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Fabrication of Bimetallic Zinc Oxide and Silver Nanoparticles Using Laser Ablation for Their Effective Decontamination

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ABSTRACT

In this work, pulsed laser ablation in deionized water was used to successfully create bimetallic zinc oxide–silver (ZnO–Ag) nanocomposite. ZnO and Ag nanoparticles showed clear absorption peaks at about 370 and 403.4 nm, respectively, according to Ultraviolet–Visible Spectroscopy (UV-Vis). Photoluminescence analysis showed a strong emission at 590.9 nm attributed to defect-related radiative recombination, enhanced by the presence of silver. Raman spectroscopy identified broad OH-related bands near 3419 cm^{-1}, suggesting high surface hydroxylation and reactivity. Scanning electron microscopy (SEM) revealed that the ZnO–Ag nanocomposites have an irregular porous structure and an average particle size of 47.3 nm. A light was shone to study the photodegradation of methylene blue (MB) using ZnO–Ag nanocomposites. The results demonstrated that this nanocomposite has a high capacity for charge separation and increases the production of reactive oxygen species (ROS) as a result of the oxidative reaction between ZnO and Ag. The results also demonstrated the nanocomposite's ability to remove pollutants and act as a photocatalyst.

1. INTRODUCTION

Nanotechnology is considered a revolution in materials science, offering practical solutions for environmental remediation, most notably its contributions to water purification. Nanomaterials play a crucial role in removing pollutants. The preparation techniques using sol-gel, non-chemical reactions, hydrothermal reactions, and wet chemical methods all produce spherical, monodisperse zinc oxide nanoparticles with adjustable size and composition by controlling the formation and growth mechanisms during the preparation process [1].

ZnONPs and AgNPs possess excellent optical, antibacterial, and photocatalytic properties, making them valuable materials distinct from other nanomaterials. Laser ablation is a promising and important technology for the future due to its ability to modify parameters and precisely control the size and shape of nanoparticles [2].

In 2017, ZnONPs ranging in size from 40 to 120 nm were prepared using a 300 mJ laser energy and 15 Hz repetition rate laser by researchers Abou Zeid and Leprince-Wang [3] and Kaliraj et al. [4].

Methylene blue (MB) is a coloring agent and poses environmental risks when discharged into wastewater [5]. Textile factories produce harmful chemicals such as ammonia, chlorine, and anthraquinones, which contribute to ecosystem pollution [6].

In addition, some dyes are toxic and carcinogenic, posing a risk to human health [5]. Therefore, a crucial aspect of the textile industry is focusing on the proper disposal of wastewater using various techniques such as membrane adsorption [7], advanced oxidation [8], and electrochemical techniques [9]. Adsorption technology is particularly effective and cost-efficient in removing pollutants.

Scientists are currently focusing on improving the degradation capacity of nanomaterials by mixing them with an absorbent material; for example, they used nanoparticles of zinc [10], silver [11], gold [12], palladium [13], copper [14], nickel [15], magnesium [16], and titanium [17] have been used in the photodegradation of MB. Given the simplicity of the pulsed laser ablation technique, this study employs it to prepare bimetallic ZnO-Ag nanoparticles in deionized water. The photocatalytic activity of ZnO-Ag nanocomposites is examined using visible light spectroscopy, particularly in the degradation of MB dye. The results indicate that ZnO–Ag exhibits both adsorption and degradation properties, confirming its potential as an efficient photocatalyst.

2. MATERIALS

The following materials were used in this study: Zinc oxide (ZnO; 99.5%), silver (Ag; 99.5%) with dimensions of 2 cm and a thickness of 5 mm, MB dye ($C_{16}H_{18}N_3SCl$), and Distilled water was used throughout the article.

3. METHODS

3.1 Preparation of ZnO and Ag nanoparticles

ZnONPs were synthesized using a pulsed laser ablation method. A pure zinc oxide target with dimensions of 2 cm and a thickness of 5 mm was placed in a cylindrical glass beaker filled with 5 ml of distilled water at room temperature. The laser beam was focused (Q-switch Neodymium-doped Yttrium Aluminum Garnet Nd-YAG laser), fundamental wavelength (λ = 1064) nm and energy per pulse (E pulse = 250) mJ/pulse, at a repetition rate of 3 Hz, through a convex lens onto the surface of the metal target, and with 500 pulses. The Petri dish containing the zinc plate is placed on a rotating disc during the ablation process to ensure that the pulses are distributed over a wider area from the zinc plate, resulting in the production of homogeneous nanoparticles, and to protect the target surface from corrosion caused by the accumulated heat of the laser pulses. Silver nanoparticles were prepared by placing the silver target with dimensions of 2 cm and 5 mm in a cylindrical glass beaker filled with 5 ml of distilled water at room temperature and it was excised by the same method and with the same laser parameters as those used for the zinc oxide target.

3.2. Preparation of methylene blue dye

MB dye solution was prepared in a dissolved liquid (water) at a concentration of 0.001M with a mass of (0.0095 gm) in 30 ml of water, then a concentration of 0.00001M was prepared from the original concentration using the dilution equation (C1 V1= C2 V2), where the final concentration was prepared by dilution by adding the suspended liquid of the ZnO–Ag nanoparticles so that the original dye concentration was 0.00001M with the addition of nanoparticles in 20 ml of deionized water.

3.3 Photodegradation of methylene blue in the presence of ZnO–Ag nanoparticles

The photodegradation of blue dye in the presence of catalyzed zinc oxide and silver nanoparticles was studied under normal light and at different exposure times (5, 15, 20, 25, 30, 40) minutes. The degradation rate was determined using Ultraviolet–Visible Spectroscopy (UV-Vis).

4. CHARACTERIZATION TECHNIQUES

Synthesized zinc oxide (ZnO) and zinc silver oxide (ZnO–Ag) nanoparticle samples were characterized using various analytical techniques. The morphologies of the as-synthesized nanoparticles were characterized using a field-emission scanning electron microscopy (FESEM, Tescan Lyra-3) at an accelerating voltage of 20 kV. The chemical compositions of the as-synthesized nanoparticles were studied using X-ray photoelectron spectroscopy (XPS, ESCALAB250Xi, Thermos Fisher Scientific). Raman spectroscopy was performed using a Lab-Ram HR Evolution Raman spectrometer at a wavelength of 1064 nm at 30% variable power. A HORIBA Jobin Yvon Fluorolog-3 fluorescence spectrometer was used to obtain the photoluminescence (PL) spectra of the samples synthesized at room temperature. The optical properties of zinc oxide nanoparticles and bimetallic zinc oxide nanoparticles were studied using a UV-Vis spectrometer (Shimadzu SolidSpace-3700). 10 mm diameter quartz vessels were used to study the absorption of zinc oxide nanoparticles and bimetallic zinc oxide nanoparticles. Absorption spectra were recorded between 200 and 900 nm.

5. RESULTS AND DISCUSSION

5.1 Ultraviolet–Visible Spectroscopy absorption spectroscopy

The UV-Vis spectra of ZnONPs and silver nanoparticles (AgNPs) exhibit two peak positions centered at approximately 370 and 403.4 nm, as shown in Figures 1 and 2. The energy gap can be calculated using the following Planck equation: $E_g = \frac{1240}{\lambda_{max}}$, its equal to 3.35 eV and 3.07 eV for ZnONPs and AgNPs respectively.

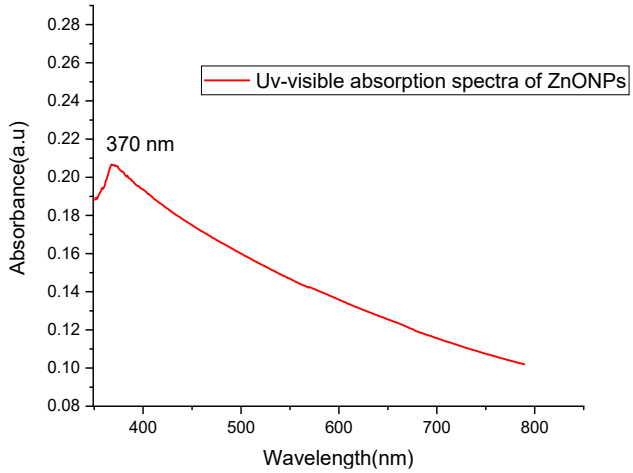

Figure 1. The absorption spectra of ZnONPs

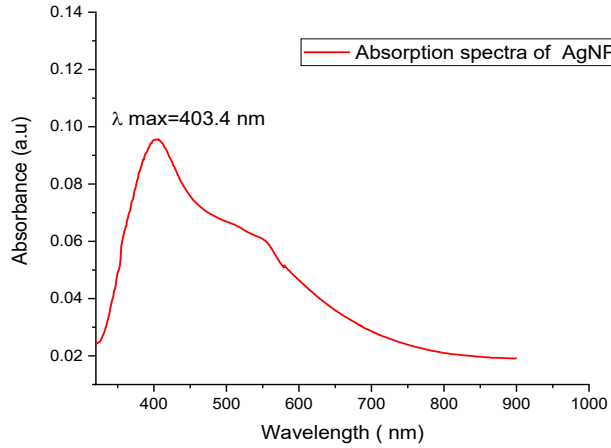

Figure 2. The absorption spectra of AgNPs

A new absorption peak at 404 nm due to the ZnO–Ag nanocomposite is shown in Figure 3. It shows that the nanocomposite has a red shift of 20 nm and 0.6 nm for ZnONPs and AgNPs respectively due to the occurrence of the quantum confinement phenomenon resulting from the high laser energy. It is noted that the dominance of AgNPs over of ZnONPs.

The interaction between ZnO NPs and Ag NPs in UV-Vis spectroscopy reveals an interfacial electronic coupling

characterized by plasmon–exciton interaction and charge transfer. The electronic states of ZnO are altered by this interfacial bonding, which causes absorption peak shifts and verifies the creation of a genuine ZnO–Ag Nanocomposite as opposed to a physical mixture.

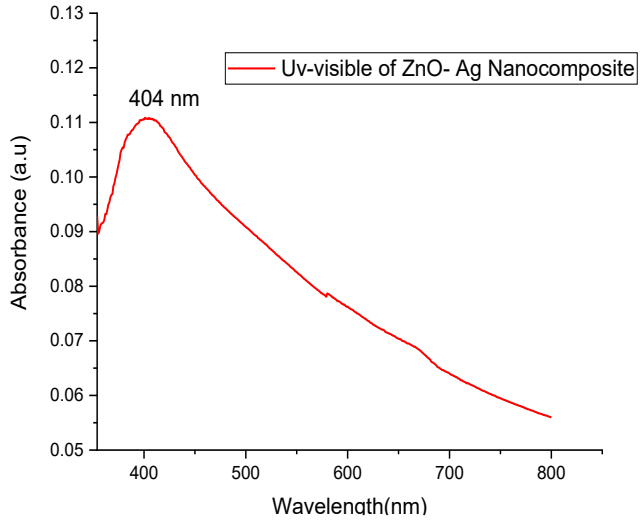

Figure 3. The absorption spectra of ZnO–Ag Nanocomposite

5.2 Photoluminescence spectroscopy (PL)

Pure ZnO typically exhibits emission in the ultraviolet region due to recombination of electron and gap pairs at the band-edge. However, when ZnO is doped with silver, changes in the electronic structure occur. At an excitation wavelength of 290 nm, the photoemission occurs at 590.9 nm, due to radiative transitions arising from defect states within the ZnO, which are enhanced by the presence of silver atoms are shown in Figure 4. These defects often include oxygen gaps or internal Zn sites, which opens up the possibility of using this system in environmental or biological sensors that rely on emission in this region.

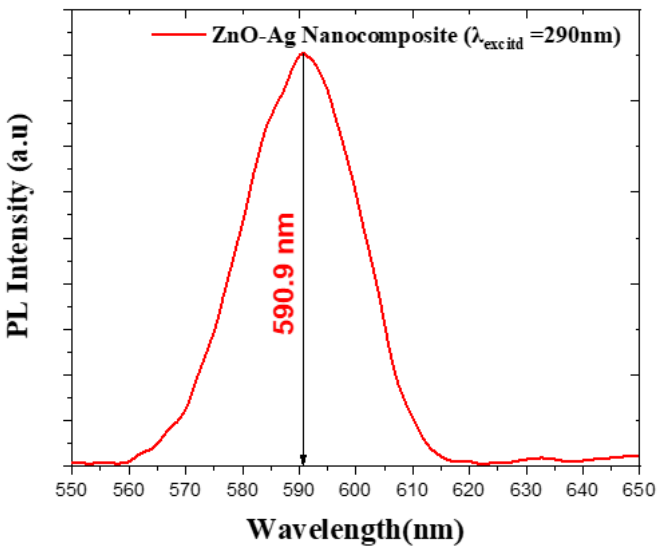

Figure 4. Photoluminescence spectra of ZnO–Ag nanocomposite

5.3 Raman Spectra Analysis of ZnO–Ag nanoparticles

Raman spectra were measured at room temperature for nanocomposite of zinc oxide and silver (ZnO–Ag) over the spectral range of 2500 to 4500 cm^{-1}. It is worth noting that the preparation of these particles using the laser ablation technique in water results in surfaces with a hygroscopic nature. This is attributed to the structural properties of zinc oxide, which is characterized by a high surface area and a pronounced tendency to retain moisture, especially upon transformation into a nanostructure. This leads to strong physical and chemical adsorption of water molecules on the particle surface. The spectral analysis results revealed a broad absorption band near 3419 cm^{-1}, which can be attributed to hydroxyl groups (–OH) attached to the surface of the ZnO particles. This indicates the presence of adsorbed water molecules or hydroxyl residues on the surface, likely due to interaction with the aqueous medium during synthesis. In addition, narrower and less intense bands were observed at 3770 and 3894 cm^{-1}, as shown in Figure 5, which are associated with isolated hydroxyl groups (–OH). These bands suggest surface water adsorption or the formation of terminal Zn–OH bonds on the particle surface. This is a result close to what the researcher mentioned in the references [18]. This is evidence of the presence of the summit within the range of known bands.

Raman peaks reflect the high surface reactivity of the nanoparticles, a key feature in photocatalytic applications (photocatalysis). This is due to the important role of hydroxyl groups in enhancing light absorption and facilitating interactions with pollutants. Furthermore, this nanostructure enhances the particles' ability to adsorb organic dye molecules, contributing to increased efficiency in photodegradation processes for environmental pollutants.

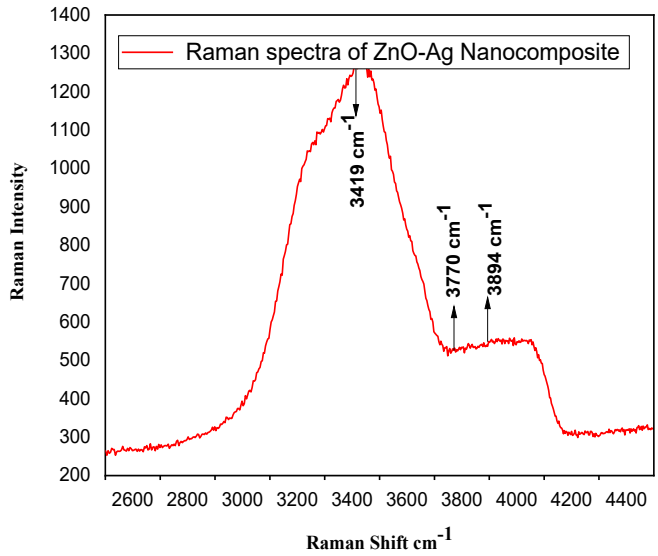

Figure 5. Raman spectra analysis of ZnO–Ag nanocomposite

5.4 Scanning electron microscope of ZnO–Ag nanoparticles

A scanning electron microscopy (SEM) image of a zinc oxide/silver (ZnO–Ag) nanocomposite was used to explain the surface morphology with an irregular, interconnected, and porous network structure, with distinct brightness variations indicating surface roughness. These surface properties are characteristic of ZnO–Ag nanocomposites as shown in Figure 6.

This structure arises from the self-growth of ZnO

nanoparticles and their arrangement within a three-dimensional matrix, where the silver particles appear either as dispersed particles on the zinc oxide surface or partially embedded within its structure. This complex structure results in an increased specific surface area, enhancing the material's ability to interact with the surrounding medium. This is critical in catalytic applications, such as photocatalysis for the decomposition of organic dyes in aqueous media. We also note that the distribution of nanoparticles is irregular, as there is a large portion of very small particles (less than 20 nm) with a small number of larger particles, and the average diameter of the particles is about (47.3 nm), as shown in Figure 7.

5.5 Photodegradation of methylene blue in the presence of ZnO-Ag nanoparticles

The photocatalytic degradation of MB using ZnO-Ag nanocomposites was demonstrated in Figure 8 at varying irradiation durations. Ag nanoparticles increase photocatalytic activity by enhancing light absorption and generating heated electrons. The control of the ZnO–Ag interface enhances the generation of ROS and the separation of charges. The capacity of ZnO to capture electrons and facilitate charge separation is improved by the introduction of defects. Ag and ZnO function in a synergistic manner, with Ag serving as an electron sink and ZnO supplying active sites for the generation of ROS. The efficiency of MB degradation is enhanced by the increased energy input and ROS generation that result from longer irradiation times. The absorbance decreases gradually as the irradiation time increases, suggesting that the MB degradation process is enhanced as a result of the increased energy input and ROS generation. This behavior is similar to what researchers found in the references [19-21], since nanomaterials have a large surface / volume and the ability to absorb pollutants.

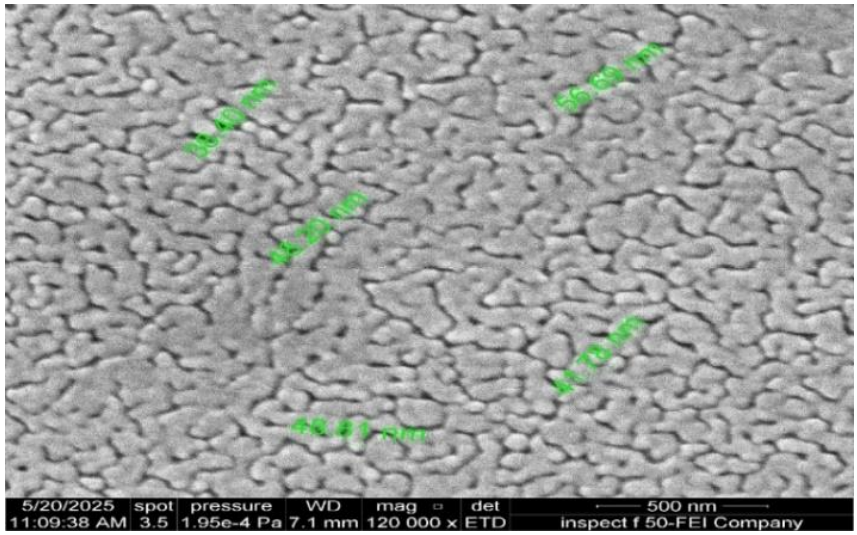

Figure 6. Scanning electron microscope of ZnO–Ag nanocomposite

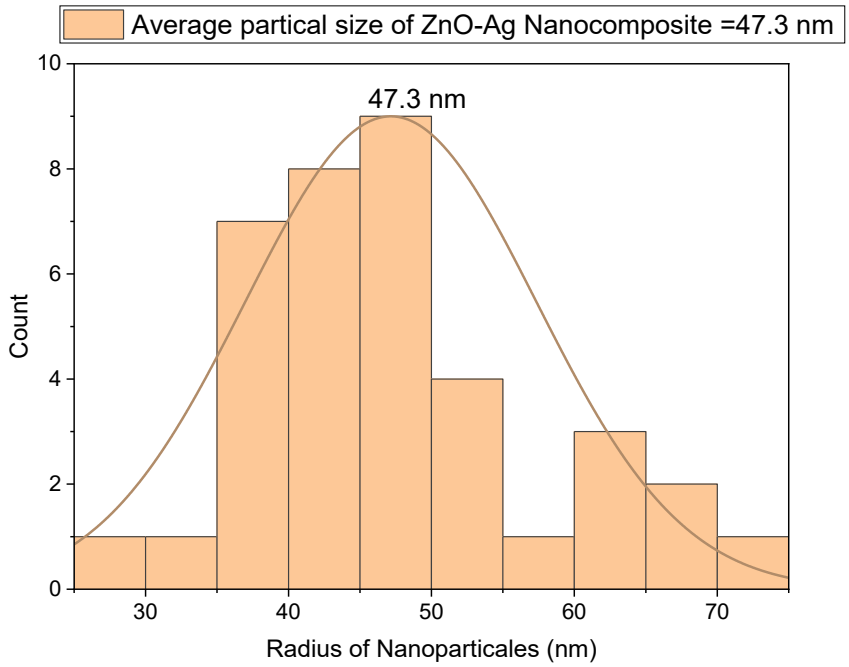

Figure 7. Particle size distribution diagram of ZnO–Ag nanoparticles

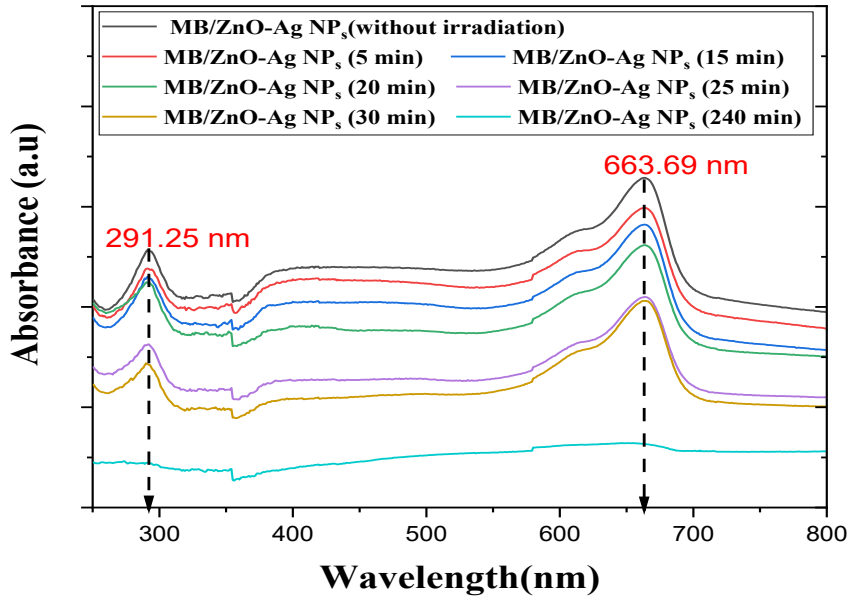

Figure 8. Photocatalytic degradation of methylene blue on ZnO–Ag nanocomposite

6. CONCLUSION

ZnO–Ag nanocomposite was synthesized via pulsed laser ablation in deionized water, showing strong optical, structural, and photocatalytic properties. The synthesized nanocomposite was characterized using UV-Vis, Fluorescence measurements, Raman spectroscopy and SEM. The composite demonstrated excellent performance in degrading MB dye under illumination, due to broad light absorption, hot electron generation, and with ~ 47.3 nm porous morphology, enhanced photoluminescence, and increased production of reactive oxygen species (ROS).

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