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Research Article Nanomaterial



Efficient photocatalytic degradation of organic dyes with Ag doped ZnO nanoparticles under UV light irradiation

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Abstract: Semiconductor based photocatalyst for degradation of cationic and anionic dye pollutants under UV light was demonstrated for wastewater treatment. In the present work, un-doped and Ag (mol% = 0, 0.05, 0.075 and 0.1)-doped ZnO nanoparticles were synthesized by co-precipitation method. X-ray diffraction (XRD) pattern confirms that the samples are found to be of highly crystalline hexagonal wurtzite structure. Scanning electron microscopy (SEM) image reveals that the morphology of the doped ZnO nanoparticles are highly affected by the addition of silver (Ag). Fourier transform infra-red (FTIR) and energy-dispersive spectra (EDS) demonstrates the presence of Ag in the ZnO lattice. UV–Vis absorption spectra shows that the Agdoped ZnO nanoparticles exhibit a red-shift with reduced bandgap energy compared with un-doped ZnO nanoparticles. Hence, our aim is to find the effect of Ag- doping in ZnO nanoparticles for the improvement of photocatalytic degradation. The photocatalytic activities of these Ag-doped nanoparticles were evaluated by measuring the rate of photo-degradation reaction of hazardous methylene blue (MB) and methyl orange (MO) dye under UV-light irradiation. Photocatalytic efficiency in the degradation of MB and MO dyes was compared with the efficiency of un-doped ZnO. The effects of various factors like solution pH, photocatalytic dosage and UV irradiation time with concentration of MB and MO on photocatalytic degradation were studied. It was found that cationic dyes shows better photo-degradation than the anionic dyes. The main objective of the present work was optimizing the various factor for better photocatalytic activity, complete mineralization and evaluating the photocatalytic-degradation mechanism.

Keywords:, Ag-Doping, Nanoparticles, Methylene Blue, Methyl Orange, Photocatalytic Degradation

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I. INTRODUCTION

Economically growing countries like India are facing environmental pollution due to industrial developments. In particular, industrial effluents discharged from cosmetics, textile, paper and paint industries contain high levels of organic contaminants including toxic chemicals and heavy metals¹. Untreated industrial effluents release into water system has become one of the most serious water pollutions that severely affect the ecosystem and human health. Appropriate treatment is essential for effective destruction of effluents before they are released into the ecosystem². There are several techniques available for the wastewater treatment such as activated carbon adsorption, chemical oxidation, coagulation, ion exchange, reverse osmosis, electrodialysis and advanced oxidation processes (AOP) have been found to be ineffective for the mineralization of toxic organic compounds³⁻⁷. Among them, photo-catalysis is an effective process that is used for heterogeneous photocatalytic degradation of organic effluents using a semiconductor as a photo-catalyst⁸. Many researchers are recently focused in conversion of organic dyes into harmless chemicals using photo-catalysts. ZnO is a versatile multifunctional wide band gap semiconductor like TiO₂ and it has numerous superior properties such as high electron transport, light sensitive, thermal and chemically stable, non-toxic and sustainable with hydrogen environment, 2,10 nanoparticles are effective photo-catalysts for degradation of toxic pollutants under UV irradiation. ZnO has better activity than TiO_2 for photocatalytic degradation of some organic dyes^{11,12}.The photocatalytic efficiency of ZnO nanoparticles mainly depends on the ability of electron-holes (e-/h+) pair formation under light illumination. However, the rapid recombination of photo-excited electron-holes (e⁻/h⁺), aggregation and low surface area of the bulk form are the main factors for the reduction of photocatalytic efficiency. Numerous methods, such as doping of metals and nonmetals, combining with different semiconductors are used to overcome these drawbacks. The metal (Ag) doping of ZnO is most efficient way to improve its photocatalytic efficiency, because metal doping accelerates the charge carrier separation and change its physical and chemical properties 13-²⁰. ZnO has been prepared by a variety of techniques such as hydrothermal method²¹, combustion²², and sol-gel method²³. Among these, co-precipitation²⁴ shows some advantages over the other methods. The present work explains the effect of various concentrations of Ag dopant on the structural and photocatalytic activity of ZnO nanoparticles. Furthermore, the samples were characterized by XRD, SEM, FTIR and UV-Vis spectrophotometer. To the best of our knowledge, this is the first report of application of Ag-doped ZnO nanoparticles for the degradation of cationic dye (methylene blue) and anionic dye (methyl orange) in water.

2. EXPERIMENTAL

2.1 MATERIALS AND METHODS

All the chemical used in the experiments are analytical grade and were used without further purification. Zinc nitrate hexahydrate $[Zn(NO_3)_2.6H_2O]$, silver nitrate $Ag(NO_3)$, methylene blue (MB), methyl orange (MO) and sodium

hydroxide [NaOH], were purchased from Alfa Aesar chemicals, India. Double distilled water (DDW) was used for all of the solutions.

2.2 Photo-catalyst Preparation

In a typical synthesis, 10 g of Zn (NO₃)₂ 6H₂O was dissolved into 20 ml of DDW. In another container, 8 g of NaOH in 100 ml DDW. Then NaOH added to the zinc nitrate solution. Both these solutions were added dropwise under continuous magnetic stirring until pH adjusted into 11. Then the above solution was heated in in a vacuum oven at 150 °C for Ih. Afterwards, white color precipitates were separated by centrifugation and washed twice with DDW followed by ethanol and dried at 100 °C for 3h. The dried participate was calcined at 450 °C for Ih and finally un-doped ZnO nanoparticles formed. For synthesis of Ag doped ZnO nanoparticles containing various concentrations (mol% = 0.05, 0.075 and 0.1) of silver nitrate dissolved in water were added to the zinc nitrate solution with stirring. The coprecipitation method was selected based on available literature as described above²⁴

2.3 Characterizations

The crystalline phases of un-doped and Ag-doped ZnO nanoparticles were determined by powder X-ray diffractometer (X'PERT PRO MPD) using CuKα radiation (λ = 0.15405 nm) operated at 40 kV voltage and 30 mA current with a scanning rate of 2° per min²¹. The particle size and morphological features of the samples were observed by scanning electron microscope (SEM-JEOL JS-6390) with energy dispersive X-ray spectrometer (EDX)²¹. Band gap energies of the samples were analysed from UV-visible spectra recorded UV-Vis double-beam using (Systronics: AU-2707). spectrophotometer Transform Infrared (FT-IR) spectra of prepared samples were recorded by using a FTIR Spectrometer (JASCO FTIR-410) in the range 400-4000 cm⁻¹ with a resolution of 4 cm⁻¹ at room temperature using KBr disc. Electrical characteristics (I-V) curve of the samples were carried out using a Keithley electrometer 2400 model

2.4 Evaluation of Photocatalytic activity

Photocatalytic activity of the un-doped and Ag-doped ZnO nanoparticles was evaluated for degradation of MB and MO dye solution under UV light irradiation. The photocatalytic procedure was conducted by adding 10 mg of photo-catalyst in 50 mL, 10 ppm of MB and MO dye solution. The reaction mixture was stirred for 30 min. under a dark to reach adsorption-desorption equilibrium. After that the dye solution was irradiated with an 8 W UV lamp was switched on. At different time intervals (0, 15, 30, 45, 60 and 75 min.) samples were taken out and centrifuged to separate the catalyst and then the supernatant was analysed in a UV-visible double beam spectrophotometer maximum wavelength (λmax) observed at ~664 and 464 nm to study the photocatalytic degradation. The photo-degradation efficiency (D%) of the MB and MO dye was calculated using the equation²⁵

 $(D\%) = [C-C_0/C_0] \times 100$

Where, C₀ and C are the initial and final degradation time of MB and MO (mg/L) dye solution used.

3. RESULT AND DISCUSSION

3.1 XRD Analysis

In order to investigate the crystal structure of un-doped ZnO and Ag doped ZnO nanoparticles were characterized by powder X-ray diffraction (PXRD) data. Fig. I(a) depicts the PXRD patterns of un-doped ZnO and Ag doped ZnO

nanoparticles with different concentrations of Ag, i.e. 0.05, 0.075 and 0.1%. The XRD pattern of all samples have strong diffraction peaks are assigned to (100), (002), (101), (102), (110), (103), (200), (112) and (201) planes of hexagonal wurtzite structure of ZnO in the Miller indices (JCPDS-36-1451) ²⁶. There is no extra peaks or other unidentified peaks corresponds to Ag or its oxide phases in the XRD pattern.

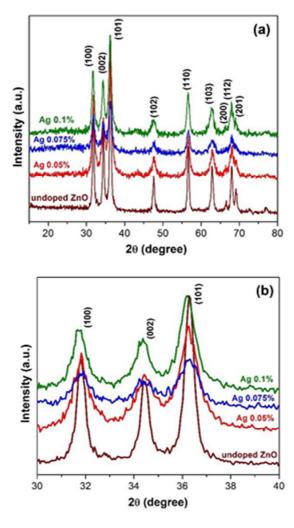


Fig. 1. (a) XRD patterns of un-doped and Ag-doped ZnO nanoparticles (b) expanded view of XRD patterns shift.

Table I. Lattice parameters and crystallite size of undoped and Ag-doped ZnO nanoparticles.									
Ag doping (mol%)	hkl	a[Å]	c[Å]	c/a	Average Crystallite Size (nm)				
ZnO	10 0 00 2 10 1	3.2555	5.2184	1.6029	23				
Ag _{0.05} Zn _{0.95} O	10 0 00 2 10	3.2562	5.2190	1.6028	20				

	I				
Ag _{0.075} Zn _{0.925} O	10 0 00 2 10	3.2584	5.2210	1.6023	19
$Ag_{0.1}Zn_{0.9}O$	10 0 00 2 10 1	3.2586	5.2240	1.6031	18

Fig. 1(b) shows a magnification of the peak at 31.8 (100), 34.3 (002) and 36.3° (101) were a slight peak shift to lower angles observed in the 0.05, 0.075, and 0.1% Ag-doped ZnO nanoparticles. This result confirms that Ag impurity has been successfully incorporated into the ZnO lattice structure. The lattice constants and average crystallite size of major XRD peaks calculated for different samples are listed in Table I. In our study, the effect of Ag doping on the average crystallite size (D) of was calculated for all samples by Debye-Scherrer's equation, and are found to be 23, 20, 19 and 18 nm, respectively²⁷. These results indicate that the D value in the Agdoped ZnO nanoparticles decreased as the doping concentration increased. Furthermore, the increasing Ag content decreases the lattice parameters and average crystallite size values. Other studies have found the same results were reported by Pal et al.28 in Codoped ZnO nanoparticles, Saravanan et al.29 in Ag-doped ZnO nanoparticles and Udom et al.30 in Ag-doped ZnO nanowires.

3.2 SEM with EDX Analysis

The surface morphology and chemical composition of the prepared samples were investigated by SEM with EDX. SEM images of undoped ZnO and Ag (0.05, 0.075 and 0.1 mol.%) doped ZnO nanoparticles are shown in Fig. 2(a-d). Fig. 2(a) shows the formation of irregular spherical morphology of the un-doped ZnO. From the Fig 2(b-d), it was precisely discovered that well prepared samples are in nanometer size and the surface morphology changes with the silver concentration. The variation of surface morphology with Ag dopant is due to the variation of number of nucleation sites²¹. The silver concentration reduces the particle size and it was confirmed from the XRD results. The obtained results are similar to the results reported by Pung et al³¹.

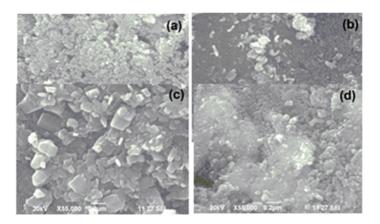
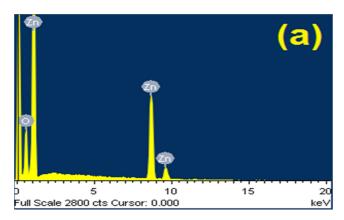


Fig. 2. SEM images of (a) undoped (b) 0.05, (c) 0.075 and (d) 0.1 mol.% Ag-doped ZnO nanoparticles.

The EDX spectra for un-doped ZnO and Ag (0.1%)-doped ZnO nanoparticles are shown in Fig. 3 (a, b). EDX spectrum of un-doped ZnO shows presence existence characteristic peaks of oxygen (O) and zinc (Zn) elements. The EDX pattern of Ag-doped nanoparticles also include elements such as existence of silver (Ag) together with Zn and O, indicating the successful doping of Ag in the ZnO matrix³¹.



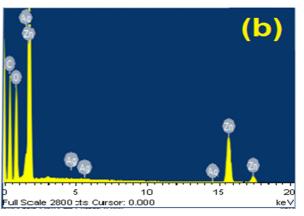


Fig. 3. EDX images of (a) un-doped and (b) 0.1 mol.% Ag-doped ZnO nanoparticles.

3.3 UV-Vis Analysis

In order to investigate the optical properties of un-doped ZnO and Ag (0.05, 0.075 and 0.1 mol.%) doped ZnO nanoparticles were investigated by UV-Vis. absorption spectroscopy. Fig. 4 (a-d) shows the UV-Vis absorption spectra of un-doped ZnO and Ag (0.05, 0.075 and 0.1 mol.%) doped ZnO nanoparticles. Un-doped ZnO has an absorption cut-off edge was observed around 370 nm. The

adsorption of Ag-doped ZnO nanoparticles was shifted to the longer wavelength from ZnO with the increase of the molar ratio of Ag (0.05, 0.075 and 0.1 mol.%). After Ag-doping display an additional hump absorption in the visible region observed at 373, 375 and 377 nm. The calculated optical band gap energies were 3.35, 3.33, 3.30 and 3.28 eV for un-doped ZnO and Ag-doped ZnO nanoparticles, respectively. According to some previous reports similar result was observed by Hassan et $al.^{32}$.

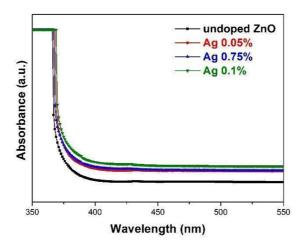


Fig. 4. UV-Vis absorbance spectra (a) un-doped (b) 0.05, (c) 0.075 and (d) 0.1 mol.% Ag-doped ZnO nanoparticles.

3.4 FT-IR Analysis

The FTIR spectra of un-doped ZnO and Ag (0.1%)-doped ZnO nanoparticles are presented in Fig. 5 (a, b). The absorption peaks present at 3441 and 1625 cm⁻¹ which corresponds to the -OH stretching and bending vibration of the water molecule³³.

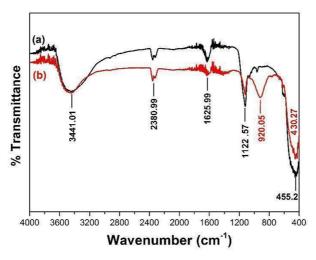


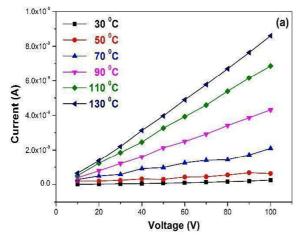
Fig. 5. FT-IR spectra of (a) un-doped and (b) 0.1 mol.% Ag-doped ZnO nanoparticles.

The band at 2380 cm⁻¹ corresponds to symmetric and asymmetric C–H bond³⁴. The observed FTIR spectrum exhibits several well-defined absorption bands at 1122 and 920 cm⁻¹ and they are attributed to sulphate and C=C bonds, respectively³⁵. The absorption peaks appears at 400 and 550

cm⁻¹ were assigned to the metal-oxygen (M-O) stretching mode³⁶ and the absorbance peak noticed in the spectra around 455 and 430 cm⁻¹ corresponds to the presence of Zn-O and Ag-O stretching bond, respectively.

3.5 I-V Characteristics

Electrical properties of un-doped ZnO and Ag (0.1%)-doped ZnO nanoparticles was measured by using the I-V characteristics and it is presented in Fig. 6 (a, b). From IV curves, electrical conductivity of un-doped ZnO and Ag (0.1%)-doped ZnO taken under varying temperatures from 30 to 130 °C.



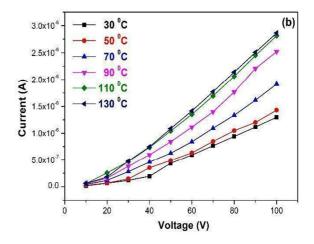


Fig. 6. I-V characteristics of (a) un-doped and (b) 0.1 mol.% Ag-doped ZnO nanoparticles.

This study reveals that (0.1%) Ag-doped ZnO nanoparticles shows better conductivity than the un-doped ZnO and it may due to the presence of more charge carrier's mobility³⁷.

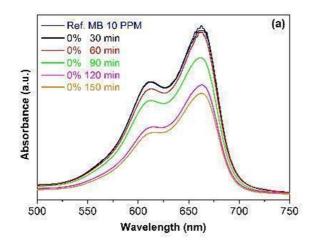
3.6 Photocatalytic Activity

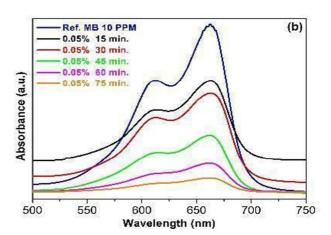
3.6.1 Optimized Factors to the Catalytic Activity

The photocatalytic degradation of un-doped ZnO and Ag-doped ZnO nanoparticles were selected for the evaluation of

photocatalytic activity under UV-visible light. In order to study the effect of the UV light on the degradation of MB and MO dye. The dyes were prepared in various concentrations like acidic, neutral and alkaline medium. The photocatalytic activity was carried out and optimised for un-doped ZnO catalyst with MB and MO dye concentration of 10 ppm, catalyst concentration of 10 mg, pH = 6 and UV irradiation time up to 150 min.

3.6.2 Impact of UV irradiation on Degradation of MB and





For the above mentioned conditions the photocatalytic degradation was carried out for Ag-doped ZnO nanoparticles. The Fig. 7(a-d) shows the comparative spectra of degradation of MB dye with un-doped ZnO and Ag (mol%

= 0.05, 0.075, 0.1)-doped ZnO nanoparticles with respect to time. The high intensity absorption peaks was observed at 664 nm for MB dye. It was observed that the degradation of MB dye increases with increase of dopant concentration²⁴.

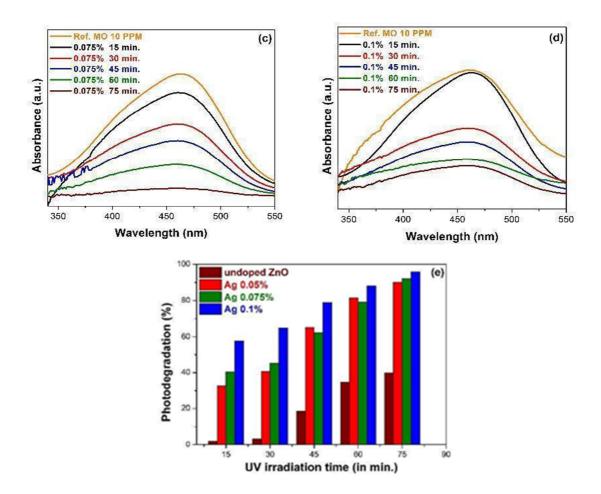


Fig. 7. MB dye degradation of (a) un-doped (b) 0.05, (c) 0.075, (d) 0.1 mol.% Ag-doped ZnO nanoparticles (MB dye:10 ppm, catalyst dose:10 mg, pH=6) and (e) effect of irradiation time of UV on MB photo-degradation.

The efficiency of un-doped and Ag-doped ZnO nanoparticles under UV light reaches 38, 90, 92 and 95% after 75 min light irradiation. Ag (0.1%)-doped ZnO nanoparticle shows the highest degradation efficiency of 95% under UV radiation for

MB dye, while under similar conditions, a much lower efficiency (38%) was observed for un-doped ZnO nanoparticles under visible light radiation²⁴.

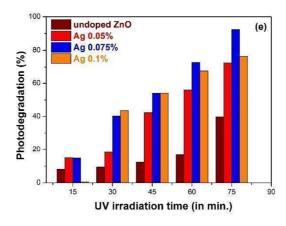


Fig. 8. MO dye degradation of (a) un-doped (b) 0.05, (c) 0.075, (d) 0.1 mol.% Ag-doped ZnO nanoparticles (MO dye:10 ppm,

catalyst dose:10 mg, pH=6) and (e) effect of irradiation time of UV on MO photo-degradation. The comparative spectra for degradation MO dye with un-doped ZnO and Ag (mol% = 0.05, 0.075, 0.1)-doped ZnO nanoparticles with respect to time was presented in Fig. 8(a-d). The high intensity absorption peaks of MO dye were observed at 464 nm. The effect of irradiation time of UV on MO

dye degradation is shown in Fig. 8 (e). The degradation efficiency increases with increase of dopant concentration and the obtained values are 36, 72, 92 and 76%, respectively for un-doped, 0.05, 0.075 and 0.1 mol.% Ag-doped ZnO nanoparticles. It can be seen that maximum degradation (92%) of MO dye was achieved for Ag (0.075%)-doped ZnO nanoparticles in 75 min. It should be noted

that MB was degraded more faster than MO in the presence of the Ag-dopant. The possible photocatalytic degradation mechanism of MB and MO on Ag-doped ZnO nanoparticles under UV-visible light irradiation can be explained from the equation I to 6. During the catalytic process, photons can excite electrons in the valence band (VB) to be moved up to the conduct band (CB) and generate the same amount of electron-hole pair (e- and h+). The photo-excited electrons will be transferred from ZnO to Ag nanoparticles under

the potential energy. The photocatalytic activity is effective thereby preventing the recombination between electrons and holes on the surface of ZnO. Afterwards, the electron on Ag nanoparticles can produce the O_2^- superoxideradical, while the holes in the VB can react with H_2O_2 to produce OH hydroxylradical³⁸. These free radicals can degrade organic compounds to CO_2 and $H_2O_3^{11}$

$$ZnO + h\gamma \longrightarrow ZnO(h^{+}_{VB} + e^{-}_{CB}) \qquad (1)$$

$$ZnO (e^{-}_{CB}) + Ag \longrightarrow ZnO + Ag (e^{-}_{CB}) \qquad (2)$$

$$Ag (e^{-}_{CB}) + O_{2} \longrightarrow O_{2}^{-} + Ag \qquad (3)$$

$$ZnO (h^{+}_{VB}) + H_{2}O \longrightarrow ZnO + H^{+} + OH \qquad (4)$$

$$(\cdot O_{2}^{-} + \cdot OH) + dye \longrightarrow degradation \qquad (5)$$

$$(MB \text{ or } MO) \qquad products$$

$$(\cdot O_{2}^{-} + \cdot OH) + degradation \longrightarrow CO_{2} + H_{2}O \qquad (6)$$

$$products$$

Products Till now, many studies have proven that electrons can transfer from Ag to the CB of ZnO. Then, they are scavenged by adsorbed O_2 molecules to yield superoxide radical anions (O_2) to degrade dye molecules³⁸. Therefore, the photocatalytic activity will be enhanced.

4. CONCLUSION

In summary, various mol% of Ag-doped ZnO nanoparticles was synthesized using co-precipitation method. The structural, morphological, optical and electrical properties as well as the photocatalytic activity under UV irradiation of the resultant samples were characterized by XRD, SEM with EDX, UV–Vis FT-IR and IV analysis. Doping of Ag modified the morphology and average crystallite size of ZnO nanoparticles. In photocatalytic activity, Ag-doped ZnO nanoparticles enhances the degradation of MB dye under UV irradiation than the degradation of MO dye. The optimum

reaction conditions for the degradation of MB and MO under UV-visible light irradiation of samples were pH= 6.0, catalyst dosage of 10 mg, and MB and MO concentration of 10 ppm. The possible mechanism of photocatalytic activity was studied and Ag enables the trapping of electrons in Ag-doped ZnO nanoparticles. Ag (0.1 mol%)-doped ZnO nanoparticles may be assumed as a promising photo-catalyst for the degradation of toxic organic effluents at room temperature.

5. AUTHOR CONTRIBUTION STATEMENT

Mr. Senthil Kumar, M conceptualized and gathered the data with regard to this work. Dr.Arunagiri, C analysed these data and supervised

6. CONFLICT OF INTEREST

Conflict of interest declared none.

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