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Ag-Doped ZnO–CuO Nanocomposites: Coprecipitation Synthesis and Evaluation of Photocatalytic and Antioxidant Activities

Kalpa C. Naik [1], Smita G. Gite [1], Pranali Sonawane [2] and Ratnamala Sonawane [1,*]

[1] *Department of Chemistry, The Institute of Science, 15, Madam Cama Road, Mumbai, 400 032, India.*
[2] *Department of Chemistry, New England College, Henniker, US.*

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Abstract

Ag-doped ZnO–CuO nanocomposites were successfully prepared by a simple coprecipitation technique and studied for photocatalytic and antioxidant properties. The structural and morphological properties of the synthesized ZnO–CuO nanocomposites were studied by X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy, Scanning electron microscopy (SEM), and Energy-dispersive X-ray analysis (EDAX). The XRD studies confirmed the formation of crystalline ZnO, CuO, and Ag, while the SEM studies revealed a heterogeneous and aggregated nanostructure on a nanoscale. The photocatalytic activity of Ag-doped ZnO–CuO nanocomposites was also studied by decomposing methylene blue dye under light irradiation, showing enhanced activity compared to undoped particles. The improved activity is due to a combined effect of Ag doping and a ZnO/CuO heterojunction, which helps to separate charges and decrease electron-hole recombination. In addition, antioxidant activities were also investigated using a free radical scavenging method, and it was found that Ag-doped ZnO/CuO nanocomposites exhibited strong antioxidant activity that was concentration-dependent. Based on this observation, it could be determined that Ag-doped ZnO/CuO nanocomposites produced through the coprecipitation method possess strong photocatalytic and antioxidant properties, which could be employed in future environmental applications and could be used as a medicine to cure diseases involving cellular oxidation.

Keywords: Nanocomposites; Doped; ZnO-CuO; Coprecipitation method; Photocatalytic Activity; Antioxidant

1. Introduction

In recent years, nanotechnology has come out to be a powerful tool for the development of advanced materials by improving their physicochemical and biological properties [1]. Among all nanomaterials, attention has been focused on metal oxide nanoparticles, including ZnO and CuO, because of their wide applications in catalysis, antibacterial coating, sensors, water purification, and biomedical fields due to their unique properties [2]. Besides excellent UV-absorption, stability, and inherent antimicrobial properties, ZnO is widely applied as a semiconductor and a catalyst, too [3]. CuO represents a p-type semiconductor with good electrical conductivity as well as catalytic activity and antimicrobial activity [4], it is also capable of generating ROS [5]. When these two oxides are combined, ZnO–CuO nanocomposites demonstrate improved charge separation, larger surface area, and enhanced catalytic and biological activities compared to their individual components [1].

Doping metal oxides with noble metals such as silver (Ag) is an effective strategy to further enhance their performance [2] Silver possesses strong antimicrobial properties and the ability to facilitate electron transfer, suppress charge recombination, and increase surface reactivity. Incorporating Ag into ZnO–CuO nanocomposites can therefore create a synergistic system in which the optical, catalytic, antibacterial, and antioxidant properties are significantly improved

* Corresponding author: Ratnamala Sonawane

[3]. Ag doping not only introduces new active sites but also modifies the structural, morphological, and electronic characteristics of the composite, making it more efficient for various biological applications [4]

The antibacterial activity of nanomaterials has gained considerable importance due to the rise in antibiotic-resistant pathogens. Metal oxide nanoparticles exert antibacterial effects mainly by generating reactive oxygen species, disrupting cell membranes, releasing metal ions, and interfering with cellular processes [5]. Ag-doped ZnO–CuO nanocomposites are particularly promising because Ag ions enhance ROS production and exhibit strong interaction with microbial cell walls, leading to rapid bacterial inhibition [6]. Such materials can be used in medical devices, wound dressings, antimicrobial coatings, and water disinfection technologies.

Similarly, antioxidant activity is crucial in preventing oxidative stress caused by excessive free radicals in biological systems [7]. Nanocomposites that possess efficient radical-scavenging activity can play an important role in biomedical applications, including drug delivery, tissue engineering, and protective materials. The combination of ZnO, CuO, and Ag provides multiple redox-active sites that can interact with free radicals, leading to effective antioxidant behavior [8].

The coprecipitation method has been widely used for synthesizing metal oxide nanomaterials due to its simplicity, reproducibility, cost-effectiveness, and ability to produce uniform nanoparticles [9]. This method enables the controlled doping of Ag into the ZnO–CuO matrix, ensuring the good dispersion of metal ions within the composite. The resulting nanocomposites typically exhibit well-defined morphology, high purity, and improved surface functional groups, which directly contribute to their enhanced photocatalytic activities [10].

Ag-doped ZnO-CuO nanocomposites have been widely noticed owing to the improvement in the physicochemical characteristics. The coexistence of ZnO and CuO generates a heterojunction, which increases the efficiency of charge separation. However, the incorporation of Ag further increases the absorption capabilities of light and enhances the recombination rate of electrons and holes. In this current research, the preparation of Ag-doped ZnO-CuO nanocomposites was achieved through a simple co-precipitation technique. In addition, it also investigated its efficacy as a photocatalyst as well as its free radical-scavenging ability. It is especially important to highlight that the obtained result exhibited improvement not only in photocatalysis ability, but also in free radical-scavenging ability, confirming the importance of doping Ag.

2. Materials and Methods

2.1. Chemicals

The chemicals, Zinc Nitrate Hexahydrate, Copper Nitrate, Sodium hydroxide, and silver nitrate, from Sigma Aldrich (99.99 %).

2.2. Preparation of nanoparticles

A 0.03 M $AgNO_3$ solution was freshly prepared in 10 mL of distilled water and mixed with 100 mL of an equimolar $Cu(NO_3)_2$ and $Zn(NO_3)_2$ $(H_2O)_6$ solution under continuous magnetic stirring at room temperature. The mixture's pH was adjusted to nine using 1 M NaOH, and the reaction was maintained for 20 minutes to ensure uniform precipitation. The formation of a bluish-green precipitate indicated successful synthesis. The precipitate was filtered under vacuum, repeatedly washed with deionized water to eliminate residual ions, dried at 80 °C for one hour, and subsequently calcined at 750 °C for two hours in a muffle furnace. The resulting grey-black nano-powders were then subjected to characterization to analyze various properties, including structural, morphological, and optical [11], [12].

2.3. Antioxidant activity

The antioxidant activity of the synthesized compound was evaluated using the DPPH free radical scavenging assay. A 0.1 mM DPPH solution was freshly prepared in methanol and kept protected from light. Different concentrations of the sample (100–6.25 µg/mL) were prepared from a 1 mg/mL stock solution. In each test tube (or well), equal volumes of sample solution and DPPH reagent were mixed and incubated for 30 minutes at room temperature in the dark. The decrease in absorbance was measured at 517 nm using methanol as the blank. Ascorbic acid was used as the positive control, and all measurements were performed in triplicate. The percentage radical scavenging activity was calculated by the following equation: % inhibition = [(A control – A sample)/A control] × 100. IC_{50} value, the concentration necessary to scavenge 50% of DPPH radicals, was calculated from the plot of concentration–response[7], [13], [14].

2.4. Preparation of dye solutions

Prepare a 0.1 mg/L solution, weigh 0.0100 g of methylene blue dye powder, and dissolve it in 100 mL of deionized water in a 100 mL standard amber-colored volumetric flask. Stir the solution for 5-10 minutes to create a stock solution of 100 mg/L. 1 mL is taken from the stock solution and diluted with deionized water just before use [15], [16].

3. Results and Discussion

3.1. UV-Visible

The optical absorption characteristics of the prepared ZnO nanoparticles (NPs) and ZnO–CuO nanocomposites (NCs) were analyzed using a UV–visible spectrophotometer (UV-1900i, Shimadzu,) across a wavelength range of 190–1100 nm. The corresponding spectra are presented in **Fig. 1.** Measurements were conducted at room temperature within the 300–700 nm range. The synthesized nanoparticles exhibited a distinct surface plasmon resonance (SPR) peak between 370 and 380 nm, which is consistent with earlier reports indicating that ZnO nanoparticles typically show maximum absorption around 358 nm [17]. The band gap energies (Eg) were determined using the formula $Eg = 1240/\lambda$ (nm), where λ represents the wavelength of the absorption edge onset [18].

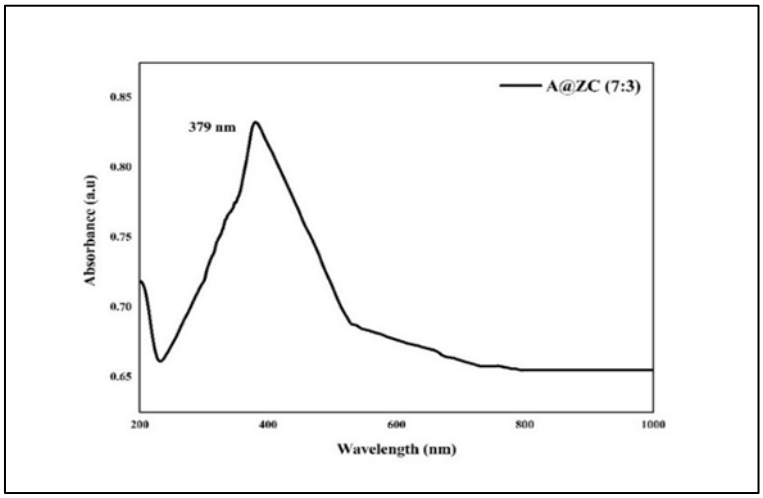


Figure 1 UV of Ag@ZnO-CuO Nanocomposites

3.2. Nanoparticle Tracking Analyzer

As part of the Nanoparticle Tracking Analysis (NTA) process, a colloidal suspension of Ag@NiO-CuO nanocomposite was examined to assess its size distribution and concentration. A 532 nm laser was used in the Malvern NanoSight LM20 system for the measurements. A stabilizing suspension of nanocomposite was formed in deionized water by ultrasonically dispersing the nanocomposites for 10 minutes before being diluted to an optimal concentration range for reliable NTA detection. The size distribution was narrow, with an average hydrodynamic diameter of approximately 43 nm, consistent with the nanoscale particle size after synthesis. NTA also permitted real-time observation and quantification of particle migration, enabling the calculation of the diffusion coefficient and, once more, ensuring the colloidal stability of the suspension [20].

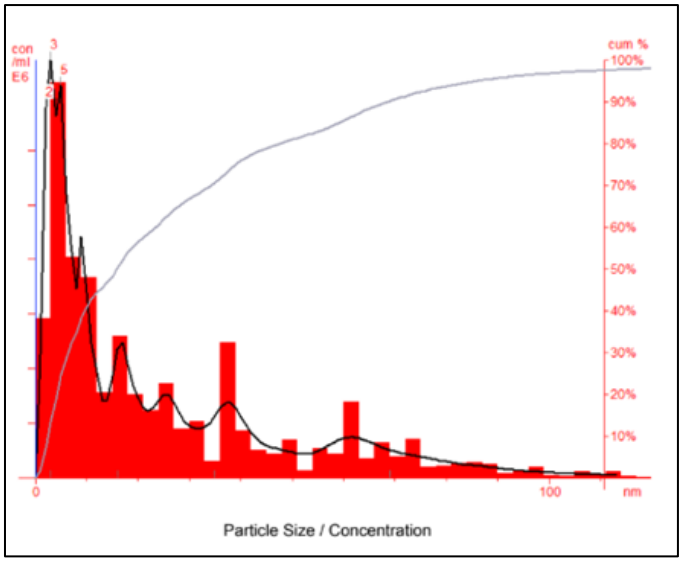


Figure 2 NTA of Ag@ZnO-CuO Nanocomposites

3.3. X-ray Diffractometer

X-ray powder diffraction analysis was performed to investigate the phase composition of the synthesized composites. The sample was scanned from 20 to 80 degrees. The XRD pattern of the sample is shown in Fig. 4. The diffraction peaks, which include (100), (002), (101), (102), (110), (103), (200), and (201), correspond to ZnO (JCPDS card no. 36-1451) and can be indexed by the hexagonal structure. The peaks marked by CuO, including (110), (111), (112), (202), and (022), correspond to CuO particles (JCPDS No. 48-1548) with a monoclinic structure. The peaks marked by Ag, including (111), (103), (110), and (220), correspond to Ag (JCPDS Card no. 04-0783) with a cubic structure. Distinct peaks of diffraction are observed for hexagonal ZnO and monoclinic CuO, along with four peaks of cubic Ag. Aside from the characteristic peaks, no additional peaks are detected, indicating the sample contains only ZnO, CuO, and Ag particles [21].

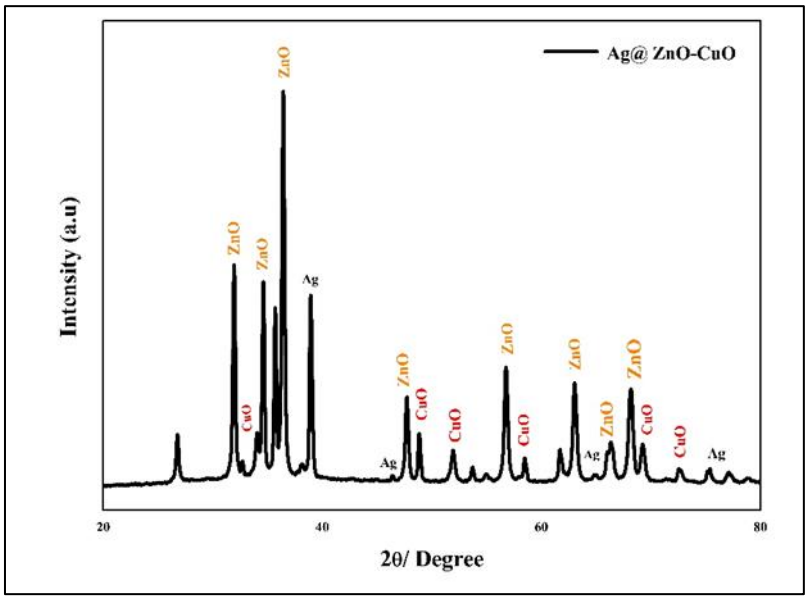


Figure 3 XRD of Ag@ZnO-CuO Nanocomposites

3.4. Scanning Electron Microscope and EDAX

The SEM images of Ag-doped ZnO–CuO nanocomposites reveal a heterogeneous surface morphology of densely packed granular and plate-like particles distributed in the matrix. Particles are seemingly irregular in shape, whereas their sizes vary from submicron to up to a few microns, suggesting the successful formation of a mixed metal-oxide composite. The

coarse and compact texture on the surface of the material gives an indication of the strong agglomeration among ZnO, CuO, and Ag-modified phases that may arise out of high surface energy. Crystalline fragments with sharp edges in some regions and rod- and flake-like structures in other regions are observed, manifesting the coexistence of a number of oxide phases. Shining white patches observed in some areas could be indicative of the presence of Ag nanoparticles, since they normally have higher electron reflectivity. Overall, SEM micrographs confirm the successful incorporation of Ag into the ZnO–CuO structure and reflect a well-developed composite morphology that might contribute to an increased surface activity for improvement of catalytic or antimicrobial performance

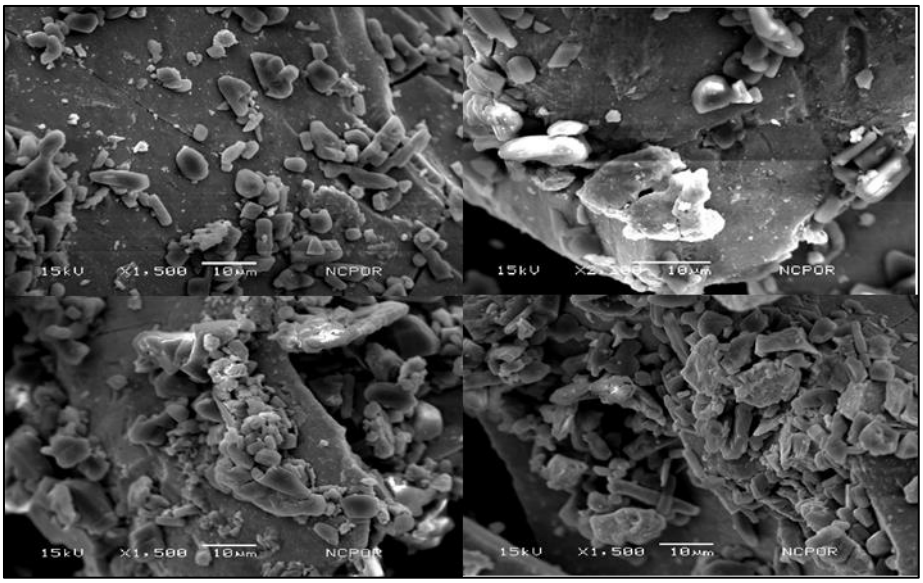
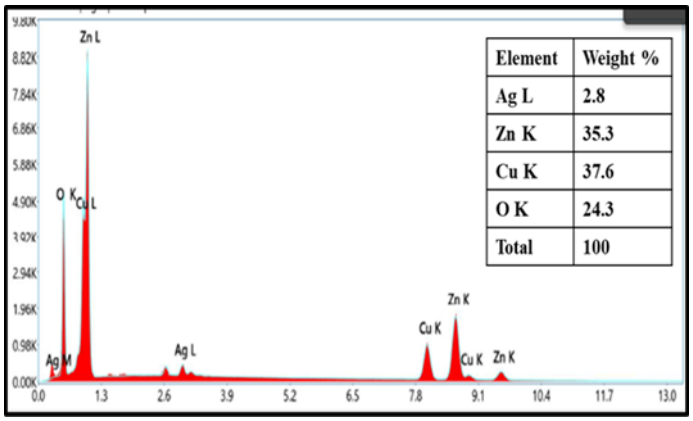


Figure 4 SEM Images of Ag@ZnO-CuO Nanocomposites

EDAX Analysis: The elemental composition of the Ag-doped ZnO–CuO nanocomposite is further confirmed from the EDAX spectrum. The prominent peaks corresponding to Zn, Cu, O, and Ag are clearly seen, which confirms that all the constituent elements were incorporated successfully. Quantitatively, Cu (37.6 wt%) and Zn (35.3 wt%) are present in major proportions, which is expected from the mixed metal-oxide framework. Oxygen contributes 24.3 wt%, which also supports the formation of oxide phases. A distinct Ag peak with 2.8 wt% confirms the effective doping of silver within the composites. In general, EDAX results confirm the elemental purity and successful synthesis of the Ag-doped ZnO–CuO nanocomposite.



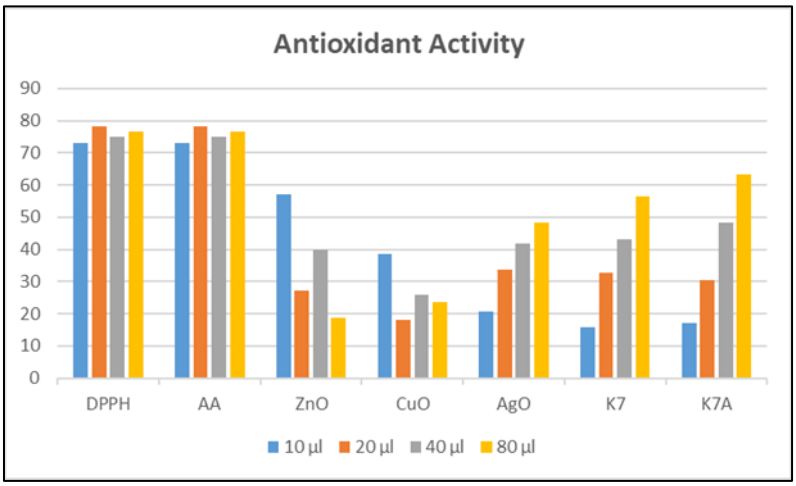
Element	Weight %
Ag L	2.8
Zn K	35.3
Cu K	37.6
O K	24.3
Total	100

Figure 5.1. EDAX of Ag@ZnO-CuO Nanocomposites

4. Antioxidant Activity

The antioxidant activity of the sample was evaluated using the DPPH free-radical scavenging assay across concentrations ranging from 10 to 80 µg/mL. The results showed a clear dose-dependent improvement in scavenging efficiency, as reflected by the gradual increase in absorbance values with increasing concentration. At lower

concentrations (10 and 20 µg/mL), the sample exhibited moderate antioxidant activity, while a stronger radical inhibition was observed at higher concentrations (40 and 80 µg/mL). This trend indicates that the compound possesses significant hydrogen-donating ability, which enhances its potential to neutralize free radicals. Overall, the data confirm that the antioxidant activity of the sample increases proportionally with concentration, demonstrating its effectiveness as a potential natural antioxidant agent [22], [23].



*K7- ZnO-CuO Nanocomposites.

*K7A- Ag@ZnO-CuO Nanocomposites.

Figure 6 Antioxidant Activity of Ag@ZnO-CuO Nanocomposites

The antioxidant activity results show a clear dose-dependent increase in free radical scavenging across all tested samples. The standard antioxidants, DPPH and ascorbic acid (AA), exhibited the highest and most consistent activity at all concentrations, confirming their strong radical-quenching efficiency. Among the synthesized materials, ZnO and CuO displayed moderate activity that increased steadily with concentration, while AgO, K7, and K7A showed comparatively higher scavenging potential at higher doses, particularly at 80 µL. Notably, the K7A sample demonstrated the greatest improvement with increasing concentration, indicating enhanced antioxidant performance. Overall, the results suggest that the prepared compounds, particularly K7 and K7A, exhibit promising antioxidant properties that intensify with increasing concentration.

5. Photodegradation analysis

For the photocatalytic degradation study of methylene blue (MB), a stock solution with a concentration of 100 mg L^{-1} was prepared by dissolving the dye completely in distilled water under continuous stirring. The solution was stored in a dark place at room temperature. Standard solutions of the dye were prepared by diluting the stock solution. The absorbance measurements were performed with a wavelength range from 190 to 1100 nm using UV-Visible-1900i (Shimadzu). A standard calibration curve was developed by plotting the known concentrations of methylene blue (MB) dye against their corresponding absorbance values measured at 664 nm.

The resulting linear equation from this plot was employed to estimate the unknown MB concentrations during the photocatalytic degradation studies. Each degradation experiment was conducted in triplicate to ensure reproducibility. The degradation efficiency of MB was calculated using the following equation:

$$\text{Degradation (\%)} = \frac{(C_i - C_t)}{C_i} \times 100$$

where C_i and C_t represent the initial and time-dependent concentrations of MB, respectively.

It was investigated whether photocatalytic activity could be enhanced by visible light irradiation to determine the efficiency of dye removal. Compared to doped ZnO-CuO nanocomposites, undoped ZnO-CuO nanocomposites were slower to degrade due to the limited charge separation efficiency and moderate surface area for dye adsorption. When electrons and holes are generated during irradiation, they are photocatalyzed to decompose the MB molecules.

However, rapid recombination of charge carriers reduced the overall degradation rate. In contrast, Ag-doped ZnO-CuO nanocomposites showed significantly enhanced photocatalytic activity. Adding Ag nanoparticles effectively suppressed the recombination of electrons and holes generated by photo-activation. It improved the charge separation efficiency and increased the number of reactive species with which dyes could be degraded (such as hydroxyl radicals and superoxide radicals). Furthermore, Ag nanoparticles enhanced visible light absorption, thereby speeding up degradation, due to their Surface plasmon resonance (SPR) effect [24], [25], [26].

Results indicate that ZnO Nanoparticles takes 135 min which takes longer time as compare to ZnO-CuO and Ag-doped ZnO-CuO nanocomposites, degraded more quickly than their undoped counterparts within 90 minutes, and more than 90% of MB dye has been degraded for Ag-doped samples. In contrast, undoped nanocomposites required a longer time to achieve similar levels of degradation, and undoped nanocomposites took longer to reach comparable levels of degradation. In this way, Ag doping enhances the photocatalytic activity of ZnO-CuO nanocomposites, making them more effective in removing organic dye pollutants from wastewater quickly and efficiently [27], [28].

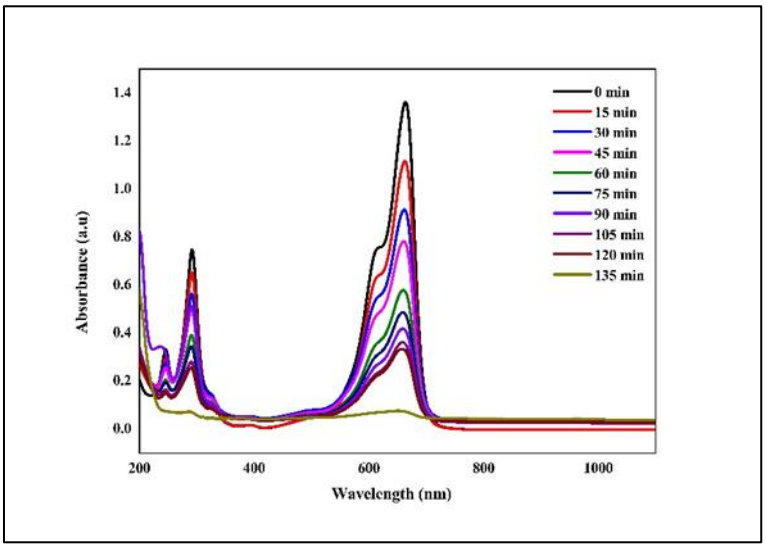


Figure 7 Photocatalytic Activity of ZnO Nanoparticles

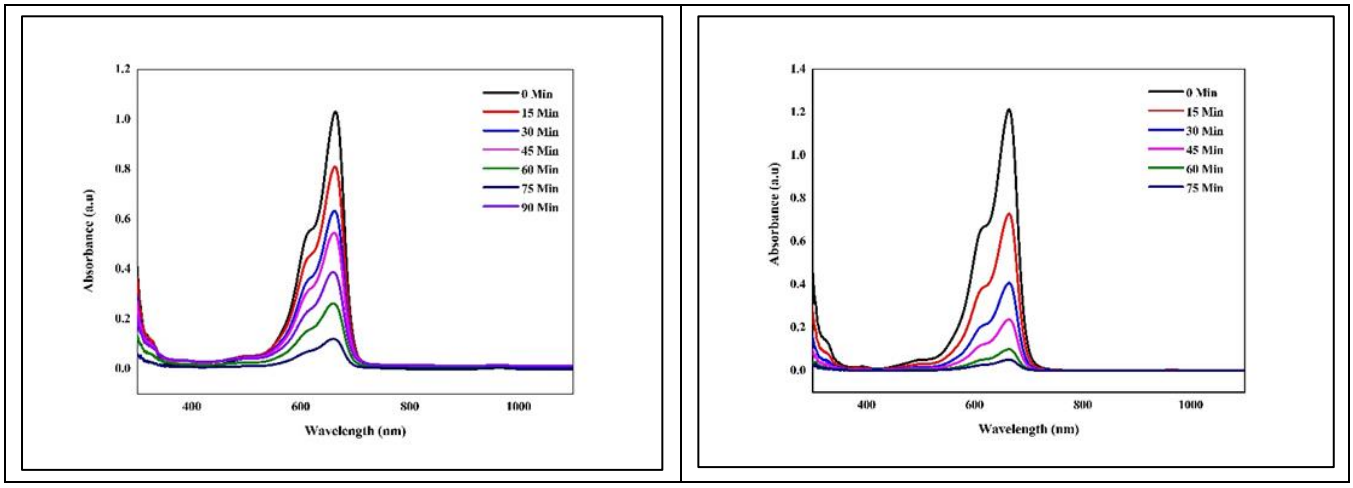


Figure 8 Photocatalytic Activity of ZnO-CuO and Ag-doped ZnO-CuO nanocomposites

6. Conclusion

In this experiment, Ag-doped ZnO-CuO nanocomposites were successfully prepared using a simple and low-cost coprecipitation technique. The structural and morphological results confirmed the successful preparation of well-crystallized ZnO-CuO nanocomposites with high Ag incorporation efficiency. The Ag doping and ZnO-CuO

heterostructure were essential for improving charge separation efficiency and rate of electron-hole pair recombination, which contributed greatly to the enhanced photocatalytic degradation efficiency upon light illumination. Additionally, the prepared nanocomposites showed high antioxidant activity with a marked concentration-dependent free radical scavenging ability. The results can be ascribed to the combined effects of ZnO, CuO, and Ag NPs. The above findings confirmed that Ag-doped ZnO-CuO nanocomposites are promising candidates for multiple applications, including the environmental and antioxidant-related biomedical sector.

Compliance with ethical standards

Disclosure of conflict of interest

No conflict of interest to be disclosed.

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