Surfactant Free Synthesis of Magnesium Oxide Nanotubes by Simple Chemical Co-Precipitation Method

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Abstract: A simple cost effective preparation of Magnesium oxide nanoparticles in nanotube morphology is reported using Chemical co-precipitation method. As prepared magnesium oxide nanoparticles were characterized using UV-visible spectroscopy, X-ray Diffraction, Field Emission Scanning Electron Microscope and Energy dispersive X-ray spectroscopy. As prepared magnesium oxide nanoparticles were found in nanotube morphology whose inner and outer diameter were 31 nm and 78 nm. The band gap of as prepared nanotubes were found to be 5.37eV with maximum absorbance at 200 nm.

Keywords: Chemical co-precipitation method, magnesium oxide nanoparticles, nanotube morphology.

I. INTRODUCTION

In recent years, Magnesium oxide nanoparticle is of great interest among wide band gap metal oxides for the researchers. Because of their peculiar properties, magnesium oxide nanoparticles are being used in many applications like solar cells [1]-[3], sensors [4]-[6], catalysts [7]-[9], antibacterial or antipathogenic applications [10]-[12] and environmental remediation applications [13]-[14] etc. The performance of nanoparticles in all these applications depends mainly on their size, morphology and structure [15]-[16]. Nanstructured magnesium oxide can be synthesized in different morphologies like spherical [10], nanorod [17], nanoflake [9], hierarchical nanostructure etc [6]. There are many methods to synthesize magnesium oxide nanoparticles like hydrothermal method [14], chemical method [17], sol-gel method [18], combustion method [13] and electrospinning method [15] etc. Magnesium oxide nanoparticles have also been synthesized by Green route using neem leaves [19]. For synthesis of Magnesium oxide nanoparticles, Magnesium nitrate (Mg(NO3)2) [5], [7], Magnesium acetate (Mg(CH3COO)2) [8] Magnesium sulfate heptahydrated (MgSO4.7H2O) [20] and Magnesium chloride (MgCl2.6H2O) [6], [9] etc are being used as precursors.

In the present work we have synthesized magnesium oxide nanotubes by Chemical Co-precipitation method and studied their optical, structural and morphological properties. Chemical Co-precipitation method is easy and cost-effective method for synthesis.

II. EXPERIMENTAL DETAILS

A. Materials/Chemicals Used

Magnesium acetate tetrahydrate Pure ((CH3COO)2Mg.4H2O) (Merck), Sodium Hydroxide (NaOH) (Qualigens), Ethanol (Merck) and Distilled water (Merck) were used with no further purification

B. Synthesis of Magnesium Oxide Nanoparticles

Magnesium oxide nanotubes were synthesized by simple Chemical Co-precipitation method. Two separate homogeneous aqueous solution of Magnesium acetate (0.2 M, 50 mL) and Sodium hydroxide (0.4 M, 50 mL) were prepared at constant stirring. Prepared aqueous solution of sodium hydroxide was mixed drop wise with aqueous solution of magnesium acetate at constant stirring. The colour of the solution turned to milky white from transparent. The mixture was stirred at constant stirring for 3 hours. Then the solution was kept for precipitation and the precipitate was filtered after washing it several times by distilled water and ethanol. The whole reaction was performed at room temperature.

C. Characterizations

For studying the different properties of synthesized sample following characterization techniques were used: The crystallographic information was obtained with the powder XRD method on a Proto A-XRD Bench top Diffractometer with fixed Cu-Kα (0.154 nm). The morphology and surface properties were investigated using Nova Nano SEM 450. UV-Vis absorption spectra were recorded on the Unicam 5625 UV-Visible spectrometer in the range 195 nm - 325 nm.

III. RESEULT AND DISCUSSION

A. UV-Visible characterization of as synthesized material

For studying the optical properties and band gap of synthesized nanoparticles UV-Vis characteristic has been taken. Fig. 1 shows the UV-Vis spectra of synthesized sample. It shows intense absorbance peak exist at 200 nm while a broad absorbance peak exist at 270 nm. Fig. 2 shows the tauc plot of synthesized sample. The band gap of as-synthesized nanomaterial is 5.73 eV. The band gap of as-synthesized material is determined by the equation:

\[ a\hbar^2 = A (\hbar \nu - E_g) \]
Where $\alpha$ is the absorption coefficient, $E_g$ is the absorption band gap, $A$ is constant, $n$ depends on the type of transition, $h$ is plank constant and $\nu$ is the frequency. As Magnesium oxide has direct allowed transitions, we have chosen $n = 1/2$. The band gap of the material is calculated from the $(\alpha h \nu)^2$ vs $h \nu$ plot (Tauc plot).

**Fig. 1:** UV-Visible spectra of synthesized Magnesium oxide nanomaterial

**Fig. 2:** Tauc plot of synthesized magnesium oxide nanomaterial

**B. Structural analysis of synthesized nanomaterial**

Fig. 3 shows the XRD pattern of synthesized magnesium oxide nanoparticles. The XRD peaks assigned with ‘*‘ and ‘#’ symbol correspond to the diffraction peaks obtained for Rhombohedral structure of MgO (JCPDS file no. 27-0759) and Hexagonal phase of Mg(OH)$_2$ (JCPDS file no. 75-1527) respectively. The average crystallite size has been calculated for MgO using the Scherrer formula $[D = k\lambda/\beta\cos(\theta)]$. In this formula, $k$ is the constant ($=0.91$), $\lambda$ is the wavelength of X-ray radiation used, $\beta$ is the full width at half maximum (FWHM) intensity of diffracted peak and $\theta$ is the Bragg diffraction angle. The calculated average crystallite sizes for MgO nanoparticles are 34.04 nm.

**Fig. 3:** XRD pattern of synthesized nanomaterial

**C. Morphological studies of synthesized nanomaterial**

The FESEM images of synthesized nanomaterial are shown in Fig. 4 at different magnifications. It can be clearly seen that Magnesium oxide nanoparticles possessed nanotubes morphology with agglomeration. The diameter of outer wall of nanotubes is approximately $\approx$ 78 nm and inner diameter is approximately $\approx$ 31 nm. The diameters of nanotubes were analyzed using Image J software.

**Fig. 4:** FESEM images of synthesized magnesium oxide nanotubes at (a) 300 nm magnification and (b) 200 nm magnification.

**D. Chemical composition analysis of synthesized nanomaterial**

The energy dispersive X-ray spectrum of synthesized nanomaterial is shown in Fig. 5 which shows only Mg and O peak. The other peaks correspond to C and Cu which were used for holding the sample. This confirms the high purity of magnesium oxide nanomaterial synthesis.

**Fig. 5:** EDX spectrum of synthesized magnesium oxide nanomaterial
IV. CONCLUSION

From our present work, it is easy to conclude that Magnesium oxide with nanotube morphology can easily be synthesized using Chemical Co-precipitation method. With this method, we can easily synthesize magnesium oxide in nanotube morphology without using any surfactant or capping agent. The synthesized magnesium oxide nanotubes have shown wide band gap (5.73 eV) and the can be used in different applications like solar cells, catalysts and sensors etc.

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REFERENCES


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