A facile synthesis of nano-magnesia by ultrasonication assisted co-precipitation method for antibacterial activity

Aqsa Amir[1], Muhammad Faisal[2], Muhammad Asif Hussain[3] , Ehsan Ul Haq[3], Kabeer Raza[2], and Zaeem ur Rehman[2]

[1]Institute of Advanced Materials, Bahauddin Zakariya University, Multan, Pakistan
[2]Department of Metallurgical and Materials Engineering, University of Engineering and Technology, Lahore, Pakistan
[3]Institute of Metallurgy and Materials Engineering, University of the Punjab, Lahore, Pakistan

Abstract. Magnesium oxide (MgO) nanoparticles found considerable interest from the researcher because of their versatile biocompatible properties and the plethora of applications including anticancer, antimicrobial, antidiabetic, drug delivery, and tissue engineering etc. The growing applications of the MgO nanoparticles necessitate exploring new synthesis routes with faster production rates. Method: In this study, MgO nanoparticles were synthesized by ultrasonication-assisted co-precipitation method and calcined at 800 °C. MgO nanoparticles were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), and energy-dispersive X-ray spectroscopy (EDS) analysis. XRD results showed that the particles have a body-centered cubic (BCC) structure with a crystallite size of about 19.07 nm. SEM results displayed the spherical morphology of MgO nanoparticles. The impurity elements were absent as determined through EDX analysis and showed the high purity of the synthesized MgO. These particles are tested for in-vitro biological applications. The antibacterial activity of MgO nanoparticles on different bacteria was determined by the minimum inhibitory concentration (MIC) test. MIC test revealed that antibacterial activity increases by increasing the concentration of MgO nanoparticles. The synthesized nano-MgO showed high purity and spherical morphology and characterization analysis revealed that nano-MgO and biocompatible and can be applied in biomedical applications as verified by their bacterial activity test.

1 Introduction

Recently, the advancement in nanoscience and nanotechnology have created a revolution in the technological world, which focuses on materials having innovative and enhanced biological, physical, and chemical characteristics [1]. Nanoparticles of metal oxides have exceptional photocatalytic, and hydrophobic properties and their stability is also remarkable [2]. Consequently, materials with these advanced properties found applications in different fields such as antibacterial agents, coatings, catalysis, batteries, implants for medical concerns, semiconductors, and capacitors [3–5]. So, the antibacterial effectiveness and bioactivity of inorganic metal oxides can be enhanced further through nanotechnology [6]. Contaminations caused by bacteria remain the main concern as according to an estimate approximately 48 million pathogenic disease cases are attributed to bacteria. To control the bacterial population, antimicrobial agents with enhanced activity are necessary to be developed [7]. Oxides of metals in Nano form such as Calcium oxide (CaO), Magnesium oxide (MgO), Titanium oxide (TiO_2), and Zinc oxide (ZnO), etc. have been examined as inorganic anti-bacterial oxides [8]. Magnesium oxide MgO nanoparticles hold special interest among all examined inorganic metal oxide. The nano MgO has been utilized in catalysis, electronics, petrochemical, and corrosion and wear resistance coatings [9]. Wood chips and different shavings are used with MgO nanoparticles to construct materials that are sound-proof, lightweight, and heat-insulating [10]. MgO nanoparticles are applied in refractory material, fibers for refractories, coating filler, instruments for refractory and insulations, magnesite-chrome brick, smelting furnaces, additional furnaces used at high temperatures, and base plates of ceramics [11]. MgO finds a wide variety of applications in biomedical applications due to its stability under severe processing environments and it is considered a safe material for human beings due to its biocompatible and biodegradable properties. MgO finds applications in toxic waste remediation, biopaint, hard tissue implants, scaffolds, and anti-bacterial agents against food-borne pathogens [12]. MgO is preferred over TiO_2 because it does not require photoactivation to display anti-microbial activity without photo-activation, while Titanium oxide TiO_2 necessitates photo-activation [13].

Various methods including chemical, physical, and biological methods have been applied to produce nanoparticles of MgO [14]. The most abundantly used techniques to synthesize the nanoparticles of MgO comprise sol-gel, co-precipitation, solvo- and hydro-thermal, chemical reduction, and microemulsion [11]. Sol-gel and co-precipitation techniques have been used most of all due to their simplicity in processing and the precursors used. Although the purity level of the MgO produced through these methods is high, the production rate is low. Therefore, these processes need modifications for higher yields of MgO. In the current study, the ultrasonication-assisted co-precipitation technique was used to prepare nanoparticles of MgO. The MgO nanoparticles were tested for antibacterial activity against *Escherichia coli (E. colli), Staphylococcus aureus (S. aureus), Pseudomonas aeruginosa (P. aeruginosa), Klebsiella Spp,* and *Staphylococcus epidermidis (S. epidermidis).*

2 Experimental

2.1 Synthesis of MgO

The Magnesium nitrate ($Mg(NO_3)_2$ and sodium hydroxide (NaOH) were purchased from Atta Chemicals (Pvt.) Limited and were used in as received form. Two separate solutions of $MgNO_3$ and NaOH were prepared in 250 mL distilled water. The constant stirring was done for a few minutes in an ultrasonication bath. The $MgNO_3$ solution was sonicated and NaOH solution was dropped in with a burette at room temperature (Reaction Eq. 1). After a few minutes milky white precipitates appeared indicating the synthesis of MgO. The solution was sonicated for a few min to complete the reaction. The resultant solution was centrifuged and dried in an oven at 110 °C for 5 h (Reaction Eq. 2). The resultant solid cake was ground in mortar and pestle. The crystalline MgO powder was achieved by calcining at 800 °C for 8 h.

$$Mg(NO_3)_2 + 2NaOH \rightarrow Mg(OH)_2 + 2NaNO_3 \quad \text{(Eq. 1)}$$

$$2Mg(OH)_2 \rightarrow 2MgO + H_2O \quad \text{(Eq. 2)}$$

2.2 Characterization and Testing of MgO

X-ray diffraction patterns were acquired by X-ray diffractometer (BRUKER-AXS, D8-ADVANCED, Japan) with step size 0.1° per sec. The range of the XRD pattern was 20 ° to 90°. Peaks of MgO nanoparticles were obtained using X-rays of alpha copper having wavelength 1.54 Å. The resultant pattern was compared with standard samples and crystallite size was determined by Debye Scherrer equation. The morphological information of the powder was obtained by Scanning Electron Microscopy (SEM) by SEM: TESCAN LYRA3. The elemental analysis of the MgO powder was carried out with Energy Dispersive X-Ray (EDX) detector INCA Energy 200, (Oxford Instruments, UK) attached with SEM.

The antibacterial activity of MgO nanoparticles was determined by the minimum inhibitory concentration (MIC) test. MIC test was carried out as follows: the MgO nanoparticles stocks were prepared at 10,000 µg/mL concentration. Mueller Hinton agar solution was prepared and autoclaved the media were cooled to 50 °C in a water bath. The MgO nanoparticles were added to reach the final concentrations as given in Table 1. The plates were made, and organisms were cultured on MgO nanoparticles supplemented on plates.

3 Results and Discussion

3.1 X-Ray Diffraction Analysis

XRD results were obtained in the range of 10° to 90° as shown in Figure 1. Miller indices of the peaks and angles are (111) at 37.15°, (200) at 43.11°, (220) at 62.46°, (311) at 74.79°, and (222) at 78.76° [15]. XRD pattern confirmed the BCC crystal structure for MgO. The lattice parameter of synthesized MgO was found 4.212 experimentally and compared with the standard pattern of MgO (JCPDS Card number 4-829). The experimental value of the lattice parameter was close to the standard value which is 4.213. There was no extra peak present in the obtained pattern and all peaks matched with the standard pattern of MgO (JCPDS Card number 4-829) which proved the purity of the synthesized MgO nanoparticles [16]. The average crystallite size was 19.07 nm as determined by the Debye Scherrer formula.

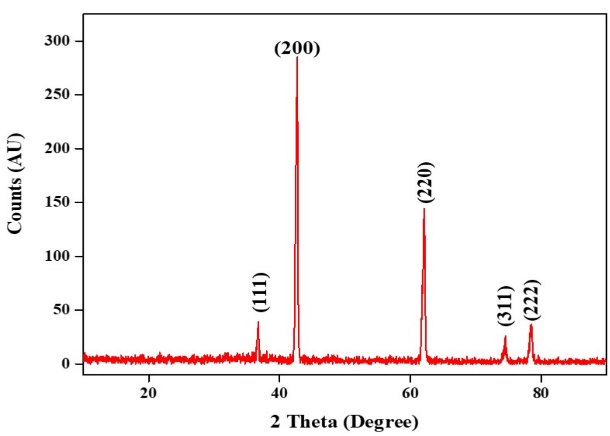

Fig. 1 XRD spectrum of the synthesized and calcined MgO nanoparticles

3.2 SEM & EDX Analysis

SEM was used to identify the morphology of MgO nanoparticles in Figure 2. The micrograph of SEM represents that the MgO nanoparticles are in the form of globules. The aggregates of MgO nanoparticles were present as evident from the SEM image, however, particle size is nanometer range. The particle size results of SEM analysis reinforce the XRD results where crystallite size was found in the nanometer range as well. Hence, nanoparticles of MgO were synthesized successfully by the ultrasonication co-precipitation method in a shorter time than the simple co-precipitation method.

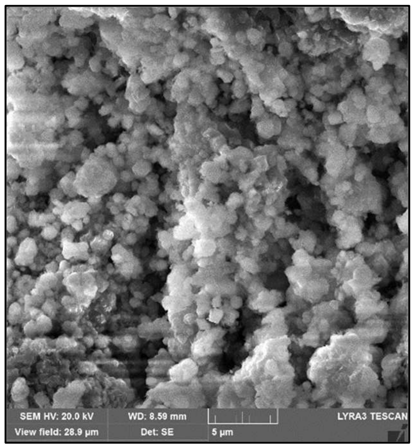

Fig. 2 SEM micrograph of MgO nanoparticles showing the spherical morphology.

EDX spectrum was used to evaluate the elemental composition of synthesized MgO nanoparticles. Figure 3 demonstrates the EDX graph of MgO which confirmed the Mg and O elements presence (carbon tape used for SEM and EDX analysis). Since the antibacterial activity of the MgO nanoparticles can be affected by the presence of impurity elements. A small amount of impurity can badly affect the biocompatibility of the MgO nanoparticles. No extra peak of impurity element was detected as shown in the EDX spectrum. The results acquired by the XRD and EDX analysis confirmed the purity of the MgO nanoparticles. Therefore, synthesized MgO nanoparticles can be applied for antibacterial analysis because of their purity and biocompatibility.

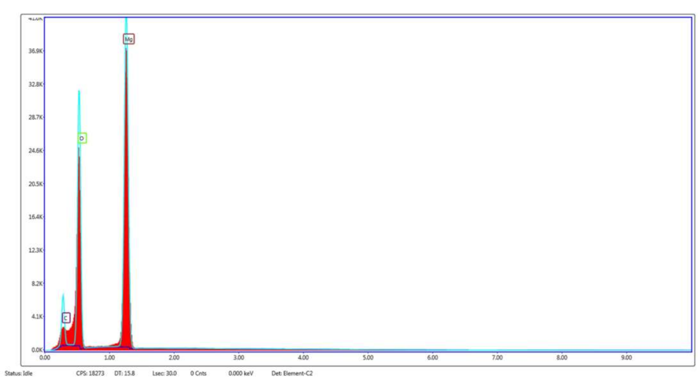

Fig. 3 EDX spectrum of MgO nanoparticles

3.3 Antibacterial Activity Analysis

The results of antibacterial activity were determined by minimum inhibitory concentration (MIC) as shown in Table 1. The MIC tests were conducted on five different photogenic bacteria with different concentrations of MgO nanoparticles and they showed different antibacterial activity against each concentration. MIC results revealed that the activity of the MgO nanoparticles boosted with increasing concentration of MgO. MgO nanoparticles with 1x concentration were effective on only two bacteria (i.e., *S. aureus*, and *S. epidermidis*) while the other three remained active as shown in Table 1. When the concentration of MgO nanoparticles was increased to 2x and 3x, their effectiveness increased and particles with these concentrations killed all bacteria as shown in Table 1. From the MIC test, prepared MgO nanoparticles can be used for antibacterial applications for five common pathogens where MgO worked more efficiently against *S. aureus* and *S. epidermidis*.

Table 1: MIC results for prepared MgO nanoparticles to check the antibacterial activity of the five common pathogens.

Conc. of MgO	*S. aureus*	*P. aeruginosa*	*S. epidermidis*	*E. colli*	*Klebsiella Spp.*
1x	Yes	No	Yes	No	No
2x	Yes	Yes	Yes	No	No
3x	Yes	Yes	Yes	Yes	Yes

4 Conclusions

MgO nanoparticles were synthesized successfully by ultrasonication-assisted co-precipitation method in a shorter time. The synthesized MgO nanoparticles demonstrated spherical morphology. The crystallite size of the MgO was 19.07 nm. Lattice parameters of synthesized MgO nanoparticles are approximately equal to the standard values and no impurity was detected. Anti-bacterial activity found by the MIC test represented that as the concentrations of the MgO increased the anti-bacterial activity was enhanced.

The authors extend their acknowledgment to the Bahauddin Zakariya University, Multan, and the University of the Punjab Lahore, Pakistan for their technical support in completing this work.

References

1 S. Bayda, M. Adeel, T. Tuccinardi, M. Cordani, F. Rizzolio, Mol. **25,** (2019).
2 M.S.S. Danish, L.L. Estrella, I.M.A. Alemaida, A. Lisin, N. Moiseev, M. Ahmadi, M. Nazari, M. Wali, H. Zaheb, T. Senjyu, Met. **11,** (2021).
3 M. Asif Hussain, M. Yang, H.-S. Jang, S.-Y. Hwang, B.-H. Um, B.G. Choi, J.W. Kim, Ind. Eng. Chem. Res. **55,** (2016).
4 Z. Ahmad, A. Maqbool, M.A. Hussain, R.A. Pashameah, A. Shahzadi, N. Nazar, S. Iqbal, A.K. Alanazi, M.N. Ashiq, H.M. Abo-Dief, Fuel **331,** (2023).
5 K. Mageshwari, S.S. Mali, R. Sathyamoorthy, P.S. Patil, Powder Technol. **249,** (2013).
6 T. Shabatina, O. Vernaya, A. Shumilkin, A. Semenov, M. Melnikov, Mater. **15,** (2022).
7 L. Serwecińska, Water. **12,** (2020).
8 A. Nigam, S. Saini, A.K. Rai, S.J. Pawar, Ceram. Int. **47,** (2021).
9 L.I.U. He, J.I.N. Xu, D. Bin, Pet. Explor. Dev. **43,** (2016) 1107–1115.
10 S.L. Daisy, A.C.C. Mary, K. Devi, S.S. Prabha, S. Rajendran, S.S. Zahirullah, Int. J. Nano Corr. Sci. Eng. **2**, (2015).

11 J. Hornak, Int. J. Mol. Sci. **22**, (2021).
12 S. Abinaya, H.P. Kavitha, M. Prakash, A. Muthukrishnaraj, Sustain. Chem. Pharm. **19**, (2021).
13 D.F. Emerich, C.G. Thanos, Expert Opin. Biol. Ther. **3**, (2003).
14 M. Nejati, M. Rostami, H. Mirzaei, M. Rahimi-Nasrabadi, M. Vosoughifar, A.S. Nasab, M.R. Ganjali, Inorg. Chem. Commun. **136**, (2022).
15 M.A. Hussain, M. Faisal, K. Raza, M.A. Rafiq, M. Usman, W. Amin, A. Maqbool, B.A. Ahmed, Ceram. Int. **50**, (2024).
16 K.G. Rao, C.H. Ashok, K.V. Rao, C.S. Chakra, Int. J. Sci. Res. **3**, (2014).