

RESEARCH ARTICLE

Mechanosynthesis, Characterization and Antibacterial Activity of MgO@SiO₂ Nanocomposite

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ABSTRACT

MgO@SiO₂ nanocomposite was synthesized using mechanochemical method and its formation was confirmed by FTIR and Uv-visible spectroscopic techniques. The antibacterial effect of MgO@SiO₂ nanocomposite was carried out on bacterial isolates; gram-positive bacteria (*Bacillus subtilis*, *Klebsiella pneumoniae*) and gram-negative bacteria (*Pseudomonas aeruginosa*, *Escherichia coli*, and *Salmonella typhi*) using Agar well diffusion method. The results showed that MgO@SiO₂ nanocomposite can find application as antibiotics against the investigated microbes.

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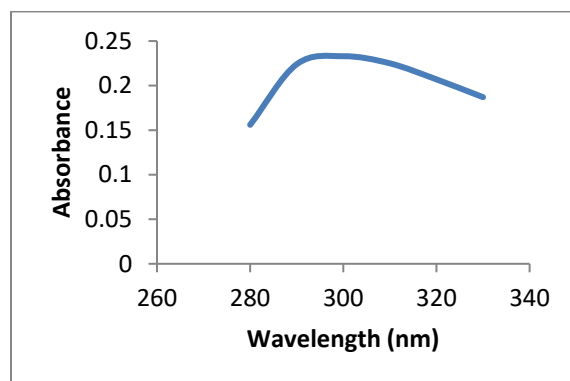
INTRODUCTION

In nanoscience, the most important factor is the fabrication of nanoparticles. Nanoparticles can be prepared by either top up methods; examples include chemical etching, grinding, ball milling, thermal/laser ablation and sputtering and or bottom up methods which includes; Chemical/Electrochemical, precipitation, vapour deposition, atomic/molecular condensation, sol gel processes, spray pyrolysis, laser pyrolysis and aerosol pyrolysis as well as green synthesis (such as using Bacteria, fungi, plant extracts etc). Currently, bottom up methods are widely used in preparation of nanoparticles but top up methods remains relevant and efficient [1]. Thus, one of the simplest ways of synthesizing nanoparticles/nanocomposites of some metals, metal oxide and alloys in the form of powder is mechanical method. Mechanochemical

reactions are defined by the reactions induced through mechanical energy. The reactions occur between solids with no, or minimal, addition of solvents. There are various types of grinding mills such as planetary, vibratory, rod, tumbler etc with one or more balls of various sizes placed in together with the reagents. The mechanical milling could be easily carried out with mortar and pestle technique/method [2, 3].

Resistance of bacteria to different classes of drugs and disinfectants has attracted public attention. And to solve the problem, an antibacterial agent that is highly efficient must be developed. An antibacterial agent could be organic or inorganic and or their combination. Organic antibacterial agents like organic acids, essential oils, bacteriocins and enzymes have been widely used but bacteria have developed resistance to it. Thus, inorganic antibacterial agents have attracted much interest

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Fig. 1. Uv-visible spectrum for MgO@SiO₂

for bacterial control [4]. Recently, some inorganic nanoparticles and nanocomposites such as Mg(OH)₂, MgO, MgO-SiO₂, CdO-MgO, CdO-ZnO-MgO, magnesium ferrite spinel nanoparticles and Kaolin/Gum Arabic were synthesized and applied as antibacterial agents[5-15].

Therefore, the focus of this study is to synthesize MgO@SiO₂ nanocomposite using mortar and pestle and test its antibacterial potency.

MATERIALS AND METHODS

Apparatus/Instruments

For this research the following apparatus that were used ; weighing balance, whatman filter paper, mortar and pestle, sieve, beakers, conical flasks, test tubes, measuring cylinder, watch glass, oven, funnels, hot plate, autoclave, petri dish, incubator, refrigerator, laminar flow cabinet, cotton wool, aluminium foil paper, candle, matches, universal container, cork borer, metre rule, masking tape, spatula, glass rod stirrer, syringes, micropipette, wire loop, Ultraviolet -Visible spectrophotometer, SEM, and FTIR.

Reagents

The reagents used in this research include, MgO, Silica gel, Ethanol, Hydrochloric acid (HCl), Tetraoxosulphate (VI) acid (H₂SO₄), barium chloride (BaCl₂), Normal saline, nutrient Agar, and Muller Hinton agar.

Synthesis of MgO@SiO₂ Nanocomposite

Mechanochemical synthesis method was used in synthesizing MgO@SiO₂ nanocomposite in accordance as reported by some researchers [2,

3]. MgO@SiO₂ nanocomposite was prepared according to 1:1 ratio of MgO to silica gel as a precursor of SiO₂. The appropriate ratio of the precursors was mixed and grounded using mortar and pestle for one hour to obtain homogeneous mixture. The homogeneous mixture was calcined at 500°C in the furnace for 2 hours in order to get rid of water molecules associated with the precursors. Finally, the product was removed from furnace, cooled and stored for further analysis.

Antimicrobial Activity Assay

The antimicrobial activity was tested by using the agar-well diffusion method as described by Shehu et al.2018 [15]. The bacterial isolates; gram-positive bacteria (*Bacillus subtilis*, *Klebsiella pneumoniae*) and gram-negative bacteria (*Pseudomonas aeruginosa*, *Escherichia coli*, and *Salmonella typhi*) were first grown in a nutrient broth for 12–18 h before use and standardized to 0.5 McFarland standards (10⁶ cfu ml⁻¹). One hundred microliter of the standardized cell suspensions were spread on a Mueller-Hinton agar (Hi Media) and the agar medium was punched with a 6 mm diameter wells and filled with different concentration(100, 200, 300, 400 and 500 µg/L) of MgO@SiO₂ nanocomposite solutions in equal amounts. The plates are observed for zone of inhibition after 24 h incubation at 37°C.

Characterization

The synthesized MgO@SiO₂ nanocomposite was characterized using spectroscopic techniques; Fourier Transform Infra-red (FTIR) and Ultraviolet-Visible spectroscopy.

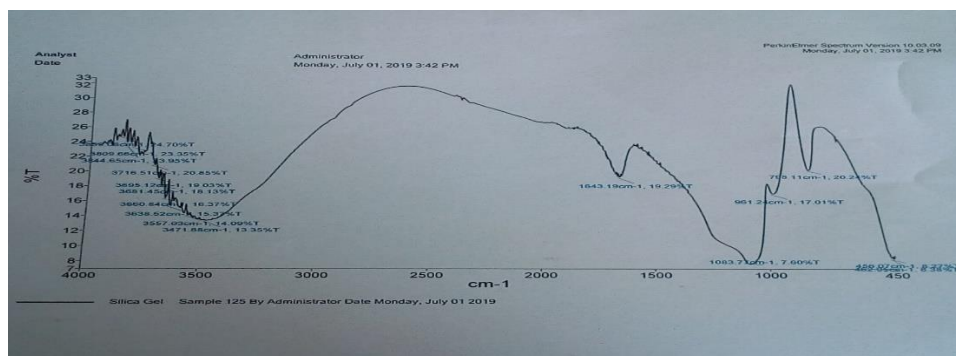


Fig. 2. FTIR spectrum of Silica gel

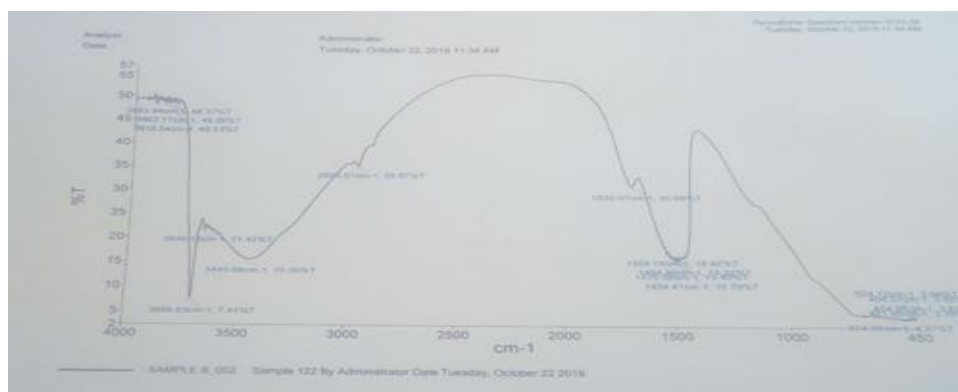


Fig. 3. FTIR spectrum of MgO

RESULTS AND DISCUSSION

UV/Visible spectroscopy

The UV/Visible spectrum of MgO@SiO₂ Nanocomposite is shown in Fig.1 which showed surface plasmon resonance at 300nm. Elsewhere the surface plasmon resonance for silica has been reported to be 270nm [10].

Fourier Transform Infra Red (FTIR) spectroscopy

The functional groups analyses were carried out using FTIR spectrometer. The FTIR of the precursor, silica gel is shown in Fig. 2. The peaks observed includes; 3809.65 – 3471.85, 1643.19, 1083.71, 961.24, 788.11, 456.07, and 462.05 cm⁻¹. The band at 1083.71 cm⁻¹ corresponds to asymmetric stretching vibration of Si-O-Si bond. The peaks at 961.24 and 788.11 cm⁻¹ corresponds to Si-OH bond. The peaks at 3809.65 – 3471.85 and 1643.19 cm⁻¹ indicates H-O-H stretching and bending of adsorbed water. The peaks at 456.07, and 462.05 cm⁻¹ correspond to Si-O bond. These reports are

in agreement with previous reports [5, 11, 16, 17]. The FTIR spectra of the second precursor, MgO, Fig. 3, shows the bands at 3898.94-3443.68 cm⁻¹, 3698.83 cm⁻¹, 2925.01 cm⁻¹, 1635.07 cm⁻¹, 1504.07-1434.40 cm⁻¹, 454.85-481.94 cm⁻¹, 524.12-574.95 cm⁻¹. The peaks at 3898.94 – 3443.68 and 1635.07 cm⁻¹ indicates H-O-H stretching and bending of adsorbed water. The sharp band at 3698.83 cm⁻¹ corresponds to non bonded hydroxyl on the surface of MgO which is the obvious feature of MgO. The peak at 2925.01 cm⁻¹ indicates C-H stretching of alkane and its presence could be probably due to solvent or stabilizing agent used during synthesis of MgO. The peaks at 1504.07-1434.40 cm⁻¹ represent carbonate in carbonyl group. The peaks at 454.85-481.94 cm⁻¹, and 524.12-574.95 cm⁻¹ confirm the presence of Mg-O vibration. The FTIR spectra of MgO@SiO₂, Fig. 4, showed almost similar bands, however, with few exceptions. Thus, the peaks at 961.24 and 788.11 cm⁻¹ corresponds to Si-OH bond (free silanol group) which is present

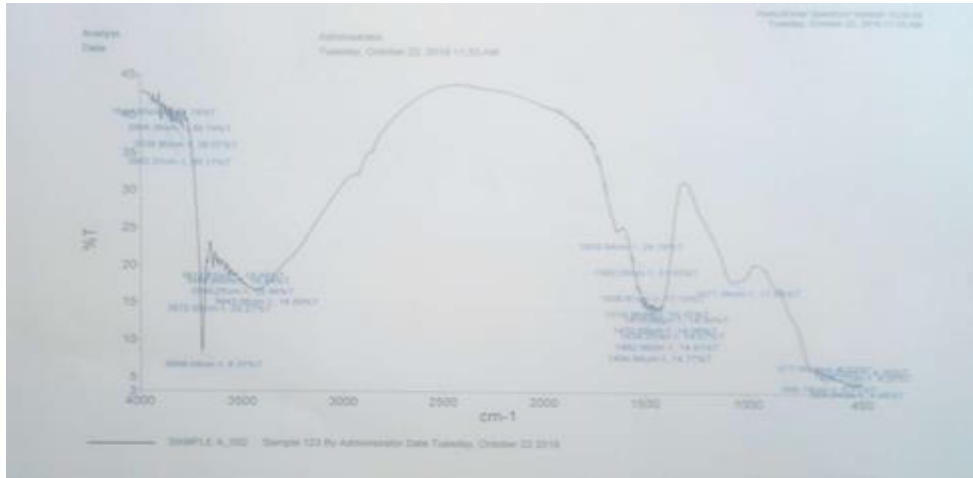


Fig. 4. FTIR spectrum of MgO@SiO₂

Table 1. Antibacterial results

Concentrations of MgO@SiO ₂ (µg/L)	Zone of inhibition(mm)				
	<i>E. coli</i>	<i>B.subtilis</i>	<i>P.aeruginosa</i>	<i>K.pneumoniae</i>	<i>S. typhi</i>
200	6	8	12	6	13
300	7	9	14	7	17
400	12	10	17	8	18
500	17	13	20	10	22
300µg/L, Augmentin (control)	18	20	26	20	28

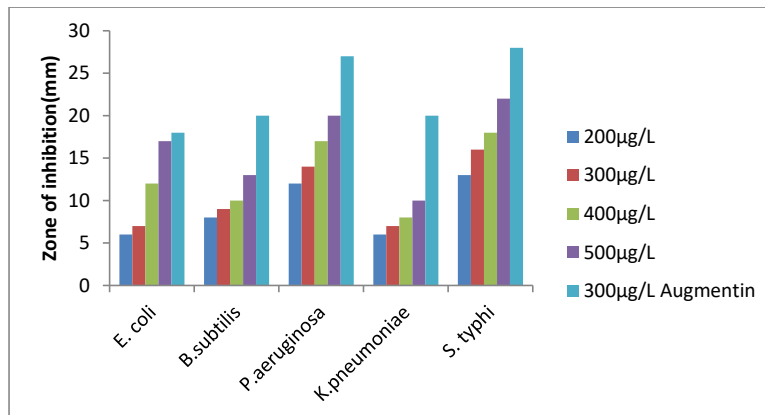


Fig. 5. Zone of inhibition of MgO@SiO₂ nanocomposite against pathogens

in the silica gel, Fig. 2 were absent in the MgO@SiO₂ nanocomposite spectra. This indicates that molecules of water were removed after calcination. This is due to fact that free silanol group which is responsible for adsorption of water molecules was removed after calcination [16].

Antibacterial Results

Result of antibacterial studies of MgO@SiO₂ nanocomposite against *Bacillus subtilis*, *Klebsiella pneumoniae*, *Pseudomonas aeruginosa*, *Escherichia coli*, and *Salmonella typhi* are presented in (Table 1 and Fig. 5). Augmentin was used as control

throughout the studies at concentration of 300 300µg/L. Different concentrations of 200, 300, 400 and 500µg/L of MgO@SiO₂ nanocomposite was tested against each pathogen. Generally, the inhibition zone increases with increase in concentrations of MgO@SiO₂ nanocomposite of all the bacteria. Similar observations were report by Shehu et al. 2018 [17]. At higher concentration of 500µg/L, the zones of inhibition were in the following order; 22, 20, 17, 13, and 10 mm for *S. typhi*, *P.aeruginosa*, *E. coli*, *B.subtilis* and *K.pneumoniae* respectively. The zone of inhibition for control (Augmentin) was found to be higher compared to MgO@SiO₂ nanocomposite for each pathogen except for *E. coli* where it is almost the same, Fig. 5. This indicates high activity of MgO@SiO₂ nanocomposite against *E. coli* due to the comparability with Augmentin. For each concentration investigated, *S. typhi*, showed higher zone of inhibition as compared to other pathogens, Table 1 and Fig. 4. This shows that MgO@SiO₂ nanocomposite is more effective against *S. typhi* than any other pathogen under investigation for this study. According to Ehi-Eromosele et al.[14], antibacterial activity of MgFe₂O₄ against *E. coli*, *P.aeruginosa*, and *B.subtilis* were 17mm, 12mm and 0mm respectively. For *E. coli*, the result is similar this study but for *P.aeruginosa*, and *B.subtilis*, the current study showed higher activity. MgO nanostructure was synthesized, characterized and its antibacterial studies were investigated. The antibacterial activity of the MgO against for *S. typhi*, *E. coli*, and *B.subtilis* were found to be 15mm, 16mm and 25mm respectively,[9]. For *S. typhi*, and *E. coli*, current study showed higher antibacterial activity whereas for *B. subtilis*, it has high activity than this study. These results showed that MgO@SiO₂ nanocomposite can find application as antibiotics against the investigated microbes.

CONCLUSION

MgO@SiO₂ nanocomposite was synthesized using mechanochemical method and its formation was confirmed by FTIR and Uv-visible spectroscopic techniques. The antibacterial effect of MgO@SiO₂ nanocomposite was carried out on bacterial isolates; gram-positive bacteria (*Bacillus subtilis*, *Klebsiella pneumoniae*) and gram-negative bacteria (*Pseudomonas aeruginosa*, *Escherichia coli*, and *Salmonella typhi*). The results showed that MgO@SiO₂ nanocomposite can find application as antibiotics against the investigated microbes.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

REFERENCE

1. Shehu Z and Lamayi DW. Recent Advances and Developments in Nanoparticles/ Nanocomposites as Nanoadsorbent for Adsorptive Removal of Lead in Wastewater: A Review. *Nanomed Nanotechnol*, 2019; 4(3): 1-10.
2. Patil, G.G. and Sayyad S. B. Study the Synthesis of CuO/ZnO nanocomposite by Mortar and Pestle and its Characterization, *Journal of Emerging Technologies and Innovative Research*, 2017;4(11):543-546.
3. Florence S.S., Adam H, Manna C., and Can N. Green approach to synthesis and strain studies of ZnO nanoparticles, *AIP Conference Proceedings* **1976**, 020020, 2018:1-4, <https://doi.org/10.1063/1.5042387>.
4. Zhen-Xing T. and Bin-Feng L. MgO nanoparticles as antibacterial agent: preparation and activity, *Brazilian Journal of Chemical Engineering*, 2014; 31(3):591 – 601, [dx.doi.org/10.1590/0104-6632.20140313s00002813](https://doi.org/10.1590/0104-6632.20140313s00002813).
5. RSONIYA S. and NAIR V.M. Synthesis and Characterization of Nanostructured Mg(OH)₂ and MgO, *International Journal of Science and Research (IJSR)*, 2016;5(2):199-203.
6. Shah M.A. and Al-Marzouki F.M. A simple and safe method for preparation of Mg(OH)₂ nanorods in ambient air, *Int.J.Nano Dim.* 2011;2(2): 111-116.
7. Dong H., Unluer C., Al-Tabbaa A., and Yang E. Characterization of MgO Calcined from Mg(OH)₂ Produced from Reject Brine, *Fourth International Conference on Sustainable Construction Materials and Technologies, SCMT4*, Las Vegas, USA, August, 2016; 7-11.
8. Meenakshi S.D., Rajarajan M., Rajendran S., Kennedy Z.R. and Brindha G. Synthesis and characterization of magnesium oxide nanoparticles, *Elixir Nanotechnology*, 2012; 50:10618-10620
9. Karthik K., Dhanuskodi S., Gobinath C., Prabukumar S., Sivaramakrishnan S. Fabrication of MgO nanostructures and its efficient photocatalytic, antibacterial and anticancer performance, *Journal of Photochemistry & Photobiology, B: Biology*, 2019;190:8–20
10. Hadiza AM, Taura DW, Bashir Muhammad. Preparation, Characterization and *In-Vivo* Antiplasmodial Activity of Magnesium Oxide Nanoparticles on *Plasmodium Berghei* Infected Mice. *Nano Tech Appl.* 2018; 1(1): 1-5.
11. Moghaddam S.H.H., Jebali A., and Daliri K. The use of MgO-SiO₂ nanocomposite for adsorption of aflatoxin in wheat flour samples, *NANOCON*, Olomouc, Czech Republic, EU, 2010;12 -14.
12. Karthik K., Dhanuskodi S., Gobinath C., Prabukumar S. & Sivaramakrishnan S. Ultrasonic-assisted CdO-MgO nanocomposite for multifunctional applications,

- Materials Technology, 2019 34:7, 403-414, DOI: 10.1080/10667857.2019.1574963
13. Revathi V. and Karthik K. Microwave assisted CdO–ZnO–MgO nanocomposite and its photocatalytic and antibacterial studies, *Journal of Materials Science: Materials in Electronics* (2018) 29:18519–18530, <https://doi.org/10.1007/s10854-018-9968-1>.
 14. Ehi-Eromosele C.O., Olugbuyiro J.A.O., Taiwo O.S., Bamgboye O.A. and C.E. Ango O.A. Synthesis and evaluation of the antimicrobial potentials of Cobalt doped- and magnesium ferrite spinel nanoparticles, *Bull. Chem. Soc. Ethiop.* 2018;32(3):451-458, DOI: <https://dx.doi.org/10.4314/bcse.v32i3.4>.
 15. Shehu Z., Lamayi D.W., Sabo M.A., and Shafiu M.M.,^{*} Synthesis, Characterization and Antibacterial Activity of Kaolin/Gum Arabic Nanocomposite on Escherichia Coli and Pseudomonas Aeruginosa”, *Research Journal of Nanoscience and Engineering*, 2018;2(2):23-29.
 16. Christy A. A. Effect of Heat on the Adsorption Properties of Silica Gel, *IACSIT International Journal of Engineering and Technology*, 2012;4(4):484-488.
 17. Agnieszka P., Iwona L., Marcin W., I Dominik P., Teofil J. Synthesis of Mg(OH)₂ from magnesium salts and NH₄OH by direct functionalisation with poly(ethylene glycols), *Physicochem. Probl. Miner. Process.* 2012; 48(2):631–643.