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A COMPARATIVE STUDY ON ANTIBACTERIAL PROPERTIES OF MgO NANOPARTICLES PREPARED UNDER DIFFERENT CALCINATION TEMPERATURE

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MgO nanoparticles were synthesized via wet chemical method by applying different calcinations temperature. The temperature effects on the surface morphology, size of nanoparticles have been reported. The synthesized nanoparticles were characterized and confirmed by X-ray diffraction (XRD) and Scanning Electron Microscope (SEM) analysis. It exhibits the increase of crystallinity with sharp intense peak and increase of size with varying morphologies respectively with respect to the increase of calcinations temperature. Antibacterial studies were done on gram positive (S.aureus) and gram negative (E.coli) bacteria by agar diffusion method. The zone of inhibition was found to more for gram positive bacteria compared with gram negative bacteria. Antibacterial property of prepared nanoparticles is decreases with increases of calcinating temperature. Since this method of preparation produced better yield in simple and cost effective way. Hence it can be applied for more production of magnesium oxide nanoparticles.

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1. Introduction

Nanoscience and technology have emerged over the past decade as the forefront of science and technologies [1]. Nanotechnology plays an important role in the industrial revolution. It is concerned with materials whose structures exhibit significantly novel and improved physical, chemical and biological properties [2]. The intrinsic properties of nanoparticle are mainly determined by size, shape, composition, crystallinity and morphology. Nano-scale particles provide a narrow size distribution, which is required to obtain a uniform material response [3]. It has been reported nanoparticles can be prepared from aqueous solution by different ways such as hydrothermal, precipitation and microemulsion routes [4-6]. Wet chemical synthesis of nanoparticles is a valuable alternative to conventional process and gas phase synthesis with known commercial applications [7].

Inorganic materials such as metal and metal oxides have attracted lot of attention over the past decade due to their ability to withstand harsh process conditions [8, 9]. Metal oxides such as TiO_2, ZnO, MgO and CaO are of particular interest as they are not only stable under harsh process conditions but also generally regarded as safe materials to human beings and animals [10]. Metal and Metal salts are toxic to microbes at very low concentrations and they kill microbes by binding to intracellular proteins and inactivating them [11]. 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 [12]. Magnesium oxide is an interesting basic oxide that has many applications in catalysis, adsorption and in the synthesis of refractory ceramics [13-16]. It is a unique solid of high ionic character, simple stoichiometry and crystal structure and also it can be

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prepared widely with variable particle sizes and shapes [17]. 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 sites and structural defects on their surface [18]. The present study was carried out with the main objective of evolving a simple method for the synthesis of MgO nanoparticles. MgO nanoparticles are characterised by XRD and SEM analysis. Antibacterial studies of prepared MgO nanoparticles under different calcinations temperature were done by S.aureus (gram positive) and E.coli (gram negative) by agar diffusion method.

2. Experimental

2.1 Nanoparticle Preparation

Magnesium nitrate, Sodium hydroxide and Starch are the chemicals purchased from Merck. Magnesium oxide nanoparticles were prepared by wet chemical method using magnesium nitrate and sodium hydroxide as precursors and soluble starch as stabilizing agent. Starch act 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 12.83 g (0.1 M) was added to the above solution. Then the solution was kept under constant stirring using magnetic stirrer for complete dissolution of contents. After complete dissolution, 4g (0.2 M) sodium hydroxide solution (25 ml) was added in drops along the sides of the container under constant stirring for 2 hours and allowed to settle for 24 hours. The supernatant liquid was then discarded carefully and the remaining solution was centrifuged (10,000 rpm at 25°C) for 10 minutes. Centrifugate was washed three times using distilled water to remove the by products and the excessive starch that bound with the nanoparticles. The nanoparticles of magnesium hydroxide were placed in furnace at 300°C, 500°C and 700°C for 4 hours. During this process, conversion of magnesium hydroxide into magnesium oxide takes place. The following reaction explains the formation of magnesium oxide nanoparticles.

$$\mathbf{Mg\,(NO_3)_2\,.6H_2O + 2\,NaOH \;\rightarrow\; Mg\,(OH)_2 + 2NaNO_3}$$
$$\mathbf{Mg\,(OH)_2 \xrightarrow{\Delta} MgO + H_2O}$$

2.2 Disc diffusion method for Antibacterial activity

Antibacterial activity was carried out by the disc diffusion method using the suspension of bacteria spread on nutrient agar. Dip the swab into the broth culture of the organism. Gently squeeze the swab against the tube inside to remove excess fluid. Use the swab to streak agar plate or a nutrient agar plate for a lawn of growth. This is best accomplished by streaking the plate in one direction, then streaking at right angles to the first streaking, and finally streaking diagonally. We end by using the swab to streak the outside diameter of the agar. The inoculated plates were incubated at appropriate temperature for 24 hours. Antibiotic discs can be placed on the surface of the agar using a dispenser that dispenses multiple discs at the correct distance apart, or by obtaining individual discs and placing them on the surface of the agar using flame sterilized forceps. The antibacterial activity was evaluated by measuring the zone of inhibition against the test organisms. Zone of inhibition is the area in which the bacterial growth is stopped due to bacteriostatic effect of the compound and it measures the inhibitory effect of compound towards a particular microorganism. Finally we measure (mm) diameters of zones of inhibition of the control strain and test with a ruler, callipers [19].

2.3 Characterization of MgO Nanoparticles

X-ray diffraction (XRD) analysis of drop-coated films on glass substrates from MgO sample was carried out on a JEOL IDX 8030 instrument operating at 40 kV with a current of 30 mA using Cu Ka. The surface morphological features (shape and particle size) of the MgO

Nanoparticles were measured using a JEOL JSM 6390 model (Scanning Electron Microscope). E.coli (gram negative ATCC 10536) and S.aureus (gram positive ATCC 11632) by qualitative measurement using agar diffusion test (Kirby Bauer disc diffusion method).

3. Results and discussions

3.1 XRD analysis

Fig. 1 shows the XRD pattern of the sample prepared by wet chemical method. The XRD pattern of MgO nanoparticles at 300°C and 500°C shows some of the unidentified peaks, the broadness of the peak and amorphous nature pointed out the presence of impurities and inadequate of calcinations temperature but at 700°C, the presence of the sharp peaks, absence of unidentified peaks and good agreement with the JCPDS data (89-7746) exhibit the increase of crystallinity and complete formation of pure nanocrystalline MgO without any foreign material.

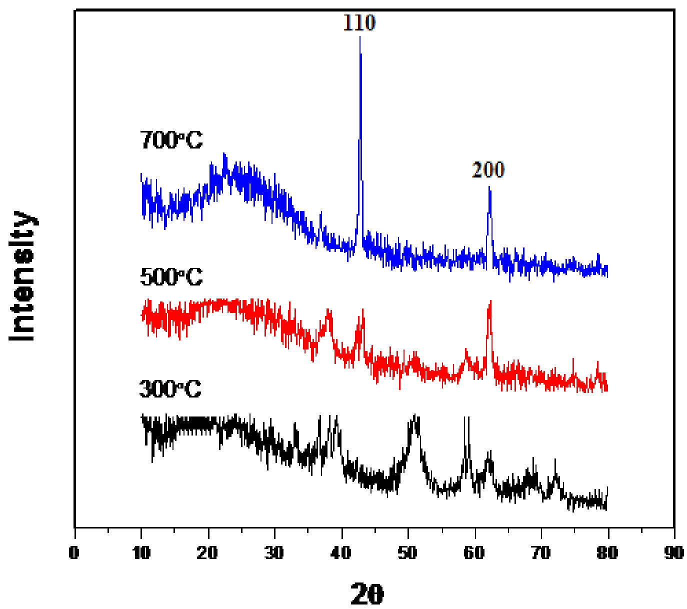

Fig 1. XRD spectrum of MgO nanoparticles at three calcinating temperature.

The average crystallite size from the Debye Scherrer's equation of the strongest intense peak covering the plane (110) and (200) of MgO nanoparticles at 300°C, 500°C and 700°C were 4.6 nm, 8.5 nm and 13.3 nm respectively. The grain size of nanoparticles exhibit the increase of size with increase of temperature due to the reason that the nanoparticles tend to form aggregates at higher temperature through inter-particle resulting from electrostatic forces.

3.2 SEM analysis

Scanning Electron microscopic images of MgO nanoparticles [Fig 2 (a_1, a_2, a_3)] show magnification at 300, 500 and 700°C depicts that the nanoparticles are appearing as discrete particles. During magnesium hydroxide calcination at 300^{o}C, the dense flakes of the particles persist but it can be breakdown into isometric particles by increasing temperature to 500^{o}C which looks irregular shaped flakes. At 700°C, the morphology becomes irregular shaped aggregated particles. The size also increases in higher proportion from lower calcinations temperature to higher which is shown in following table 1.

Table 1: Morphology and size range at different calcinating temperature of MgO nanoparticles

Annealing temperature (°C)	Morphology	Size range (nm)
300	Dense flakes	30-50
500	Irregular shaped flakes	50-80
700	Irregular porous structure	70-130

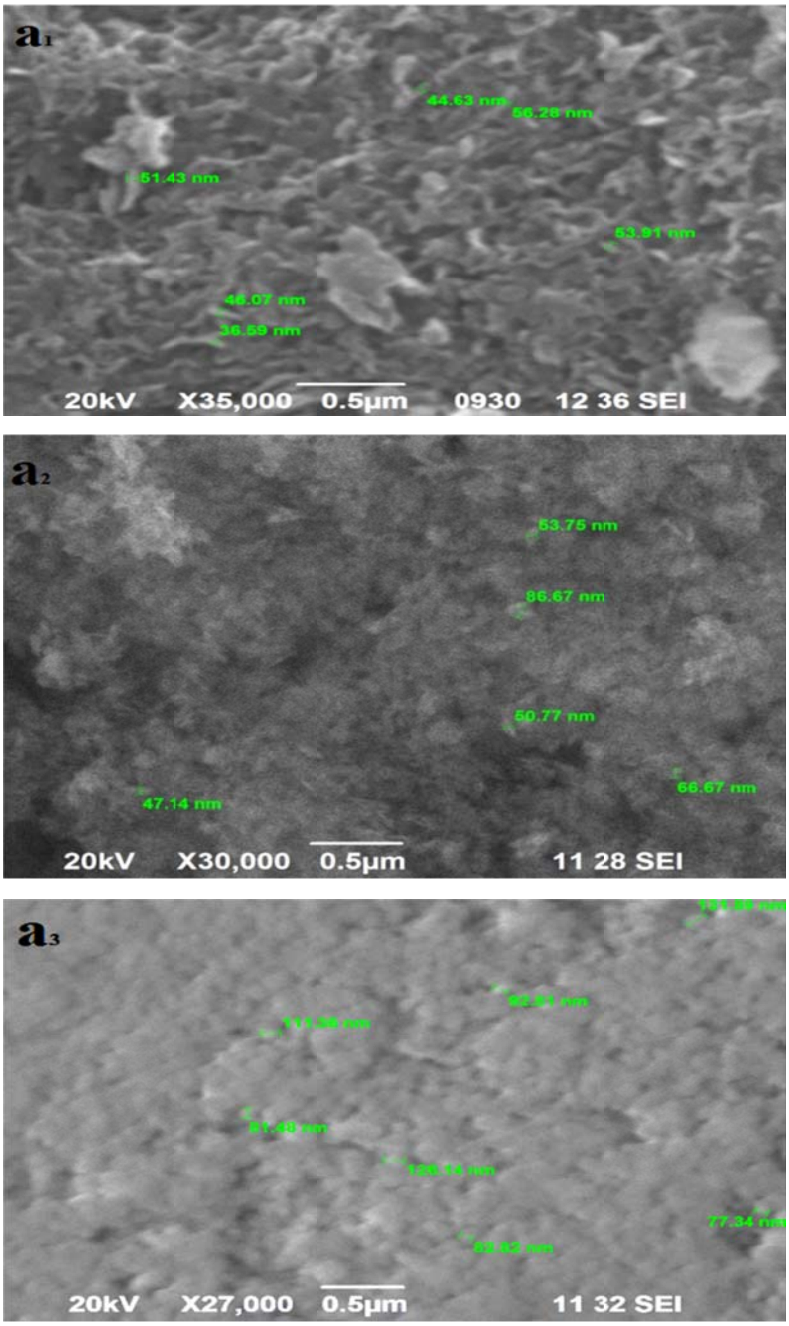

Fig 2.SEM images of MgO nanoparticles at different calcinating temperature
a_1 =300^{o}C, a_2 =500^{o}C, a_3 =700^{o}C.

3.3 Antibacterial studies

Antibacterial activities have been tested on gram positive (S.aureus) and gram negative (E.coli) bacteria. The size effect of nanoparticles on the antibacterial efficiency has been investigated. It has been found that smaller sized MgO nanoparticles have antimicrobial activities towards both gram positive and gram negative while bigger sized MgO nanoparticles have effect on gram negative only. The antibacterial activities of magnesium oxide nanoparticles with lowest cost of production compared to titanium dioxide and silver nanoparticles have been performed using well diffusion method. Antibacterial activity towards bacteria E.coli ATCC 10536 and ATCC 11632 of magnesium oxide nanoparticles at different calcinations temperature show in the table 2. From Table 2 and Figure 3 shows the difference in the zone of inhibition for the two bacteria's at different calcinating temperature. It can be shown that zone of inhibition was found to be more in gram positive bacteria compared to the gram negative bacteria. Since antibacterial property of prepared nanoparticles is decreases with increases of calcinating temperature. Zone of inhibition effects of magnesium oxide nanoparticles occurs by inhibiting the growth of microorganism using an electrochemical mode of action to penetrate and disrupt their cell walls. When the cell walls are penetrated, leakage of metabolites occurs and other cell functions are stopped, thereby preventing the organism from functioning or reproducing.

Table 2: Antibacterial assessment by agar diffusion method

Annealing temperature (°C)	Zone inhibition diameter (mm)	
	S.aureus (Gram positive)	E.coli (Gram negative)
Control	17	17
300	23	21
500	21	19
700	19	17

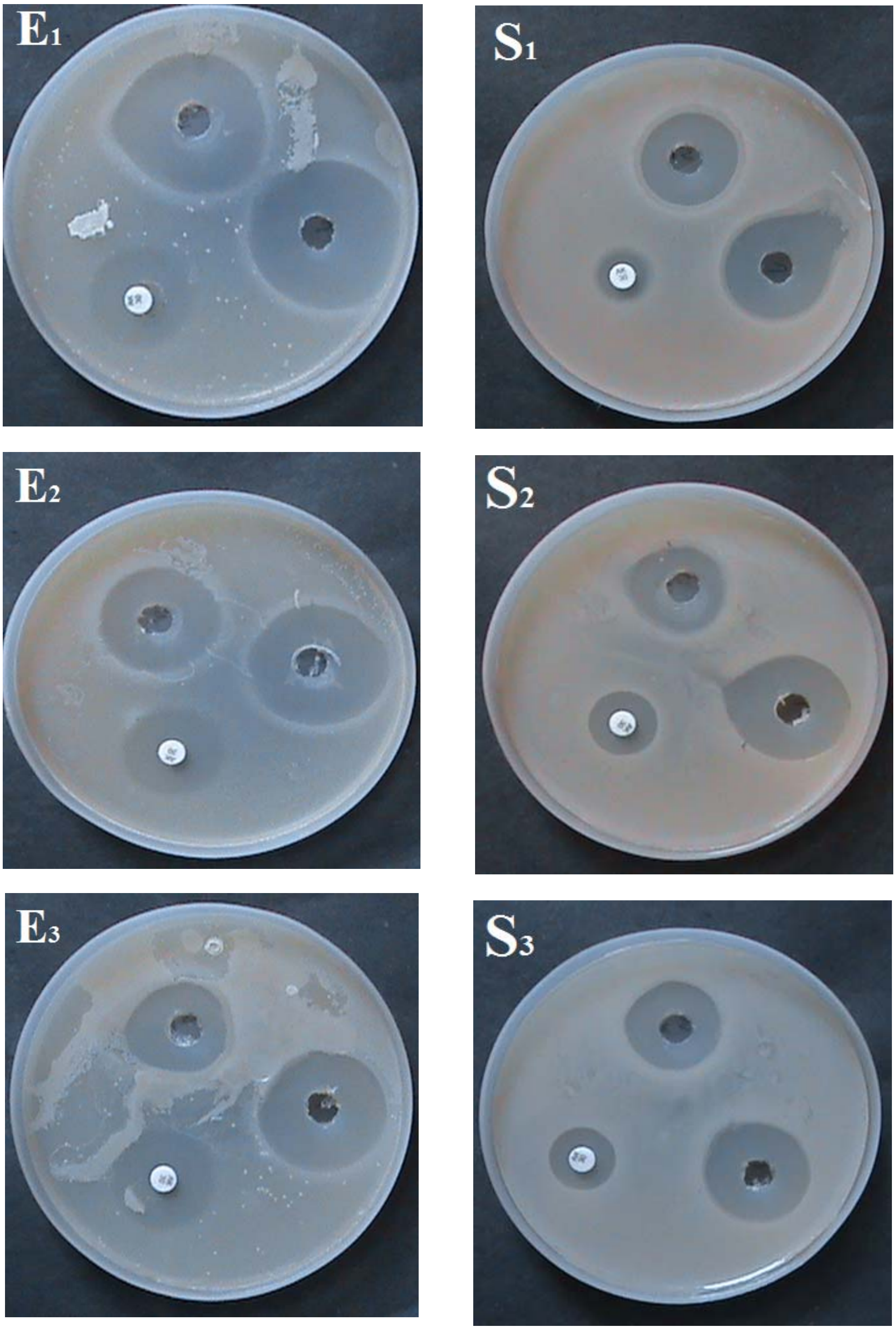

Fig 3. Antibacterial effect of MgO nanoparticles at three calcinating range against E.coli (E1, E2 & E3) and S.aureus (S1, S2 & S3)

4. Conclusions

Magnesium oxide nanoparticles are synthesized by simple wet chemical method and characterized by XRD and SEM analysis. The grain size was calculated as 4.6nm, 8.5nm & 13.3nm with respect to 300°C, 500°C & 700°C using Debye scherrers method by XRD analysis. Scanning electron microscopic images under various temperatures depicts nanoparticles are looking like dense flakes, irregular shaped flakes and irregular porous structures. Antibacterial studies done against S.aureus than E.coli by agar diffusion test. It can be shown that zone of inhibition was found to be more in gram positive bacteria compared with gram negative bacteria. Because antibacterial property of as prepared nanoparticles is decreases with increases the calcinating temperature i.e., it's depend upon the size of the nanoparticles. Since antibacterial character finds wide application in the health and hygiene of textile sector, this method of preparation of magnesium oxide will be useful in cost effective way.

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