Synthesis, Characterization and antibacterial activity of Magnesium Oxide (MgO) nanoparticles.

A Almontasser¹, A Parveen² and A Azam³

Department of Applied Physics, Z. H. College of Engineering & Technology, Aligarh Muslim University, Aligarh-202002, India.

Email;azrap2015@gmail.com

Abstract. Magnesium nitrate hexahydrate Mg(NO₃)₂·6H₂O, ammonia and distilled water were used for preparation magnesium oxide (MgO) via a precipitation method, where magnesium nitrate is used as a precursor, distilled water as a solvent and ammonia is used to maintain pH of the sample. The MgO was characterized by an X-ray diffractometer microscopy and a UV-Vis spectroscopy. In this study, The average particle size has been investigated by XRD spectroscopy, which came out to be 7 nm by using Scherrer's equation. The samples had good crystallinity with a preferred orientation in the (222) direction. The energy band gap was estimated using UV-Vis spectroscopy, which is equal to be 4.8eV. As well as, in the present paper, the main goal for preparation Magnesium oxide is to study the antibacterial activity of magnesium oxide. Antibacterial was testing by analyzing the diameter of inhibition zone appeared in disk diffusion tests and minimum inhibitory concentration (MIC) and minimum bactericidal concentration (MBC) of samples dispersed in media. The results of bacterial sensitivity of nanoparticles vary depending on the type of bacteria E. coli and S. aureus, hence revealed the efficacy of magnesium oxide nanoparticles.

1. Introduction

In past few years inorganic materials (i.e metals and metal oxides) have attracted a lot of attention due to their capability of withstanding harsh process conditions[1]. Nanoscale magnesium oxide (MgO) is an important inorganic material, it will unique properties. Many techniques have been used to synthesize MgO nanoparticles such as direct chemical transformation[2], aerogel [3], hydrothermal method [4], microwave radiation[5][6], vapor-solid process[7], laser ablation [8], sol-gel [9][10], solid-state interfacial
diffusion reaction [11], etc. There are several applications for magnesium oxide nanoparticles such as waste remediation, pharmaceutical, refractory materials and glass industry and catalysis [12–14]. Also, it has applications as both catalyst and catalyst support [15–17], etc. Many pieces of research have been focused on new and effective antimicrobial reagent developments recently,[18] the multiple drug resistance developments have been done by disease causing bacteria because of the haphazard use of antibacterial drugs and drugs which are used to treat infectious diseases. Furthermore, the association of antibiotics sometimes shows effects such as immune-suppression, hypersensitivity, and allergic reactions [19]. Therefore, developing sources of active antibacterial nanomaterials were required because of the increasing antibiotic resistance.

In the present study, we reported the successful precipitation method for preparing magnesium oxide nanopowder by using magnesium nitrate as the precursor and distilled water as a solvent. The characterization has been done using XRD which confirms the product of MgO nanoparticles, UV-Vis spectroscopy was used to evaluate the energy band gap of MgO.

2. EXPERIMENTAL

2.1 Synthesis of MgO Nanoparticles

Magnesium oxide (MgO) has been synthesized using a precipitation method as shown in Fig1. 0.02 moles of Magnesium Nitrate hydrate Mg (NO₃)₂.6H₂O was dissolved in 100 ml of distilled water for preparation of an aqueous solution of magnesium Nitrate hydroxide. The mixture of Magnesium Nitrate hydrate and distilled water was stirred for 4 hours until it becomes clear. During stirring ammonia has been added drop wise to Magnesium Nitrate hydrate solution to adjust pH to 9. Immediately a precipitate was obtained. By using methanol and distilled water, the collected precipitate was washed many times, then filtered and dried overnight in the oven at 100 °C. Dried sample was ground and calcinated at 500 °C for three hours in Muffle furnace. Finally, the white nanopowder was obtained.
3. RESULTS AND DISCUSSION

3.1 Characterization of MgO materials

The powder X-ray Diffraction (XRD) using a Philips X-ray diffractometer with CuKα radiation (λ = 0.15406 nm) was used to characterize MgO nanoparticles for identification of crystal phase. Fig.2 confirms the cubic phase of magnesium oxide, there were no characteristic peaks of impurities. Five MgO
characteristic reflection peaks were observed in Fig. 2 and indexed to the (1 1 1), (2 0 0), (2 2 0), (3 1 1), and (2 2 2) diffraction planes. The parameter of the cubic unit cell is $a=b=c=4.130$. The average crystallite size was calculated for the highest peak (2 0 0) using the Debye-Scherrer formula,

$$ D = \frac{0.9 \lambda}{\beta \cos \theta} $$

(1)

Where, $D$ is the average crystallite size, $\lambda$ is the x-ray wavelength (1.504 nm), $\theta$ is the Bragg diffraction angle, and $\beta$ is the full width at half maximum which is measured using Gaussian curve at the Peak (2 0 0). The average crystallite size of MgO was calculated to be 7 nm.

Figure 2. XRD pattern of MgO nanoparticles.
3.2 UV-Visible spectroscopy

UV-vis spectrophotometer is used to record UV-visible spectra of the prepared MgO nanopowder in the absorbance mode, and in the wavelength range between 200–900 nm to determine the absorbance of MgO NPs. We obtained the distinctive absorption bands of MgO up to 800 nm. The spectrum of MgO contains several absorption peaks as observed in Fig 3. MgO is reported to exhibit a broad absorption peak in between 260-330 nm[20]. The spectrum has two absorption humps in the range of 320–400 nm centered at 380 nm and between 500–600 nm with the peak at 550 nm.

![UV-Vis absorption spectrum of MgO nanoparticles.](image)

Figure 3. UV-Vis absorption spectrum of MgO nanoparticles.
Tauc’s relation was used for determination of energy band gap of magnesium oxide.

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

(2)

Where, \(\alpha\) is the absorption coefficient, \(h\) is the Planck's constant, \(A\) is a constant, \(E_g\) is the energy band gap, \(n\) is a constant which is equal to \(\frac{1}{2}\) for the direct band gap. The calculation for the band gap of the MgO nanoparticles was done from the Fig.4 using extrapolating the curve drawn between \((h\nu)\) and \((\alpha h\nu)^2\). Where \(\nu\) represents the frequency, \(\alpha\) refers to the optical absorption coefficient. The energy band gap was observed extrapolating the curve and it was found to be around 4.8eV.

Figure 4. Band gap spectra of MgO nanoparticles.
3.3 Antibacterial activities in solid medium

This work was performed by the bacterial test using a disk diffusion test, sterile Petri-dishes containing sterile Luria agar medium. The different concentration of magnesium oxide nanoparticles was used (10, 20 and 30 mg/ml). The nanoparticles were loaded on filter paper. The Luria agar plate was spread with the bacterial suspension of concentration (1ml of $10^4$ CFU ml$^{-1}$) before we place the disks on the plate using a sterile swab. All the petri dishes were placed in the incubator at 37 $^\circ$C for 24 hrs, and then we have measured the diameters of inhibition zones. Inhibition zone diameter (mm) of bacterial growth was calculated using the inhibition zone, which are equidistant from each other, and the average value of the inhibition zone diameter was calculated. The tests were carried out at least three times using both E. coli and S. aureus, and the reported data represent average values.

Fig 5(a) exhibit the zone of inhibition of MgO nanoparticles at different concentration against Staphylococcus aureus and Escherichia coli in Fig 5 (b). From the Table1 it is indicated that the magnesium oxide nanoparticles inhibit the growth of both gram-negative and positive bacteria. As shown in Fig 6 The inhibition zone increases with the increase in the concentration of MgO nanoparticles.

Figure 5 Inhibition zone of MgO against (a) S.aureus and (b) E.coli.
Table 1. Diameter of the inhibition zone with the different concentration of MgO nanoparticles.

| S.No | Concentration of MgO nanoparticles (mg/ml) | Diameter of inhibition zone (mm) ±SD |
|------|------------------------------------------|-------------------------------------|
|      |                                          | S. aureus (gram positive) | E. coli (gram negative) |
| 1    | 0                                        | -         | -                        |
| 2    | 10                                       | 7± 0.5    | 8.3± 0                   |
| 3    | 20                                       | 8 ± 0     | 10.7± 0.58              |
| 4    | 30                                       | 9.3± 0.29 | 12.7± 0.58              |

Figure 6. The diameter of inhibition zone (DIZ) of different concentration of MgO nanoparticles in the presence of E.coli and S. aureus.
Conclusion

In this work, fine MgO powders were successfully synthesized by a precipitation method using magnesium nitrate, distilled water and ammonia to maintain pH. The XRD result confirmed the presence of magnesium oxide nanopowder with high purity. The energy band gap of as-synthesized MgO nanoparticles was obtained to be 4.8 eV. Fig 6 suggests the decrease in bactericidal growth with increase in MgO concentration which indicates that MgO nanoparticles inhibit the growth of bacteria. The minimum inhibition of E. coli and S. aureus was recorded with 10 mg/ml of MgO nanoparticles where is the maximum inhibitory of E. coli and S. aureus with 30 mg/ml of MgO nanoparticles.

References

[1] Comini E, Faglia G, Ferroni M, Ponzoni A, Vomiero A and Sberveglieri G 2009 *J. Mol. Catal. A Chem.* **305** 170–177.
[2] Ilyina E V, Mishakov I V and Vedyagin A A 2009 *J. Mol. Catal. A Chem.* **299** 44–52.
[3] Dong C, Song D, Cairney J, Maddan O L, He G and Deng Y 2011 *Mater. Res. Bull.* **46** 576–582.
[4] Al-Hazmi F, Alnowaiser F, Al-Ghamdi A A, Al-Ghamdi A A, Aly M M, Al-Tuwirqi R M and El-Tantawy F 2012 *Superlattices Microstruct.* **52** 200–209.
[5] Makhluf S, Dror R, Nitzan Y, Abramovich Y, Jelinek R and Gedanken A 2005 *Adv. Funct. Mater.* **15** 1708–1715.
[6] Esmaeili E, Khodadadi A and Mortazavi Y 2009 *J. Eur. Ceram. Soc.* **29** 1061–1068.
[7] Huang L, Li D, Lin Y, Wei M, Evans D G and Duan X 2005 *Adv. Funct. Mater.* **15** 1708–1715.
[8] Wang Y H, Liu H M and Xu B Q 2009 *J. Mol. Catal. A Chem.* **299** 44–52.
[9] Aguiar H, Solla E L, Serra J, González P, León B, Almeida N, Cachinho S, Davim E J C and Correia R 2008 J.M. Oliveira, M.H.V. Fernandes, *J. Non. Cryst. Solids* **354** 4075–4080.
[10] Abdullah M M, Rajab F M and Al-Abbas S M 2014 *AIP Adv.* **4** 0–11.
[16] Mishakov I V, Bedilo A F, Richards R M, Chesnokov V V, Volodin A M, Zaikovskii V I, Buyanov R A and Klabunde K J 2002 *J. Catal.* 206 40–48.

[17] Selvamani T, Yagyu T, Kawasaki S and Mukhopadhyay I 2010 *Catal. Commun.* 11 537–541.

[18] Bahadur J, Agrawal S, Panwar V, Parveen A and Pal K 2016 *Macromol. Res.* 24 488–493.

[19] Phuoc T X, Howard B H, Martello D V, Soong Y and Chyu M K 2008 *Opt. Lasers Eng.* 46 829–834.

[20] Rastogi C K, Saha S, Sivakumar S, Pala R G S and Kumar J 2015 *Phys. Chem. Chem. Phys.* 17 4600–4608.