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Photodegradation of Benzothiazole and Amantadine micropollutants with AG^0(NP)/TiO_2 nanocomposite

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Abstract: *The photodegradation of benzothiazole and amantadine micropollutants was investigated using a silver nanoparticles (AgNPs)/titanium dioxide (TiO_2) nanocomposite under ultraviolet (UV) irradiation. This study aimed to enhance the photocatalytic efficiency of TiO_2 by incorporating AgNPs, which are known to improve charge separation and increase reactive species generation. Characterization of the AgNPs/TiO_2 nanocomposite was conducted using techniques such as UV-Vis spectroscopy, X-ray diffraction (XRD), and scanning electron microscopy (SEM). The degradation kinetics of benzothiazole and amantadine were evaluated, revealing that the AgNPs/TiO_2 system significantly accelerated the degradation processes compared to pure TiO_2. The optimal conditions for photocatalytic activity, including pH, initial pollutant concentration, and catalyst dosage, were systematically optimized. The results indicated a notable reduction in the half-life of both micropollutants, highlighting the potential of AgNPs/TiO_2 nanocomposites in wastewater treatment applications. Additionally, the study provides insights into the mechanistic pathways involved in the degradation processes, contributing to the understanding of photocatalytic mechanisms for the removal of pharmaceutical contaminants from aquatic environments.*

The comprehensive characterization of Ag(NP)/TiO_2 thin films using these advanced techniques enables a detailed understanding of their structural, optical, and surface properties. Such knowledge is vital for optimizing their performance in the photodegradation of micropollutants, thus paving the way for effective environmental remediation strategies.

Keywords: *photodegradation, benzothiazole, amantadin, micropollutants, Ag^0(NP)/TiO_2, nanocomposite,*

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I. INTRODUCTION

The increasing presence of micropollutants, particularly pharmaceutical compounds and their metabolites, in aquatic environments has raised significant concerns regarding water quality and ecosystem health. Benzothiazole, a heterocyclic compound widely used in rubber production, and amantadine, an antiviral medication, are two such micropollutants that have been detected in various water bodies, posing risks to aquatic organisms and potentially entering the human food chain (Jafari Kang et al., 2017; Tuzen et al., 2018). Conventional wastewater treatment methods often fall short in effectively removing these persistent organic pollutants, necessitating the exploration of advanced oxidation processes (AOPs) that leverage photocatalysis to enhance degradation efficiency (Peiris et al., 2017).

Benzothiazole, a heterocyclic compound primarily used in rubber manufacturing and as a corrosion inhibitor, has been detected in various environmental matrices, including surface waters and sediments (Lalliansanga, et al., 2020). Its environmental persistence is largely attributed to its stable chemical structure, which resists degradation under typical environmental conditions (Syafrudin et al., 2021). The presence of benzothiazole in aquatic ecosystems can disrupt biological processes and has been linked to toxic effects on aquatic organisms, highlighting the need for effective removal strategies (Li et al., 2022).

Amantadine, originally developed as an antiviral medication for influenza, has also found use in the treatment of Parkinson's disease. However, its introduction into wastewater systems has raised concerns due to its high resistance to conventional degradation processes .Studies have reported the occurrence of amantadine in various environmental waters, with implications for both aquatic life and human health, as it may lead to the development of antibiotic resistance in microorganisms (Zhao et al., 2023). The persistence of amantadine in the environment necessitates the development of innovative remediation techniques that can effectively degrade this micropollutant.

Traditional wastewater treatment processes often fall short in effectively removing these emerging contaminants, necessitating the exploration of advanced methods. Among these, photocatalysis has gained considerable attention due to its ability to harness solar energy for the degradation of organic pollutants. Titanium dioxide (TiO_2) is a widely studied photocatalyst, known for its photocatalytic properties and environmental safety. However, its limitations, including rapid recombination of photogenerated electron-hole pairs and low absorption efficiency under visible light, hinder its effectiveness (C. Li., 2022) Recent studies have demonstrated that AgNPs/TiO_2 nanocomposites exhibit superior photocatalytic activity compared to their pure TiO_2 counterparts, particularly under UV irradiation. This enhancement is attributed to the surface plasmon resonance (SPR) effects of silver, which can increase the generation of excitons and subsequent ROS (Das &, Ghosh 2022; Zhang et al., 2020). Furthermore, the incorporation of AgNPs can lead to improved adsorption properties, thereby increasing the availability of micropollutants at the catalyst surface for degradation (Mishra S, Das AP. 2021.)

To overcome these limitations, recent research has focused on modifying TiO_2 with metal nanoparticles and other materials to enhance its photocatalytic performance. These modifications can improve light absorption, facilitate charge separation, and increase the generation of reactive oxygen species (ROS), which are crucial for the degradation of organic pollutants (Ghosh & Das, 2022). The integration of silver nanoparticles (AgNPs) into TiO_2 matrices has shown promising results in enhancing photocatalytic activity, providing a viable approach for the removal of persistent micropollutants like benzothiazole and amantadine (Solcova, et al., 2025).

This study aims to investigate the photodegradation of benzothiazole and amantadine using an AgNPs/TiO_2 nanocomposite under UV light. By optimizing the photocatalytic conditions, including pH, initial pollutant concentration, and catalyst dosage, we seek to elucidate the mechanistic pathways involved in the degradation process. The findings will contribute to the development of efficient photocatalytic systems for the removal of emerging contaminants, providing insights into potential applications for advanced wastewater treatment technologies.

II. MATERIALS AND METHODS

2.1. Chemical and Materials

Silver nitrate, Benzothiazole and Amantadine , acetic acid, titanium isopropoxide and sodium borohydride (all Sigma Aldrich Co., USA products) were used without further refinement. All the chemicals were used of AR or equivalent grade. The chemicals were employed without any further purification. The water was purified using the Millipore Water Purification system (Model: Elix 3)

2.2 Experimental Methodology

2.2.1. Preparation of Silver Nanoparticles

Silver nanoparticles (AgNPs) were synthesized using a well-established chemical reduction method, following the protocol outlined by Rashid et al. (2013). In this procedure, a 0.1 mmol/L solution of silver(I) sulfate (Ag_2SO_4) and a 0.2 mmol/L solution of sodium borohydride ($NaBH_4$) were prepared separately to ensure accurate concentrations.

First, 30 mL of the sodium borohydride solution was placed in a beaker, which was maintained in an ice bath to control the reaction temperature and prevent excessive heat generation during the reduction process. Under constant stirring, the silver sulfate solution was introduced dropwise into the sodium borohydride solution at a rate of approximately one drop per second. This careful addition allows for controlled nucleation and growth of the nanoparticles.

The reduction of silver(I) ions to zero-valent silver (Ag^0) was visually indicated by a distinct color change of the solution from pale yellow to a brighter yellow, signaling the formation of AgNPs (Equation 1):

$Ag_2SO_4 + 2NaBH_4 \rightarrow 2Ag(0) + H_2 + B_2H_6 + Na_2SO4$ (1)

Upon completion of the reaction, the synthesized AgNPs exhibited stability for several minutes, which is crucial for subsequent applications. The ability to maintain stability in colloidal suspension is essential for ensuring the effectiveness of AgNPs in various fields, including catalysis and biomedicine (Ghosh & Das, 2022).

This method not only facilitates the efficient synthesis of silver nanoparticles but also allows for the tuning of their size and morphology through variations in reaction conditions, such as temperature and reactant concentrations. Such tunability is vital for optimizing the performance of AgNPs in specific applications. (Lalhriatpuia, et al., 2023)

2.2.2 Synthesis of TiO_2 Nanoparticles

Titania nanoparticles were synthesized by simple hydrolysis of $TiCl4$ by ammonia in refluxing conditions. Titanium tetrachloride (S.D. Fine Chemical Co. Ltd., India) was used as received without any further purification. In a typical reaction, 0.8 mL of 0.5 M $TiCl4$ (in HCl) was added to 100 mL of distilled water. The pH of the solution was adjusted to 8 by dropwise addition of diluted ammonia solution with constant stirring. The mixture was then refluxed for 12 h. The white precipitate obtained was washed several times with distilled water and then dried at 100 °C (Guin, et al., 2007).

2.2.3 Preparation of Ag^0/titania sol solutions

Preparation of Nanostructured TiO_2 via Template Synthesis

A template synthetic method was employed to synthesize nanostructured titanium dioxide (TiO_2), utilizing polyethylene glycol (PEG) as a structuring agent to facilitate the formation of a titania network. In this procedure, titanium isopropoxide ($Ti(OC_3H_7)_4$, or TISP) was mixed with 2.0 g of PEG, which was subsequently dissolved in acetylacetone (AcAc). This solvent system was selected to promote the solubility of the titanium precursor and enhance the network formation of TiO_2 (Lalliansanga, et al., 2022).

Simultaneously, 10 mL of freshly prepared silver nanoparticle (Ag^0NP) solution was incorporated into the titanium precursor solution. The integration of Ag^0NPs is critical for enhancing the photocatalytic properties of the resultant TiO_2, as silver nanoparticles are known to improve charge separation and extend the light absorption range (Zhang et al., 2020).

Following the addition of Ag^0NPs, a mixed solution of ethanol (EtOH), acetic acid (AcOH), and distilled water (H_2O) was gradually introduced to the titanium solution. This step initiated both hydrolysis and condensation reactions, leading to the formation of a gel-like network. The controlled addition of the mixed solvent is essential to ensure a uniform reaction environment, which promotes the formation of homogenous TiO_2 structures (Zhang et al., 2020).

In parallel, a non-template method for the preparation of Ag^0NP-doped TiO_2 was also conducted. This alternative approach allowed for the direct incorporation of silver nanoparticles into the TiO_2 matrix without the use of a structured template, enabling further exploration of the effects of synthesis methods on the properties of the resulting photocatalysts (Ghosh & Das, 2022).

2.2.4. Fabrication of Ag(NP)/TiO_2 Film

Borosilicate glass disks, with a radius of 1.15 cm and a thickness of 0.5 cm, were utilized as substrates for the fabrication of silver nanoparticle-doped titanium dioxide (Ag(NP)/TiO_2) films. Prior to film deposition, the glass disks were meticulously cleaned using dilute nitric acid to remove impurities, followed by extensive rinsing with distilled water. The cleaned substrates were subsequently dried in an oven at 105 °C to eliminate any residual moisture, ensuring optimal conditions for film adhesion (Lalliansanga, et al., 2020).

The thin film was fabricated employing a manual dip-coating method. The glass disks were carefully immersed in a titania sol solution and maintained in a vertical position for a duration of 1 hour. Following this immersion, the disks were gently removed using forceps and placed vertically in a dry beaker for an additional 12 hours to allow for proper solvent evaporation. This step is critical in achieving uniform film thickness and composition. The coated disks were then dried at 100 °C and underwent annealing at 500 °C for 3 hours. This thermal treatment promotes crystallization of the TiO_2 matrix and facilitates the integration of AgNPs, resulting in the formation of a thin and homogeneous nanocomposite film .(Zhao, et al., 2023).

After fabrication, the coated disks were stored in a dry container under dark conditions to prevent any photodegradation or oxidation that could compromise the material properties. Additionally, the sol solutions, specifically Ag(NP)/TiO_2 and Ag(NP)/TiO_2(T), were subjected to drying at 105 °C before being annealed at 500 °C for 4 hours. This process yielded nanocomposite powders of Ag(NP)/TiO_2 and Ag(NP)/TiO_2(T), which were subsequently stored in airtight containers for characterization purposes (Zhang et al., 2020).

The methodologies employed in this fabrication process are crucial for ensuring the desired structural and functional properties of the Ag(NP)/TiO_2 films, enhancing their applicability in photocatalytic and other advanced material applications.

2.2.5. Characterization of Thin Films

The characterization of Ag(NP)/TiO_2 thin films is critical for evaluating their structural, optical, and photocatalytic properties, particularly in the context of their application for the photodegradation of micropollutants such as benzothiazole and amantadine. Several analytical techniques are employed to assess the films' characteristics comprehensively.

Structural Analysis

X-ray Diffraction (XRD): XRD is employed to determine the crystalline phases of the TiO_2 matrix and the presence of silver nanoparticles. The diffraction patterns provide information on the crystallinity, phase composition (anatase vs. rutile), and average crystallite size using the Scherrer equation (Ulhakim et al., 2024),. Saleh et al., 2018).

Scanning Electron Microscopy (SEM): SEM is utilized to examine the surface morphology and microstructure of the thin films. This technique offers high-resolution images that reveal the distribution and size of AgNPs on the TiO_2 surface, enabling an assessment of the uniformity and homogeneity of the coatings (Zhou et al., 2022, Tahir et al., 2016).

Optical Properties

UV-Vis Spectroscopy: UV-Vis spectroscopy is employed to analyze the optical properties of the Ag(NP)/TiO_2 films. This technique allows for the identification of surface plasmon resonance (SPR) peaks associated with AgNPs, as well as the assessment of light absorption capabilities of the composite films. The band gap energy of TiO_2 can also be determined from the UV-Vis spectra, which is critical for evaluating photocatalytic efficiency (Tiwaria et al., 2018).

Photoluminescence (PL) Spectroscopy: PL spectroscopy is used to investigate the electron-hole recombination dynamics within the films. A lower PL intensity typically indicates improved charge separation, which is essential for enhanced photocatalytic activity (Chen et al., 2020, Tadele Assefa Aragaw 2024) .

Surface Analysis

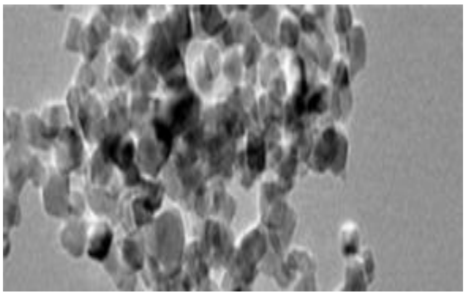
Atomic Force Microscopy (AFM): AFM is utilized to analyze the surface roughness and topography of the thin films at the nanoscale. This technique provides insights into the surface characteristics, which can influence the photocatalytic activity of the films (Tiss, et al., 2021).

Functional Groups

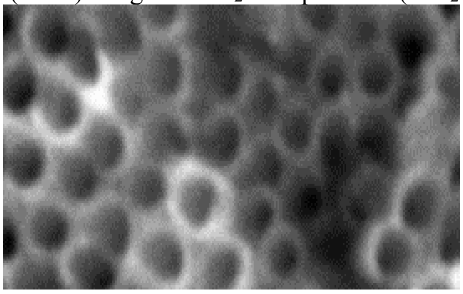
Fourier Transform Infrared Spectroscopy (FTIR): FTIR is employed to identify functional groups present in the thin films. This technique helps in understanding the chemical interactions between AgNPs and the TiO_2 matrix, which may affect the overall photocatalytic performance (Ghosh & Das, 2022, **Pasieczna-Patkowska et al ,2025).**

III. RESULTS AND DISCUSSION

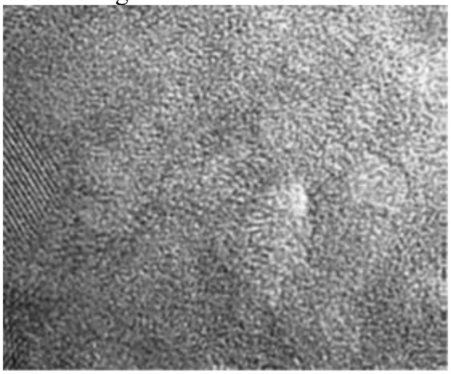
3.1. Morphological study of thin films



Transmission electron microscopic (TEM) image of TiO_2 nanoparticles (TiO_2 NPs)



Surface morphologies of bare nanotube and AgNPs



Transmission electron microscopic images of (a) Ag^0(NP)/TiO_2

3.2. Characterization of Solids

X-ray diffraction (XRD) analysis confirms that the synthesized materials are composed of a pure anatase TiO_2 phase, with crystal structures in good agreement with the standard JCPDS data (JCPDS card no. 21-1272). Figure 1 presents the XRD patterns of both bare TiO_2 nanoparticles and their 5 wt% Ag-loaded nanocomposites.

The broadening of the diffraction peaks indicates that the synthesized particles are in the nanometer size range. Crystallite sizes, estimated using the Scherrer equation applied to the most intense diffraction peaks, were found to be in the range of 10–15 nm. The calculated lattice parameters also closely match the reported values. (Guin, et al.,2007).

No distinct diffraction peaks corresponding to crystalline silver were observed, suggesting that Ag is either in an amorphous state or is X-ray amorphous. Notably, a shift in the TiO_2 (102) peak position toward lower 2θ values was observed with increasing Ag content. This shift indicates an increase in d-spacing in the 5 wt% Ag–TiO_2 sample, which may be attributed to the incorporation of Ag into the TiO_2 lattice. This finding is consistent with the results reported by Xin et al., who similarly observed a shift of the TiO_2 (102) peak to lower angles with increasing Ag content. (Xin et al 2005,Zhou et al., 2022).

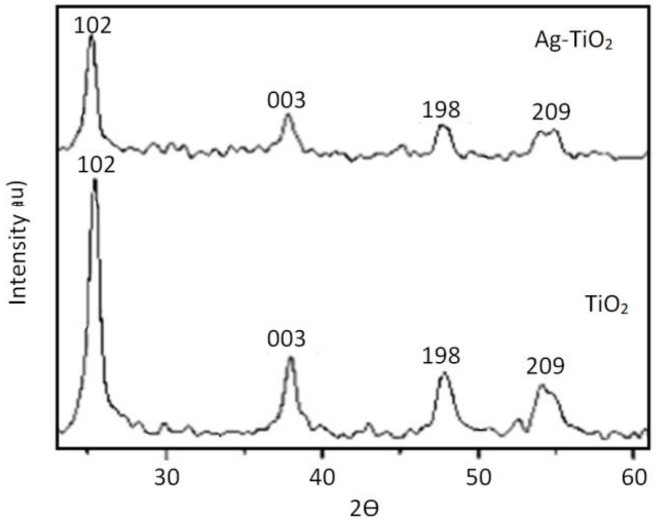


Figure 1. XRD of **(a)** pure TiO_2 and **(b)** 5 wt % Ag-TiO_2 nanoparticles

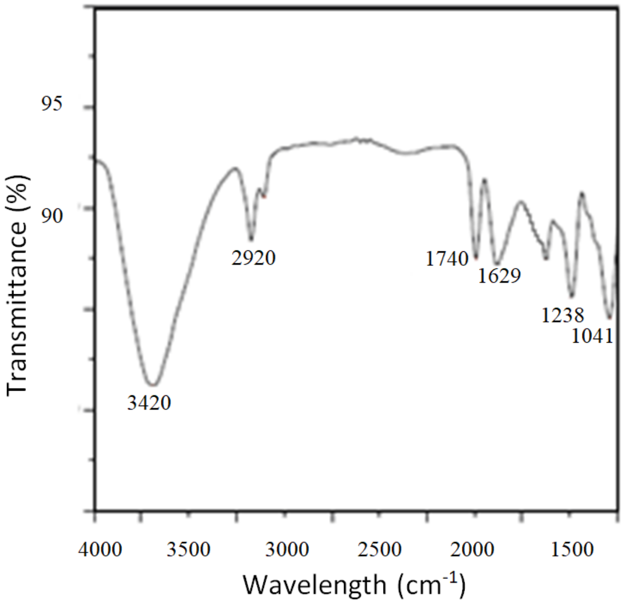


Figure 2. FTIR spectra of phytosynthesized AgNPs.

3.3. Fourier Transform Infrared (FTIR) Spectroscopy

FTIR analysis was conducted to identify functional groups in biomolecules responsible for the bioreduction of Ag^+ and the capping/stabilization of silver nanoparticles. The observed bands were compared with standard values to determine specific functional groups.

The FTIR spectrum (Fig. 2) shows characteristic absorption bands at 3420, 2920, 1740, 1629, 1218, and 1041cm^{-1}, confirming the presence of capping agents. The broad band at 3420 cm^{-1} corresponds to O–H stretching, indicating alcohols and phenols. Peaks at 2920 and 1740 cm^{-1} arise from C–H stretching in aromatic compounds. The band at 1629 cm^{-1} is attributed to non-conjugated C=O stretching. The 1218 cm^{-1} band corresponds to C=C and C=N stretching, suggesting protein presence (Prakash et al., 2013). The 1041 cm^{-1} band is due to N–H bending in amide linkages of proteins. These findings indicate that functional groups involved in capping/stabilizing AgNPs are present, and the protein secondary structure in micropollutants remains unaffected by interaction with Ag^+ or AgNPs. (Prakash et al., 2013, **Jyoti,** et al., 2015).

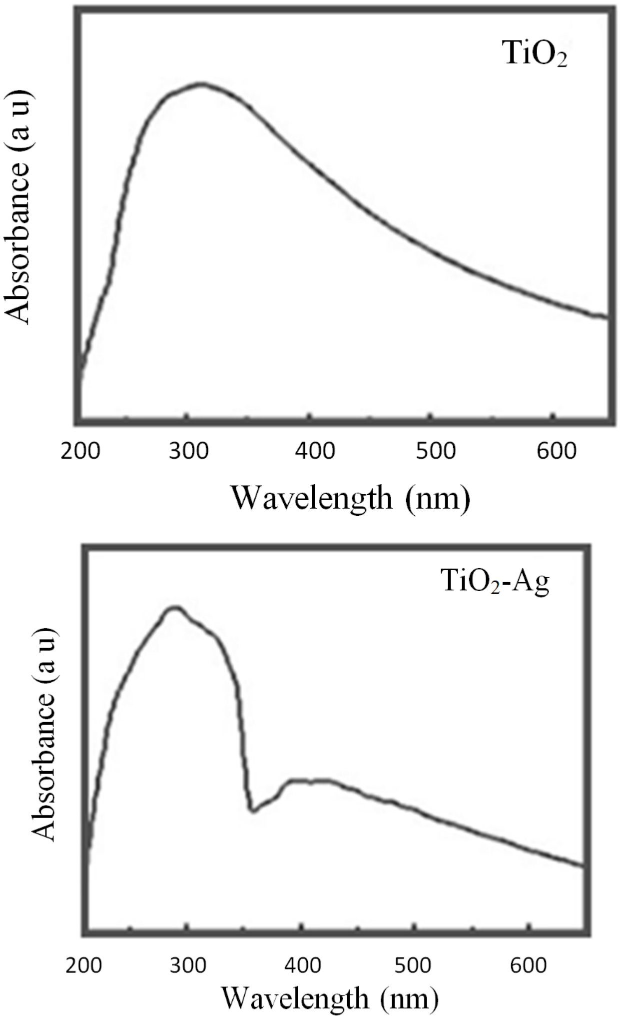


Figure 3. UV–Vis absorption spectra of **a-)** TiO_2 and **b-)** TiO_2–Ag nanocomposites (M-2)

3.4. UV–Vis absorption spectra of A TiO_2 and B TiO_2–Ag nanocomposites

The formation of TiO_2 and its corresponding composite with Ag was confirmed by UV–Vis absorption spectra, as shown in Fig. 1. A strong absorption peak at 301 nm for pristine TiO_2 was attributed to the charge transfer between O^{2-} and Ti^{4+} (Fig. 3). In the composite system, the absorption peak of TiO_2 remained at 280 nm (Fig. 3). The presence of distinct absorption peaks corresponding to the individual components of the composite clearly indicates the absence of alloy formation (Venkatasubbu et al, 2013,Jayeta et al 2022).

IV. CONCLUSION

The Ag^0(NP)/TiO$_2$ nanocomposite proved to be an effective photocatalyst for the degradation of benzothiazole and amantadine under UV irradiation. This study highlights the potential of using metal-doped semiconductors to improve the removal of persistent micropollutants from wastewater. Future work should focus on exploring the stability and reusability of the nanocomposite, as well as its performance in real wastewater matrices.

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