Synthesis and Characterization of Zinc Oxide Doped Mesoporous Silica SBA-16 and Anti-bacterial Property

Nidhi¹, Sunita Dahiya¹, Atul Kumar² and Surender Duhan²

¹Department of Physics, Baba Mastnath University, Asthal Bohar, Rohtak, Haryana, India

²Department of Physics, Deenbandhu Chhotu Ram University of Science and Technology, Murthal, Sonapat, Haryana, India

Corresponding author email: surender6561@gmail.com

ABSTRACT

In this research, article SBA-16 was synthesized by using hydrothermal process and ZnO nanoparticles by using sol-gel method. ZnO nanoparticles were introduced further into the mesopores of SBA-16 through post impregnation method. The doping of ZnO nanoparticles into SBA-16 was confirmed by their Fourier Transformed Infrared Spectroscopy (FTIR) analysis, X-ray diffraction (XRD), and small-angle X-ray Scattering graph (SAXS). The morphological analysis of the prepared sample was done by Scanning Electron Microscopy (SEM), and their lattice cell parameter with symmetry was analyzed by performing Transmission Electron Microscope (TEM). The anti-bacterial property of SBA-16-ZnO nanocomposite was examined against *E. coli* and *S. aureus* bacteria.

KEY WORDS: ZNO, SBA-16, SBA-16-ZNO NANOCOMPOSITE, ANTI-BACTERIAL PROPERTY, ETC.

INTRODUCTION

SBA-16 is a modern mesoporous, large cage, Im3m silicium substance with large pores and high hydrothermal stability. The SBA-16 also exhibits high thermal and mechanical stability and is environment friendly, making it a suitable material for a wide range of applications. It can be applied as a support material of catalysts and planning for the production of orderly mesoporous carbon in environmental treatment for adsorption and separation. Its practical uses, including catalysts, absorbents, and sensors, have been extensively published. However, the study has not been published thoroughly on many morphologies of mesoporous SBA-16 materials. The synthesis of mesoporous materials with other materials changes in synthetic sources, stirrings, and reaction temperatures may achieve morphologies.

The Zinc Oxide Nanoparticle (ZnO) is a wideband semiconductor that attracts considerable attention to fundamental studies and possible applications in different research areas. ZnO is a versatile, functional material, with its superior qualities being applied potentially in catalysts, transducers, semiconductors, microelectronics, textiles, cosmetics, water treatment, and others. In addition to antibiotics and bactericides, ZnO displays antimicrobial activity and anti-inflammatory characteristics that provide more efficient, less expensive, and less toxic alternatives. In recent years, several groups have produced the preparation of nano-size ZnO particles through sol-gel methods (Kumar et al., 2020).

Antibacterial activity against Gram-Positive gram-negative bacteria is shown by nanoparticles of zinc oxide. Nanoparticles with zinc oxide are thought to be non-toxic, bio-safe, and biocompatible. SBA-16-ZnO nanocomposite's antibacterial activity has not previously been investigated against *E.coli* and *S. aureus*. Although specific assertions were suggested, such as hydrogen peroxide development, the critical factor of the antibacterial activity or the attachment of ZnO particles on the Bacterial Surface could be a mechanism. The mechanisms of ZnO's antibacterial activity are not fully known. This work, therefore, tries to manufacture

Biosc Biotech Res Comm P-ISSN: 0974-6455 E-ISSN: 2321-4007

Identifiers and Pagination

Year: 2021 Vol: 14 No (9) Special Issue

Pages: 194-197

This is an open access article under Creative

Commons License Attribn 4.0 Intl (CC-BY).

DOI: <http://dx.doi.org/10.21786/bbrc/14.9.37>

Article Information

Received: 06th May 2021

Accepted after revision: 18th July 2021

nanocomposites SBA-16-ZnO and verify their vigorous antibacterial activity for bacteria of *E.coli* and *S. aureus* (Sirelkhatim et al., 2015).

In this research, article SBA-16 was synthesized by using hydrothermal process and ZnO nanoparticles by using sol-gel method. ZnO nanoparticles were introduced further into the mesopores of SBA-16 through post impregnation method. The doping of ZnO nanoparticles into SBA-16 was confirmed by their Fourier Transformed Infrared Spectroscopy (FTIR) analysis X-ray diffraction (XRD) and small-angle X-ray Scattering graph (SAXS). The morphological analysis of the prepared sample was done by Scanning Electron Microscopy (SEM), and their lattice cell parameter with symmetry was analyzed by performing Transmission Electron Microscope (TEM). The research work shows successful preparation of ZnO doped SBA-16, which can be significantly applicable in several industrial applications such as catalyst, anti-biofilm industries, targeted drug delivery, etc. The results will assist in discovering an appropriate alternative for controlling *E.coli* and *S. aureus* infection.

2. Experimentation

2.1. Synthesis of SBA-16: SBA-16 was synthesized to retain the pH of solution 5.94 gm 2M HCL in the solution by dissolving 3gm Pluronic P127 in 144 gm distilled water. 9gm n-butanol was applied carefully into the solution and stirred the solution for the next 3 hours to prepare a clear solution. After 3 hrs of stirring, 14.2gm TEOS dropwise applied to the earlier stirring solution and stirred at a temperature of 38°C for another 24 hours. The processed hydrothermal solutions were applied to an autoclave with Teflon and placed in the microwave autoclave at 100°C for 24 hours. The final stage was washed three times with water purified and calcinated for 5 hours at 550°C (Praseptiangga et al., 2020).

2.2. Synthesis of SBA-16/ZnO Nanocomposite: SBA-16/ZnO2 nanocomposite has been prepared by impregnation of zinc acetate solution into SBA-16. The synthesis process was performed by adding 0.901g zinc acetate into 42 ml ethanol and stirred for 2 hours at 70°C. The solution was cooled to room temperature, which resulted in a heavy stirring of 0.5 g of SBA-16. In order to extract the contaminant, the resulting blend has been dried for 24 hours at 80°C. Samples were consequently calcinated in the air by rising ambient temperature over 1 hour and keeping 550 °C for 1 hour from room temperature to 550 °C (González-Rodríguez et al., 2020).

2.3. Anti-bacterial Evaluation: The antibacterial effect of SBA-16-ZnO nanocomposite against *E.coli* and *S. aureus* bacteria was evaluate using the previously mentioned procedure. Discs of SBA-16-ZnO nanocomposite and SBA-16 were prepared, and distilled water disks were employed as control. The culture was kept for overnight incubation at 37°C. The measurement of inhibition-zone diameter was used to assess antibacterial activity.

3. Characterization:

3.1. Ftir: At a prominent peak at 3500 cm⁻¹, FTIR pattern, the O-H-bond of the silanol groups and the water molecules were reflected in the samples. The C-H vibration should be present at a low peak at 2890 cm⁻¹ and suggest that virtually all of the tensile material has been extracted. A wavenumber 1635, cm⁻¹ is the bending vibration of the C-H. Siloxane (-Si-O-Si-) is distinguished by its elevated height at 1082 cm⁻¹. The vibration of the silanol groups' Si-OH bond extending is observed at 961 cm⁻¹, and the 490 cm⁻¹ Si-O bond rocking vibration was observed. These functional classes fit TEOS's SBA-16 FTIR pattern. The drop in Si-OH group strength in the graph SBA-16-ZnO nanocomposite implies that ZnO alters the inner surface. SBA-16-ZnO nanocomposite spectrum FTIR exhibits wavelength propagation peaks at 961.64 cm⁻¹ and 474 cm⁻¹, respectively, for Si-O-Zn and ZnO. The peak position of ZnO has been marginally modified due to the rise in ZnO mass as the frequency of vibration is inversely proportional to the mass of the vibrating molecule (Cao et al., 2019).

Figure 1: FTIR Image of SBA-16 and SBA-16-ZnO nanocomposite

3.2. Xrd: X-ray diffraction of SBA-16 and SBA-16-ZnO nanocomposite is demonstrated in figure 2. The spectrum of SBA-16-ZnO nanocomposite comprises peaks with the following indexable ranges: (2) = 32.5°, (35.12°, 36.93°, (111), (200), (210) and (220). PDF #13-0311 of the JCPDS database coordinates the observed diffractions patterns effectively. A broad hump at a 24° angle suggesting the amorphous structure of SBA-16.

Figure 2: XRD graph of SBA-16 and SBA-16-ZnO nanocomposite

3.3. Saks: Their long-range order is detected by small-angle X-ray dispersion (SAXS $0.5^\circ - 5^\circ$). Non-crystalline materials are not at a higher angle. Pure SBA-16 and SBA-16-ZnO nanocomposite are represented in Figure 3. The pure SAXS pattern of the SBA-16 includes two 0.96° and 1.30° reflections, representing respectively (110) and (211) planes. The formation of the SBA-16 cubic structure Im3M is verified. SBA-16 ZnO nanocomposite also represented SAXS data with a single peak at a frequency of 0.99° but with (111) relatively low amplitude. The second highest peak in this pattern resolved due to the introduction of nanoparticles of ZnO into the pores of SBA-16 (Bruckner et al., 2021).

Figure 3: SAXS of SBA-16 and SBA-16-ZnO nanocomposite

Figure 4: SEM Image of SBA-16 and SBA-16-ZnO nanocomposite.

3.4. Sem: Figure 3 shows the ZnO doped SBA-16 nanocomposite SEM image. The appearance of spherical-shaped particles is seen in SEM images of SBA-16-ZnO nanocomposite. For the sample, the smaller particle size is about 100 nm. It should be noticed that as a result of particle agglomerations, the particles can merge and that poly-crystallinity is increasing. The picture above indicates that there are also smaller crystals around the particles in addition to spherical spheres.

3.5. Tem: The TEM experiments have been used to research the mesoporous properties of the SBA-16-ZnO nanocomposite. The TEM micrographs of the SBA-16-ZnO nanocomposite appear in figure 4. The SBA-16-ZnO nanocomposite has a three-dimensional mesoporous

hexagonal form that is similar to SBA-16. The unit cell parameter is roughly 11.6 nm, and this correlates well to 12.5 nm. Through TEM analysis, the ZnO clusters can be significantly seen on canals. This may be because, like the ZnO clusters encapsulated within the mesopores, the picture contrast between the silica frame and the ZnO cluster is high.

Figure 5: SEM Image of SBA-16 and SBA-16-ZnO nanocomposite

Figure 6

3.6. Anti-Bacterial Activity: Disc diffusion testing of SBA-16-ZnO nanocomposite inhibitions was done. Antibacterial action is shown by both SBA-16 and SBA-16-ZnO nanocomposite. The nanoparticles inhibit the development of both bacteria, as seen in Figure 6. In the present, *E. coli* and *S. aureus* were shown activity by SBA-16 and SBA-16-ZnO nanocomposites nanoparticles. The findings demonstrate that the inhibitory zone width is greatest compared with SBA-16 alone for chemically synthesized nanocomposite SBA-16-ZnO (Siddiqi 2018; Johar et al., 2015; Verma et al., 2020; Khanh Nguyen et al., 2020; Narayan et al., 2018).

CONCLUSION

Zinc Oxide nanoparticles were successfully synthesized and doped into mesoporous SBA-16. ZnO nanoparticles have been applied to the mesopores of SBA-16 by the process of post-impregnation. ZnO Nanoparticles have been doped into SBA-16 by studying Fourier Transformed Infrared Spectroscopy XRD and the small X-ray Scattering Graph. The morphological analysis for the sample was performed scanning electron microscopy, and the lattice cell parameter with symmetry was studied with the transmission electron microscope. The result indicated that SBA-16-ZnO nanocomposite exhibit

significant anti-bacterial properties.

REFERENCE

- A. Kumar, S. Gahlyan, R. Thakur, S. Devi, and S. Duhan (2020). "Synthesis of Mesoporous SBA-16 and SBA-16/ZnO Nano-Composite Thin Film for Anti-Biofilm Application," *J Nanosci Nanotechnol*, vol. 20, no. 7, pp. 4210–4215, doi: 10.1166/jnn.2020.17539.
- A. Sirelkhatim et al., (2015). "Review on Zinc Oxide Nanoparticles: Antibacterial Activity and Toxicity Mechanism," *Nano-Micro Lett.*, vol. 7, no. 3, pp. 219–242, doi: 10.1007/s40820-015-0040-x.
- D. Cao, S. Gong, X. Shu, D. Zhu, and S. Liang (2019). "Preparation of ZnO Nanoparticles with High Dispersibility Based on Oriented Attachment (OA) Process," *Nanoscale Res Lett*, vol. 14, no. 1, p. 210, doi: 10.1186/s11671-019-3038-3.
- D. Praseptianga, H. L. Zahara, P. I. Widjanarko, I. M. Joni, and C. Panatarani (2020). "Preparation and FTIR spectroscopic studies of SiO₂-ZnO nanoparticles suspension for the development of carrageenan-based bio-nanocomposite film," *Bikaner, India*, p. 100005. doi: 10.1063/5.0003434.
- J. González-Rodríguez et al., (2020). Enhanced Photocatalytic Activity of Semiconductor Nanocomposites Doped with Ag Nanoclusters Under UV and Visible Light," *Catalysts*, vol. 10, no. 1, Art. no. 1, doi: 10.3390/catal10010031.
- J. R. Bruckner, J. Bauhof, J. Gebhardt, A.-K. Beurer, Y. Traa, and F. Giesselmann (2021). "Mechanisms and Intermediates in the True Liquid Crystal Templating Synthesis of Mesoporous Silica Materials," *J. Phys. Chem. B*, vol. 125, no. 12, pp. 3197–3207, doi: 10.1021/acs.jpcc.0c11005.
- K. S. Siddiqi, A. ur Rahman, Tajuddin, and A. Husen (2018). "Properties of Zinc Oxide Nanoparticles and Their Activity Against Microbes," *Nanoscale Res Lett*, vol. 13, doi: 10.1186/s11671-018-2532-3.
- M. A. Johar, R. A. Afzal, A. A. Alazba, and U. Manzoor (2015). "Photocatalysis and Bandgap Engineering Using ZnO Nanocomposites," *Advances in Materials Science and Engineering*. <https://www.hindawi.com/journals/amse/2015/934587/> (accessed Feb. 20, 2021).
- P. Verma, Y. Kuwahara, K. Mori, R. Raja, and H. Yamashita (2020). "Functionalized mesoporous SBA-15 silica: recent trends and catalytic applications," *Nanoscale*, vol. 12, no. 21, pp. 11333–11363, doi: 10.1039/D0NR00732C.
- Q. N. Khanh Nguyen, N. T. Yen, N. D. Hau, and H. L. Tran (2020). Synthesis and Characterization of Mesoporous Silica SBA-15 and ZnO/SBA-15 Photocatalytic Materials from the Ash of Brickyards," *Journal of Chemistry*, vol. p. e8456194, doi: 10.1155/2020/8456194.
- R. Narayan, U. Y. Nayak, A. M. Raichur, and S. Garg (2018). Mesoporous Silica Nanoparticles: A Comprehensive Review on Synthesis and Recent Advances," *Pharmaceutics*, vol. 10, no. 3, doi: 10.3390/pharmaceutics10030118.
- S. V. Gudkov, D. E. Burmistrov, D. A. Serov, M. B. Rebezov, A. A. Semenova, and A. B. Lisitsyn (2021). "A Mini Review of Antibacterial Properties of ZnO Nanoparticles," *Front. Phys.*, vol. 0, doi: 10.3389/fphys.2021.641481.
- S.-H. Wu, C.-Y. Mou, and H.-P. Lin, (2013). Synthesis of mesoporous silica nanoparticles," *Chem. Soc. Rev.*, vol. 42, no. 9, pp. 3862–3875, doi: 10.1039/C3CS35405A.
- V. Tiwari, N. Mishra, K. Gadani, P. S. Solanki, N. A. Shah, and M. Tiwari, "Mechanism of Anti-bacterial Activity of Zinc Oxide Nanoparticle Against Carbapenem-Resistant *Acinetobacter baumannii*," *Front. Microbiol.*, vol. 0, 2018, doi: 10.3389/fmicb.2018.01218.
- Y. K. Mishra and R. Adelung, "ZnO tetrapod materials for functional applications," *Materials Today*, vol. 21, no. 6, pp. 631–651, Jul. 2018, doi: 10.1016/j.mattod.2017.11.003.
- Y. Wang et al., "Surfactant-assisted Nanocasting Route for Synthesis of Highly Ordered Mesoporous Graphitic Carbon and Its Application in CO₂ Adsorption," *Scientific Reports*, vol. 6, no. 1, Art. no. 1, May 2016, doi: 10.1038/srep26673.
- Y. Zhang, M. K. Ram, E. K. Stefanakos, and D. Y. Goswami, "Synthesis, Characterization, and Applications of ZnO Nanowires," *Journal of Nanomaterials*, Jul. 17, 2012. <https://www.hindawi.com/journals/jnm/2012/624520/> (accessed Feb. 20, 2021).