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Structural and Optical properties of Magnesium Oxide Synthesized by Chemical Precipitation Method

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Abstract

Cubic shaped Magnesium oxide were successfully synthesized by Chemical Co-precipitation Method by using magnesium chloride hexahydrate and sodium hydroxide at room temperature. Hydrated magnesium oxide were calcinated at 500˚C and 600˚C. A systematic study of the crystallization behavior and structural properties of calcinated magnesium oxide nanopaticles were carried out by thermogravimetric analysis, x-ray diffraction and fourier transform inferred spectroscopy. X-ray diffraction patterns indicated that nanoparticles are good crystallinity, pure MgO phase with (2 0 0) orientation. From XRD results the temperature was gradually increase the crystallite size of MgO was increased. The FTIR results indicated the formation of magnesium oxide.

Keywords: FTIR, magnesium oxide nanoparticle, SEM,TG-DTA, UV-Vis, X ray diffraction

1. INTRODUCTION

At the recent year, nanotechnology has been leading to a technological revolution in the world.[1] Nanoscience is the study and application of extremely small things. Several metal oxide nanoparticles exhibit unique physical and chemical properties that are very important in various application in variety of field.[2-4] The properties of nanocrystalline metal oxide materials were high strength with good thermal conductivity, high damping property and mechanical stability.[5-8] In this research work, the magnesium oxide nanoparticles was synthesized by chemical deposition method from $MgCL_2.6H_2O$ and NaOH. The structural properties of MgO nanopaticles were characherized by Fourier Transform inferred Spectroscopy and Thermogravimetic analysis. Thermogravimetric analysis was performed in the temperature range of 30˚C-1000˚C using DTG-60/60H SIMULTANEOUS DTA-TG ANALYZER. The MgO nanopaticles were analyzed by x-ray diffraction analysis using RIGKU MiniFlex 600 diffractometer with Cu-K_α radiation (λ =1.54060Å). Fourier Transform inferred spectra were recorded on a Thermo Scientific Nicolet iS10.

2. *Experimental procedure*

For the synthesis of nanostructured MgO at room temperature, chemical co-precipitation method is employed. In this method 1.5 M of magnesium chloride and 1.5 M of sodium hydroxide were dissolved in 100 ml of deionized water into a beaker separately. Stir them separately for half an hour using magnetic stirrer for constant stirring to yield a clear and homogeneous solution. Then, add sodium hydroxide drop by drop with bruette at room temperature for 1 hour and 30 minutes with constant stirring. After 30 minutes the color turned milky white showing the precipitation of MgO. Then the precipitate was several times washed with deionized water and filtered in filter paper. The sol-gel was dried at 110˚C for 11 hours. The dried power was calcinated at 500˚C and 600˚C for 2 hr in furnace.[9]

3. *Results and Discussion*

Chemical Reaction: $MgCL_2$ dissolved in deionized water, $MgCL_2 + H_2O \rightarrow Mg^{2+} + 2CL$

NaOH dissolved in deionized water,

$$NaOH + H_2O \rightarrow Na^{+} + OH^{-}$$

When NaOH is added to MgCL2, Mg (OH)2 was formed

$Mg^{2+} + OH^{-} \rightarrow Mg(OH)_2$

$MgCL_2 + NaOH \rightarrow Mg(OH)_2 + 2NaCL$

As prepared Mg $(OH)_2$ was further oxidized at 345.51˚C to get MgO as;

$$Mg(OH)_2 \rightarrow MgO + H_2O$$

3.1 Thermogravative Analysis

TG/DTA is a simultaneous thermal analyzer that can characterize multiple thermal properties of a sample in a single experiment. The TG component measures temperatures where decomposition, reduction or oxidation occurs. It simultaneously measures the weight changes associated with decomposition, oxidation, and any other physical or chemical changes that result in sample weight loss or gain. The DTA component shows whether decomposition processes are endothermic or exothermic. The DTA also measures temperatures corresponding to phase changes where no mass loss occurs, such as melting, crystallization and glass transitions. The thermogravimetric and derivative thermogravimetric analysis of dry MgO powder prepared at 110˚C is shown in Fig.3. According to the thermogravimetric results, the primary weight loss occurred at 64.65˚C and the secondary weight loss occurred at 345.51˚C. Phase transition formed to occur at about 345.51˚C indicating the transition from magnesium hydroxide to magnesium oxide with weight loss 36.881%. According to the result of DTA graphthe endothermic peak 64.65˚C to dehydration of adsorption water. The endothermic peak of 345.51˚Cdehydrate from OH of crystal structure and is observed MgO material.

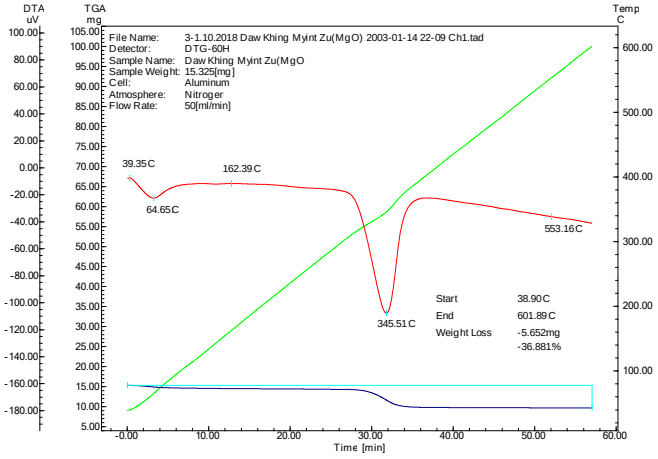


Fig. 1 Thermogravimetric spectrum of MgO at 110˚C

3.2 *XRD analysis*

Fig. 2 (a) showed the x-ray diffraction pattern of calcinated MgO powder at 500˚C and (b) showed the x-ray diffraction pattern of calcinated MgO powder at 600˚C. Table 1.1 showed FWHM and crystallite size of MgO powder at 500˚C & 600˚C. In different temperature lattice parameters were the same values. The crystallite size was determined using Scherrer's equation,

$$D = \frac{K\lambda}{\beta \cos\theta}$$

Where, D is the crystallite size

K = Scherrer constant (0.899)

β = Full width half maximum

λ = wavelength of x ray (1.54056 Å)

θ = Bragg diffraction angle

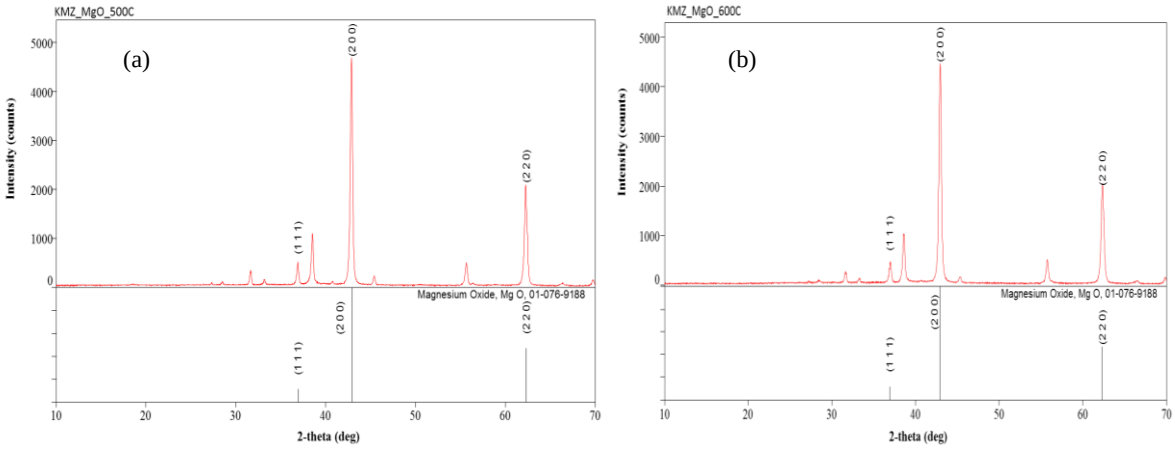


Fig. 2 x-ray diffraction pattern of calcinated MgO powder (a) at 500˚C (b) 600˚C

Table 1.1 Parameter and crystallite size of Calciated MgO at 500˚C and 600 ˚C

N0	Temperature(˚C)	Parameter (Å)	shape	Crystallite size (nm)
1	500	a = 4.2104 b = 4.2104 c = 4.2104	cubic	33.24
2	600	a = 4.2104 b = 4.2104 c = 4.2104	cubic	35.11

3.3 *FTIR analysis*

The composition of the sample was analyzed by FTIR measurement. Fig 5(a) showed the FTIR spectrum of MgO at 500˚C, and (b) showed the FTIR spectrum of MgO at 600˚C.At these two different temperature Ir broad peak at around 3854.11 cm^{-1},3751.70 cm^{-1} , 3699.35 cm^{-1} 3676.10 cm^{-1} 3422.10 cm^{-1} ,1437.66 cm^{-1} and 862.54 cm^{-1}. The broad above 3600 cm^{-1} stretching mode of vibration in Hydroxyl (O-H) group . The broad between 3600 cm^{-1} -3330 cm^{-1} and 1700 cm^{-1} – 1400 cm^{-1} have been attributed to stretching mode of – OH group. Peak at 864 cm^{-1} were attributed to different Mg-O-Mg vibration modes of MgO.

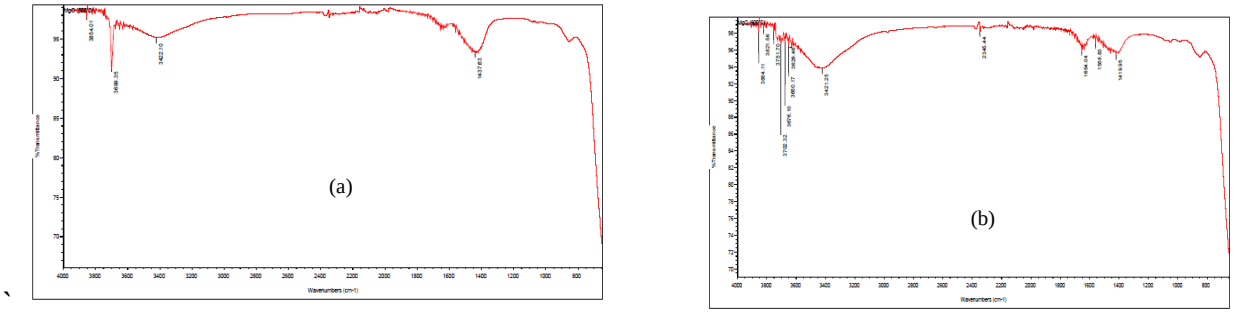


Fig. 3 FTIR spectrum of calcinated MgO at (a) 500˚C (b) 600 ˚C

3.4 *UV-Vis Analysis*

The optical properties of MgO powder were determined from the absorbance measurement in the range of 190 nm and 1100 nm UV-vis sopectrometer. Fig 4(a) showed the absorption spectrum of calcinated MgO at 500˚C and (b) showed absorption spectrum of calcinated MgO at 600˚. The evaluation of the optical band gap of the coleus using UV-Vis ansorbance spectra was done by using Tauc relation. The optical band gap were

determined by extrapolation of the linear region to zero absorbance from the plot that was drawn according to the Tauc relation when $n = \frac{1}{2}$.[8]

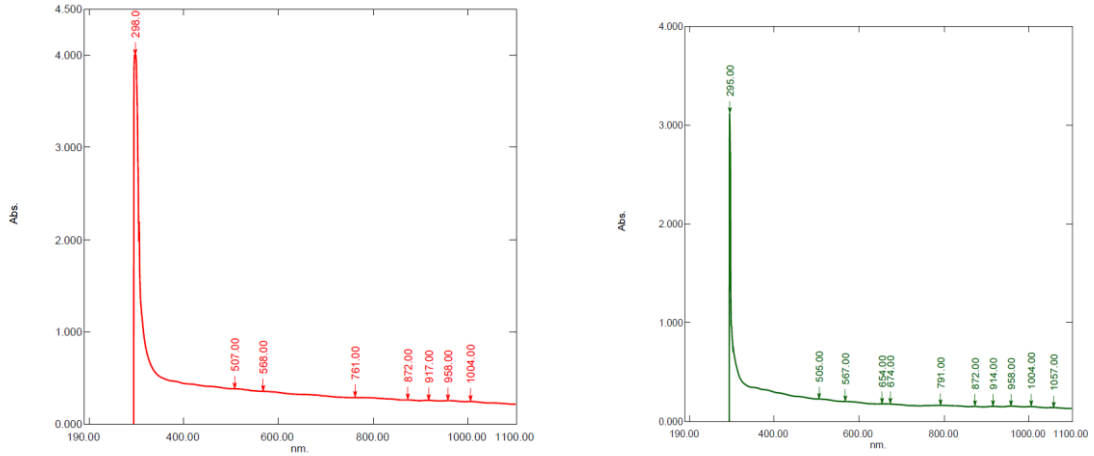
$$\alpha h\nu = A(h\nu - E_g)^n$$

Where, α = absorption coefficient (α = 2.303 A= thickness)

E_g = Energy Bandgap of the material

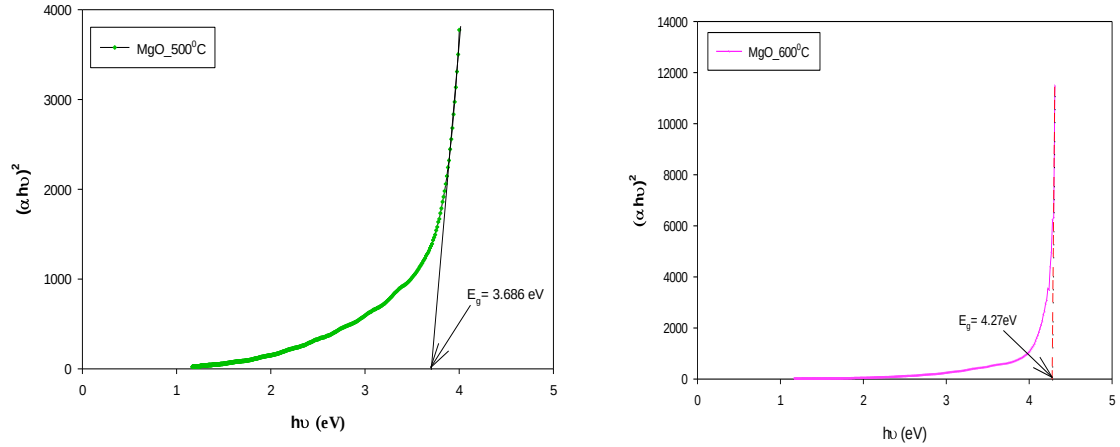
n = order of transition (1/2,3/2,2,3)

The change in $(\alpha h\nu)^2$ as a function of photoenergy (hν) of MgO calcinated at different temperatures (500˚C and 600 ˚C) was plotted and given as Fig 5 (a) and (b). The optical band gap energies of MgO are 3.686 eV at 500˚C and 4.27 eV at 600˚C.



(a) (b)

Fig 4 Obsorption spectra of calcinated MgO at (a) 500˚C (b) 600 ˚C



(a) (b)

Fig. 5 Tauc plot of MgO nanoparticles calcinated at (a) 500 ˚C and (b) 600 ˚C

3.5 SEM analysis

Fig 5 showed the SEM image of MgO nanoparticles calcinated at 500 ˚C. SEM showed the general view of the morphology of calcinated nanoparticles. If have been observed that all the synthesized nanoparticles are agglomerated in nature and spherical shaped.

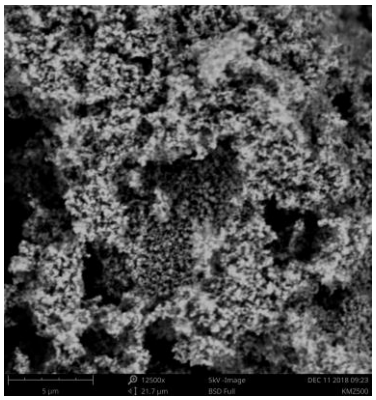


Fig. 5 SEM image of MgO nanoparticel at 500˚C for 2 hrs

4. CONCLUSIONS

Cubic shaped nanoparticles of MgO have been synthesized by chemical Co-precipitation method. Thermogravimetric analysis indicated transition from MgOH to MgO at 345˚C. From X-ray diffraction results, MgO powder showed crystalline structure with preferred orientation of (2 0 0) and the particle size was calculated as 33nm at 500˚C. FTIR spectra of MgO nano particles calcinated at 500˚C,600˚C of the IR peaks bond around 3699cm^{-1}, 1437 cm^{-1}and 862.54 cm^{-1}.These peaks were attributed to stretching mode of O-H group and Mg-O-Mg vibration MgO nanoparticles respectively. From UV-vis spectroscopy the optical band gap energies of MgO were 3.686 eV and 4.27 eV.This study leads to the conclusion, Chemical Co-precipitation method was simple and cost-effective preparation techniques and it can be manufactured with ease and simplicity of the bulk of MgO. And then pure MgO nanoparticles were suitable to use as the photoelectrode in DSSC.

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