ had a hexagonal structure with its crystalline nature. SEM analysis indicated that the rough stacked surface morphology of the particles was aggregated. FTIR analysis demonstrated the functional group present in Mg $(OH)_2$ NPs. A strong absorbance peak at 215 nm and the calculated energy band gap of 2.70 eV was observed in the Tauc plot.

Conflicts of Interest

The authors declare no conflict of interest.

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