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Study on the Synthesis and Characterization of Magnesium Oxide Nanoparticles Synthesized By Precipitation Method

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ABSTRACT:*In this work magnesium oxide nanoparticles are synthesized using a simple Co-precipitation method using magnesium nitrate and sodium hydroxide as the precursor with starch as the stabilizing agent. This method has many advantages such as low cost, easy to scale, and less time consuming. The synthesized nanoparticles are characterized by X- ray Diffraction (XRD), Scanning Electron Microscopy (SEM) and Energy Dispersive X- ray Spectroscopy(EDAX). The particle size of the synthesized nanoparticles is found to be approximately 70 nm using SEM. The crystallite size calculated using Scherrer formula is approximately 18nm. These synthesized nanoparticles can be tested for their antimicrobial activity and used in biomedical applications. The focus of the present study is to synthesize magnesium oxide nanoparticles using a simple method.*

KEYWORDS -*Magnesium oxide nanoparticles, Metal oxide nanoparticles, Nanotechnology, X- ray diffraction, scanning electron microscopy*.

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I. INTRODUCTION

During the last few decades use of nanoparticles created a new hope for solving human problems. Magnesium oxide nanoparticles consist of white powder which is nontoxic, odorless and has a high melting point and high hardness[1].It is a hygroscopic solid mineral and its lattice consists of Mg^{+} ions and O^{-2} ions.Magnesium oxide is an inorganic material with a molar mass of 40.31 g/mol and a density of 3.58 g/cm^{3}.In MgO, Mg^{+2} ions and O^{-2} ions are linked by an ionic bond[2].The advantages of magnesium oxide nanoparticles are that they are biocompatible, biodegradable and relatively low cost. In medical industries also it is used to treat heartburn, sour stomach, improve bone regeneration. It is also a strong antimicrobial and antitumor agent.
Magnesium oxide is one of the most useful ceramic materials. The crystal structure of magnesium oxide is cubic, which makes it a periclase structure. Its atomic arrangement is of the NaCl type, and this arrangement causes the common morphologies of MgO to be cubic, octahedral, and polyhedral in shape. Magnesium oxide can be produced through the decomposition of magnesium hydroxide or magnesium carbonate. Because the preparation of magnesium oxide involves a calcination process, it is difficult to keep the particle size in the nano range [3].

At the nanoscale, MgO shows high reactivity due to a great number of highly reactive edges, structural defects on the surface, unusual lattice planes and high surface to volume ratio[4]. Nano-MgO is a functional material that has been widely used in various areas and recently it has been reported that MgO has a good bactericidal performance in aqueous environments. Nano- MgO exhibits high activity against bacteria, spores and viruses because of its large surface area. The positively charged particles can interact strongly with negatively charged bacteria. Compared with TiO_2, silver, copper and other kinds of solid bactericides, nano-MgO has the advantage of being prepared from readily available and economical precursors and solvents, and therefore has considerable potential as a solid bactericidal material under simple conditions [5].

Metal and Metal salts are toxic to microbes at very low concentrations and they kill microbes by binding to intracellular proteins and inactivating them. Nanoparticles of silver and zinc oxide have taken a viable solution to stop infectious diseases, because of their antimicrobial properties. Health concerns along with customer satisfaction have made functionally finished textiles a fast-paced and fast growing industry [6]. Magnesium oxide nanoparticles have been recognized as safe materials by the United States Food and Drug Administration (21CFR184.1431)[7]. It has been reported that the shape and size of nanocrystalline magnesium oxide particles endow them with high specific surface and reactivity, because of the high concentration of edge/corner suites and structural defects on their surface.

MgOnanoparticles have wide applications in the field of catalyst support, agricultural products, paints, superconductor products, photonic devices and sensors.Magnesium oxide nanoparticles were synthesized by various methods.Abbas et al [8] hascreated MgO nanoparticles using a high voltage power supply of 21kV athigh-frequency equipment that produced a 6kHz output.Many researchers have synthesized using sol gelmethod[9-11] while others also have used hydrothermal method[12-14].The present study was carried out with the main objective of evolving a simple method for the synthesis of MgO nanoparticles.

II. EXPERIMENTAL

The Magnesium oxide nanoparticles are synthesized using the method described by Sundrajan et al [15].Magnesium nitrate, Sodium hydroxide and Starch are the chemicals purchased from Merck. Magnesium oxide nanoparticles were prepared by wet chemical methods using magnesium nitrate and sodium hydroxide as precursors and soluble starch as a stabilizing agent. Starch acts as a stabilizing agent and also prevents the agglomeration of nanoparticles.

Starch (0.1 % concentration) solution was prepared in 100 ml of distilled water and Magnesium nitrate (0.1 M) was added to the above solution. Then the solution was kept under constant stirring using a magnetic stirrer for complete dissolution of contents. After complete dissolution, 0.2 M sodium hydroxide solution was added in drops along the sides of the container withconstant stirring for 2 hours and allowed to settle for 24 hours. The supernatant liquid was then discarded carefully and the remaining solution was centrifuged for 10 minutes. The centrifuge was washed three times using distilled water to remove the byproducts and the excessive starch that bound to the nanoparticles. The nanoparticles of magnesium hydroxide were placed in a furnace at 700°C for 4 hours. During this process, conversion of magnesium hydroxide into magnesium oxide takes place. The following reaction explains theformation of magnesium oxide nanoparticles:

$Mg(NO_3)_2 .6H_2O + 2 NaOH \rightarrow Mg(OH)_2 + 2NaNO_3$

$Mg(OH)_2 \rightarrow MgO + H_2O$ … (1)

III. RESULTS AND DISCUSSIONS

Co-precipitation method is widely used for the synthesis of nanoparticles. It involves liquid-phase synthesis. The common precipitating agent used is sodium hydroxide. The basic principle involves single nucleation involves and uniform growth. The critical solute concentration that initiates the process plays a major role in the classical process, with solute diffusion that initiates the process plays a major role in the classical process, with solute diffusion on the surface causing growth. It is necessary to separate these two processes. The resulting precipitate is then washed and dried.

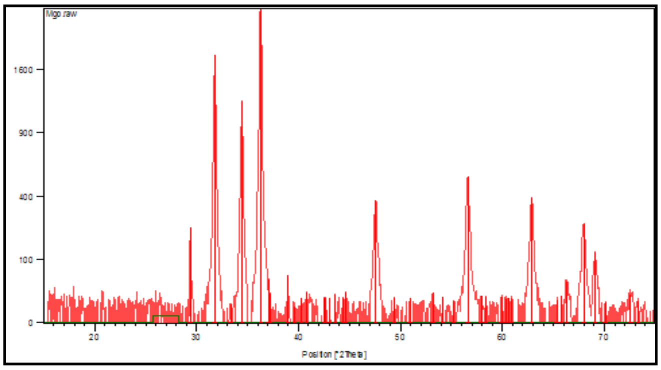

Fig. 1: X ray spectra of synthesized magnesium oxide nanoparticles

The synthesized magneisum oxide nanoparticles was confirmed by an X-ray diffraction (XRD) using Philips X'Pert diffractometer using Cu (Kα) radiation (wavelength:1.5406 A^0) at 40 kV and 40 mA at room temperature in the range of 2θ from 20^0 to 80^0.The crystalline structure of the synthesized samples was examined.XRD analysis showed a series of diffraction peaks at 2θ of 31.78^0, 34.44^0, 36.26^0, 47.57^0, 56.60^0, 62.88^0, 67.96^0 that can be assigned to (111), (200), (220), (311), (222), (400) and (331) respectively. All the peaks were indexed to the cubic phase of MgO and matched with JCPDs data (Card no. 89.7746).The lattice parameters are found to be a =b=c=4.21 A^0. The strong and sharp diffraction peaks indicate the high crystallinity of the magnesium oxide nanoparticles.

The average crystallite size of magnesium oxide nanoparticles was calculated using Scherrer formula given by:

$$D = \frac{0.9\lambda}{\beta \cos \theta} \quad \ldots (2)$$

where, D is the crystallite size (nm), β is the full width at halfmaximum of the peak.Using the above formula the average crystallite size is found to be 18nm.

Table 1: X- ray diffraction data of synthesized MgO nanoparticles

Pos (2Th.)	Height(cts)	FWHM(2Th.)	d-spacing A_0	Rel. Int.(%)	Peak
31.7831	1797.58	0.2160	2.81311	72.43	111
34.4412	1230.18	0.2160	2.60185	49.57	200
36.2643	2481.72	0.3120	2.47511	100.00	220
47.5793	374.96	0.2400	1.90956	15.11	311
56.5968	534.24	0.2640	1.62485	21.53	222
62.8850	393.33	0.2400	1.47664	15.85	400
67.9678	246.17	0.3360	1.37806	9.92	331

The XRD spectra, which is presented in Fig. 1, reveals that the nanoparticles are single crystalline and can be marked as cubic-phase (a = 0.421nm) MgO (JCPDS card 45-0946).
B. Scanning Electron Microscopy

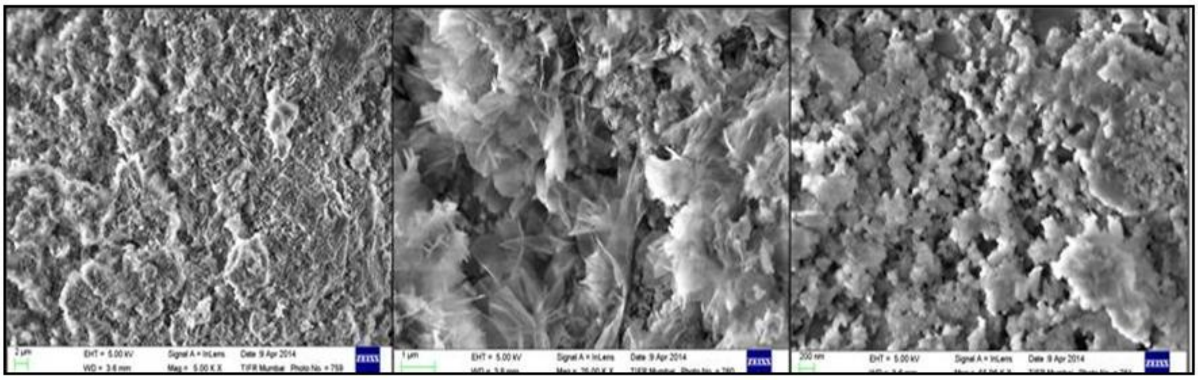

Fig. 2: SEM micrograph of synthesized magnesium oxide nanoparticles at different magnification

The size and morphology of the synthesized magnesium oxide nanoparticles was studied through aSEM micrographthat shows agglomeration of some magnesium oxide nanoparticles.
The scanning electron microscopy shows that the particles are polymorphic with the average particle size of 70 nm.

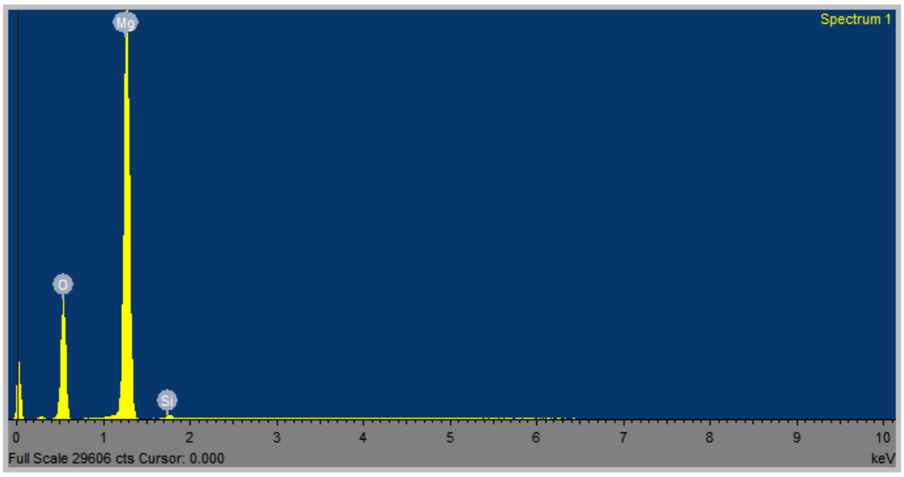

Fig. 3: Energy Dispersive X ray spectroscopic graph of synthesized magnesium oxide nanoparticles

The Energy Dispersive spectra of the synthesized nanoparticles show small Si impurity in the sample. Along with the peaks corresponding to Mg and O the peak corresponding to Si is also detected in the spectrum. Table 2 shows the atomic percentage of magnesium, oxygen and some silicon impurity.

Table 2: EDAX data of synthesized Magnesium oxide nanoparticles

Spectrum	O at wt %	Mg at wt%	Si at wt %
Spectrum 1	53.27	46.23	0.50
Mean	53.27	46.23	0.50
Std. Deviation	0.00	0.00	0.00
Max.	53.27	46.23	0.50
Min.	53.27	46.23	0.50

IV. CONCLUSION

Magnesium oxide and specifically its nanostructured form is a widely demanded and used material from the group of simple metallic oxides.Many methods can be used for the synthesis of Mg nanoparticles.In this study, a simple, efficient and economic synthesis method is used.In wet chemical precipitation method by controlling reaction parameters like time, temperature, stirring speed the nanoparticles of required shape and size can be manufactured. The particle size obtained using SEM is around 70 nm and the particles are mostly polymorphic. The further aim of this study is to standardize the process by changing the calcination temperature and studying its effect on the particle size.These particles can be tested for their antibacterial property and can be used for biomedical applications.

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