Microwave-Assisted Solvothermal Synthesis of Tungsten Oxide ($\text{WO}_3$) Nanoparticles for Microbial Inhibition

Harini S$^1$, Aswini A$^1$, S.C. Kale$^2$, Jayashri Narawane$^2$, Jayant Pawar$^3$, Snehal Masurkar$^2$, Shilpa Ruikar$^2$

$^1$Department of Electronics and Communication Engineering, Nanotechnology Division, Periyar Maniammai Institute of Science and Technology, Thanjavur, India; $^2$Department of Allied Sciences, Krishna Institute of Medical Sciences Deemed to be University, Karad, Maharashtra, India; $^3$Directorate of Research, Krishna Institute of Medical Sciences Deemed to be University, Karad, Maharashtra, India.

ABSTRACT

Introduction: Tungsten oxide is an n-type semiconductor which possesses the bandgap of 2.6 – 2.8 eV at room temperature. Additionally, tungsten oxide has the absorption capacity of 480 nm in the visible region resulted from its photocatalytic property.

Objective: To synthesize and evaluate tungsten oxide nanoparticles for microbial inhibition.

Methods: Microwave-assisted synthesis of tungsten oxide nanoparticles was carried out by solvothermal route for the development of antibacterial agent. 1 M Sodium Tungstate Dihydrate was dissolved in 100 mL distilled water which was then mixed with 20 mL of 0.1 M NaOH and the Conc. HCL was added into the reaction mixture. The precipitation of yellow colour was collected and rinsed with purified water three times. During 8 hours at 60°C and 4 hours, the precipitate having undergone drying and calcination to get tungsten oxide powder.

Results: The yellow colour precipitate was obtained after the reaction, which was characterized by UV-Vis Spectroscopy and Scanning Electron Microscopy (SEM). The $\lambda_{max}$ was found to be at 364 nm and bandgap calculated as 3.41 eV.

Conclusion: Antibacterial efficacy was determined by anti-well diffusion assay against $\text{E. coli}$ and $\text{Pseudomonas Aeruginosa}$. The bacterial cultures were found to be sensitive for $\text{WO}_3$ NPs at a concentration of 1000 µg/mL. The $\text{E. coli}$ was more sensitive for $\text{WO}_3$ NPs compared to $\text{Pseudomonas Aeruginosa}$.

Key Words: Microwave-assisted method, Solvothermal synthesis, Tungsten oxide, $\text{E. coli}$, $\text{Pseudomonas Aeruginosa}$, Microbial inhibition

INTRODUCTION

Metal oxide nanoparticles which have unique physical and chemical properties due to change in morphology under the range of 1-100nm. Metal oxide nanoparticles are important in many areas in physics, chemistry, and material science. There are various semiconductor oxides among these tungsten oxides is an important transition metal oxide semiconductor. Tungsten oxide is an n-type semiconductor which possesses the bandgap of 2.6 – 2.8 eV at room temperature. In addition to that tungsten oxide is having the absorption capacity of 480 nm in the visible region resulted from its photocatalytic property. Based on high photocatalytic property tungsten oxide has strong antibacterial activities. The properties of tungsten oxide ($\text{WO}_3$) are electrochromic, photochromic, gas chromic, Photocatalytic, Ferroelectric properties, optical properties and chromic. Tungsten oxide ($\text{WO}_3$) has obtained multiple industrial applications especially in the field of metallurgy, material science, electronic displays and optical modulators smart windows, dye-sensitized solar cell.

There are several methods to synthesize tungsten oxide nanoparticles. Among these methods, microwave irradiation method is most preferable due to less time and uniformity.

The synthesized nanoparticle was studied on bacterial cultures such as $\text{Escherichia. coli}$ and $\text{Pseudomonas aeruginosa}$. Thus experimental work was studied on the UV-Visible Spectroscopy, Scanning Electron Microscopy.
**MATERIALS AND METHODS**

**Materials**
Sodium tungstate (WO₃) with the purity of 98% was purchased from LobaChemie Pvt. Ltd, Mumbai. Sodium hydroxide pellets (NaOH) with the purity of 98% was purchased from Sisco Research Laboratories Pvt Ltd, Mumbai. Hydrochloric acid (HCl) with the purity of 35% was purchased from Loba Chemie Pvt Ltd Mumbai.

**Synthesis of WO₃ NPs**
0.1 M sodium tungstate was dissolved in 0.1 M sodium hydroxide. The concentrated Hydrochloric acid added drop-wise into the reaction mixture with continuous stirring until it reaches pH 1 (Fig. 1). The yellow colour precipitate was obtained and washed for three times using distilled water. The precipitate underwent drying and calcination for 8 hours at 60°C and 4 hours at 300 °C respectively, to get tungsten oxide powder.7,8

![Figure 1: Schematic diagram of the tungsten oxide synthesis process.](image)

**Characterization of Tungsten Oxide Nanoparticles**
Tungsten oxide was characterized by UV-Visible spectroscopy and Scanning Electron Microscopy (SEM) to analyze absorption spectra of nanomaterial, the structural and morphological properties.4,5

**Determination of Antibacterial Activity of tungsten oxide nanoparticles**
Tungsten oxide was tested for its antibacterial efficacy against bacterial culture. coli and Pseudomonas aeruginosa by Anti Well Diffusion Assay (AWDA). The bacterial inoculum was prepared to a final concentration of approximately 1 X10⁵ CFU/mL for the selected bacterial cultures. The synthesized WO₃ NPs of concentration 10, 100 and 1000 µg/mL were dispersed in 0.5 % DMSO by ultra-sonication to make the colloidal solution of nanomaterials. On the surface of agar plates, wells of 5 mm in diameter and of 18 µL in capacity were formed by using sterile gel borer. The 15 µL of WO₃ NPs suspension was placed in each well and was incubated at 37 °C ± 2 °C for 24 hours and zone of inhibition were recorded to understand antibacterial efficacy of WO₃ NPs.9,10

**RESULT AND DISCUSSION**

**Synthesis of tungsten oxide nanoparticles**
The yellow-coloured precipitate of Tungsten oxide nanoparticles was obtained. The resultant powder was dried and used for further characterization.

**UV-Visible spectroscopy**
The synthesized tungsten oxide nanoparticles were characterized in UV-Visible Spectroscopy which was shown in fig.2. In the current work, the \( \lambda_{\text{max}} \) was found at 364 nm. The same experiment was done by Wei Hao Lai et al.,6,7 by chemical deposition method the change in nanoparticles UV-Visible absorbance \( \lambda_{\text{max}} \) at 300 to 900 nm. The optical band gap was calculated by the equation (1), and the calculated bandgap is 3.41 eV.

\[
E = \frac{h\cdot c}{\lambda} \quad (1)
\]

![Figure 2: UV-Visible Spectroscopy.](image)

**Scanning Electron Microscope**
Tungsten oxide nanoparticles obtained was characterized by SEM to understand its size and morphology. Fig. 3(a) showed a bunch of nanostructures at low magnification and at high magnification, the flakes of WO₃ NPs was observed and overall size was found in the range of 200-1000nm.4,5
Microwave-assisted solvothermal synthesis of tungsten oxide (WO₃) nanoparticles for microbial inhibition

Harini et al.

Antibacterial efficacy of tungsten oxide

Tungsten oxide nanoparticles were tested against the bacteria E. coli and Pseudomonas Aeruginosa. Both bacterial cultures were found sensitive for tungsten oxide nanoparticles and got inhibited effectively at 1000 µg/mL concentration of WO₃ NPs. At different concentration, tungsten oxide nanoparticles tested against bacteria are shown in Table 1 and Figure 4.

Table 1: Antibacterial Efficacy of Tungsten oxide nanoparticles against bacterial cultures

| Microorganism / Concentrations | E. coli | P. aeruginosa |
|-------------------------------|---------|--------------|
| 10 µg/mL                      | 8 mm²   | 10 mm²       |
| 100 µg/mL                     | 12 mm²  | 14 mm²       |
| 1000 µg/mL                    | 15 mm²  | 18 mm²       |

CONCLUSION

Tungsten oxide nanoparticles were synthesized successfully which have the maximum absorbance λₘₜₐₓ at 364 nm. Nanoparticles of tungsten oxide have been synthesised and characterised by U.V. Vis. spectroscopy and distinguished by SEM. It was observed that the expansion of biofilm formation of Pseudomonas sp. and E. coli was greatly reduced by tungsten oxide nanoparticles. The solvothermal tungsten oxide nanoparticles have demonstrated substantial antibacterial action against Pseudomonas sp. and E. Coli. Thus, it shows positive activity on E. coli and Pseudomonas Aeruginosa bacteria. Moreover, from the evaluative analysis, it was deduced that the WO₃ NP has significant antibacterial potential that can be used in medicine and food industries. The MIC needed to minimize the biofilm formation as observed in microbial studies was 8 wt% of tungsten oxide NPs. Future experiments will explore how tungsten oxide nanoparticle sizes impact on antibacterial activities.

ACKNOWLEDGEMENT

We acknowledge the contribution and support as being provided by the Department of Electronics and Communication Engineering, Nanotechnology Division, Periyar Maniammai Institute of Science and Technology, Thanjavur, India, Department of Allied Sciences, Krishna Institute of Medical Sciences Deemed to be University, Karad, Maharashtra, India, Directorate of Research, Krishna Institute of Medical Sciences Deemed to be University, Karad, Maharashtra, India.

Ethical Issue: Ethical clearance was taken from institutional ethical committee, KIMSDU, Karad.

Funding Sources: Krishna Institute of Medical Sciences Deemed To Be University, Karad.

Conflict of Interest: Nil.

REFERENCES

1. Dizaj SM, Lotfipour F, Barzegar-Jalali M, Zarrintan MH, Adibkia K. Antimicrobial activity of the metals and metal oxide nanoparticles. Mat Sci Engg: C 2014;44:278-284.
2. Khan I, Abdalla A, Qurashi A. Synthesis of hierarchical WO3 and Bi2O3/WO3 nanocomposite for solar-driven water splitting applications. Int J Hydrogen Energy 2017;42(5):3431-3439.
3. Ahmadi M, Younesi R, Guinel MJ. Synthesis of tungsten oxide nanoparticles using a hydrothermal method at ambient pressure. J Materials Res 2014;29(13):1424-1430.
4. Ahmadi M, Younesi R, Guinel MJ. Synthesis of tungsten oxide nanoparticles using a hydrothermal method at ambient pressure. J Materials Res 2014;29(13):1424-1430.
5. Rezaee O, Chenari HM, Ghodsi FE. Precipitation synthesis of tungsten oxide nanoparticles: X-ray line broadening analysis and photocatalytic efficiency study. J Sol-Gel Sci Tech 2016;80(1):109-118.
6. Wasmii BA, Al-Amiery AA, Kadhum AA, Mohamad AB. Novel approach: tungsten oxide nanoparticle as a catalyst for malonic acid ester synthesis via ozonolysis. J Nanomat 2014;2014.
7. Zheng H, Ou JZ, Strano MS, Kaner RB, Mitchell A, Kalantar-Zadeh K. Nanostructured tungsten oxide–properties, synthesis, and applications. Adv Funct Mater 2011;21(12):2175-2196.
8. Ghasemi L, Jafari H. Morphological characterization of tungsten trioxide nanopowders synthesized by sol-gel modified Pechini’s method. Mat Res 2017;20(6):1713-1721.
9. Popov AL, Zholobak NM, Balko OI, Balko OB, Shcherbakov AB, Popova NR, et al. Photo-induced toxicity of tungsten oxide photochromic nanoparticles. J Photochem Photobio B: Biology. 2018;178:395-403.
10. Jain N, Bhosale P, Tale V, Henry R, Pawar J. Hydrothermal assisted biological synthesis of silver nanoparticles by using honey and gomutra (Cow Urine) for qualitative determination of its antibacterial efficacy against Pseudomonas sp. isolated from contact lenses. Eur Asian J Bio Sci 2019;13(1):27-33.