

Photocatalytic Degradation of Methyl Orange by Ag-N Co-Doped ZnO Nanoparticles

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Abstract

In this study synthesis, characterization and photocatalytic application of Silver, Nitrogen co-doped Zinc Oxide nanoparticles has been made. Four series of zinc oxide catalyst, (ZnO, Ag-ZnO, N-ZnO and Ag-N-ZnO) were prepared. All these oxides nanocomposites were prepared by aqueous combustion synthesis method using $Zn(CH_3COO)_2 \cdot 2H_2O$, $AgNO_3$ and N_2H_4CO as precursors. The as-synthesized nanoparticles were characterized by XRD and UV-Vis DRS techniques. From XRD analysis, it was clearly observed that the as-synthesized samples have ZnO predominantly N-ZnO and Ag-ZnO phases as well. The UV-Vis spectra study assured that the band gap decreased as N and Ag doped to ZnO and hence the absorption band shifts toward the longer wavelength region (red shift). The experimental results showed that Methyl orange degradation depends on the doped elements. In all cases, the photocatalytic activity gradually increased with addition of doping elements N and Ag. *As-synthesized Ag and N co-doped ZnO showed higher photoactivity than the Ag or N-doped ZnO as well as calcined ZnO in both UV and solar irradiation.*

Keywords: Degradation; Doping; Nanoparticles; Methyl orange; Photocatalysis; Zinc Oxide.

1. INTRODUCTION

The environmental problems related to industrial wastewater are becoming more & more complex with increasing diversity of industrial products. At present time, great efforts are being expended to minimize the quantity & toxicity of industrial effluents. Considering the inherent toxic nature of industrial effluents & unavailability of clean technologies, the necessity of developing new methodologies for efficient treatment of effluents is evident.^[1]

Textile industries produce large amounts of wastewater due to high consumption of water primarily in dyeing & finishing operations. A well-known characteristic of textile wastewater is a high content of polluting compounds.^[2] The sources of the polluting compounds when cotton is utilized are the natural impurities extracted from fiber, processing chemicals & dyes. The main problem occurring is that the color that remains due to the dyestuff used may cause disturbance to the agricultural system.^[3]

The dyes usually have synthetic origin & complex aromatic molecular structure which make them more stable & more difficult to be biodegraded. As international environmental standards are becoming more stringent^[4], technological systems for the removal of organic pollutants, such as dyes have been recently developed. Among them physical methods such as adsorption, biological methods (biodegradation) & chemical methods (chlorination, ozonation) are the most frequently used.^[5]

The traditional processes for treatment of these effluents prove to be insufficient to purify the important quantity of waste waters after the different operations of textile dyeing & washing. Some methods such as combined coagulation, electrochemical oxidation, & active sludge have recently been investigated & proved to be adequate. Other methods such as flocculation, reverse osmosis & adsorption on activated carbon have also been tested.^[6] The drawbacks of these methods are mainly the creation of more concentrated pollutant-containing phase. The processes by bacterial beds are less adapted because of the fluctuations of wastewater composition.^[7] Therefore, it is necessary to find an effective method of wastewater treatment capable of removing color & toxic organic compounds from textile effluents.

In recent years an alternative to conventional methods, is "advanced oxidation processes" (AOPs), based on the generation of very reactive species such as hydroxyl radicals, that oxidizes a broad range of organic pollutants quickly & non-selectively.^[8] Among AOPs, heterogeneous photocatalysis seems to be an attractive method as it has been successfully employed for the degradation of various families of organic pollutants including the dyes.

Heterogeneous photocatalysis is a process in which a combination of photochemistry & catalysis is operable & implies that light & catalyst are necessary to bring out a chemical reaction.^[9] The reason for the increased interest for the photocatalytic method is that the process may use atmospheric oxygen as the oxidant & it can be carried out under ambient conditions. The catalysts characterized by semiconductor usually are oxides that can be more appealing than the more conventional chemical oxidation methods because semiconductors are inexpensive, nontoxic, & capable of extended use without substantial loss of photocatalytic activity.^[10] The

objective of the present research was, therefore, to synthesize and characterize nano size zinc oxide in the presence of silver and nitrogen and to evaluate its photocatalytic potential for degradation of methyl orange under different energy sources.

2. EXPERIMENTAL METHODS

2.1 Synthesis

2.1.1 Preparation of Zinc oxide nanoparticles

60 g of zinc acetate dihydrate ($\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$) was added to 1.5 liters of deionized water under vigorous stirring till homogeneous solution was obtained. 0.1 M of sodium hydroxide was added into the above solution under continuous stirring till pH value reached 12. The solution was transferred to glass reactor & maintained at 100°C for 7 hrs in an oven. The product was cooled to room temperature. The precipitates were centrifuged & washed with deionized water & ethanol several times.^[11] The product was dried in an oven at 60°C for 2 hrs and 40 g of zinc oxide was obtained. The product was labeled as uncalcined zinc oxide (Znc). A portion of this product (9 g Znc) was calcined at 400°C for 4 hrs, cooled to room temperature, grinded in agate mortar & labeled as calcined zinc oxide (Zc).

2.1.2 Preparation of Silver-doped zinc oxide

9 ml of silver nitrate (0.1 M) was added to 9 g of uncalcined zinc oxide (Znc). The sample was agitated & heated at 110°C for 30 minutes. The powder was cooled to room temperature, calcined at 400°C for 4 hrs & then grinded in an agate mortar.^[12] The product obtained was labeled as silver-doped zinc oxide (AZ).

2.1.3 Preparation of Nitrogen-doped zinc oxide

15 g of zinc oxide was added to 30 g of urea, was grinded in an agate mortar & mixed well. The mixture was calcined in a ceramic crucible at 400°C for 4 hrs, cooled to room temperature & was grinded in an agate mortar.^[13] The product was labeled as nitrogen-doped zinc oxide (NZ).

2.1.4 Preparation of Silver-nitrogen co-doped zinc oxide

9 ml of silver nitrate (0.1 M) was transferred into ceramic crucible containing 9 g of NZ, & was agitated with glass rod. The crucible was put in an oven for drying at 110°C for 30 minutes. The dried powder was calcined at 400°C for 4 hrs, cooled to room temperature & was grinded in an agate mortar. The obtained product was labeled as silver-nitrogen co-doped zinc oxide (ANZ).

2.2 Characterization

For determining the particle size and type of oxides of the as-synthesized samples, X-ray diffraction (XRD), spectra were obtained on a BRUKER D8 Advanced XRD, AXS GmbH, Karlsruhe, West Germany using a Cu target $K\alpha$ ($\lambda = 1.5406 \text{ \AA}$). The measurements were made at room temperature using accelerating voltage & applied current as 45 kV and 40 mA, respectively. It was operated in the step scan rate at 0.02° (1 s step time over 2° range of $5.0 - 79.9^\circ$).

To observe the absorption edge of the as-synthesized samples, UV-Vis absorption spectra was measured using SANYO spectrophotometer model lambda (SP65). 0.1 g of the photocatalyst was dissolved in 50 ml of deionized water. The UV-Visible spectrometer was turned on & made the baseline using the deionized water. Then the absorbance of the photocatalyst solution was measured using a quartz tube.

2.3 Photocatalytic Activities

A 0.1 g of the as-synthesized photocatalyst powder & 100 ml sample was taken in the reactor tube & the suspension was stirred in dark for 40 minutes to obtain adsorption/desorption equilibrium before irradiating the sample in the reactor. During irradiation of the sample at room temperature by UV/Vis radiation, air was purged into the sample solution.^[14] The UV lamp (Philips) that predominantly emit at 254 nm with the definite power 12 W, 230 Volts & 50 Hz frequency was employed as UV source & positioned parallel to reactor. The distance between the top of reactor & UV lamp was 12 cm & the light intensity was $6.4 \times 10^{-2} \text{ W/cm}^2$.

9 ml of the sample was withdrawn at 15 minutes regular time interval. The suspension was centrifuged at 2500 rpm for 5 minutes & filtered to remove the catalyst particles before measuring absorbance. The absorbance of the clear solution was measured at $\lambda_{\text{max}} = 622 \text{ nm}$ using UV/Vis spectrophotometer (SP65) for quantitative analysis. Photocatalytic degradation of sample was monitored spectrophotometrically using each of the as-synthesized photocatalysts & was compared with commercial zinc oxide as well as with non calcined zinc oxide photocatalysts. Percentage degradation of sample was calculated using the relation $\% \text{ degradation} = [(A_0 - A_t)/A_0] \times 100$.^[2]

3. RESULT AND DISCUSSION

3.1 XRD Analysis

Figure 1 (A, B, C, D) shows the XRD spectra of Zc, AZ, NZ, & ANZ. The observed reflections in XRD spectra at $2^\circ = 34.48^\circ, 36.38^\circ, 56.68^\circ$ & 62.76° correspond to crystal planes of (002), (101), (110) and (103), respectively

which suggest hexagonal wurtzite structure of zinc oxide ^[15]. The XRD pattern of calcined zinc oxide (Zc) exhibits narrow and sharp diffraction peaks indicating its good crystalline structure.

The crystal structure of zinc oxide calcined at 400^oC studied and reported wurtzite crystal structure of zinc oxide. ^[16] At low temperature crystalline structure was poor which improved on increasing the temperature up to a certain critical temperature. Beyond that crystallinity decreased, may be due to phase change. An XRD spectrum of silver-doped zinc oxide (AZ) (Figure 1B) is the exact reproduction of the spectra of calcined zinc oxide (Zc) (Figure 1A). It suggested that doped silver atoms may be located just at the zinc oxide crystal surface rather than at lattice sites in zinc oxide. Therefore, it may not alter its crystal structure.

XRD spectra of nitrogen-doped zinc oxide (NZ) (Figure 1C) showed weak and broad diffraction peaks. It appears that nitrogen-doping disfavors the crystalline structure of zinc oxide at 400^oC. In case of silver-nitrogen co-doped zinc oxide (ANZ), its XRD spectra (Figure 1D) again have narrow and sharp diffraction peaks. The difference in the XRD spectra of nitrogen-doped zinc oxide and silver-nitrogen co-doped zinc oxide may be attributed to the difference in their calcinations periods. The former (NZ) sample was calcined only for 4 hrs whereas the later (ANZ) sample was calcined for much longer periods (8 hrs). A prolonged thermal treatment in case of ANZ may induce crystallinity. The order of crystallite size of as-synthesized photocatalysts was: Zc (45.40nm)~ AZ > ANZ(22.74 nm) > NZ(8.53 nm). Lower size of ANZ than Zc and AZ may be due to longer time of calcinations in case of ANZ.

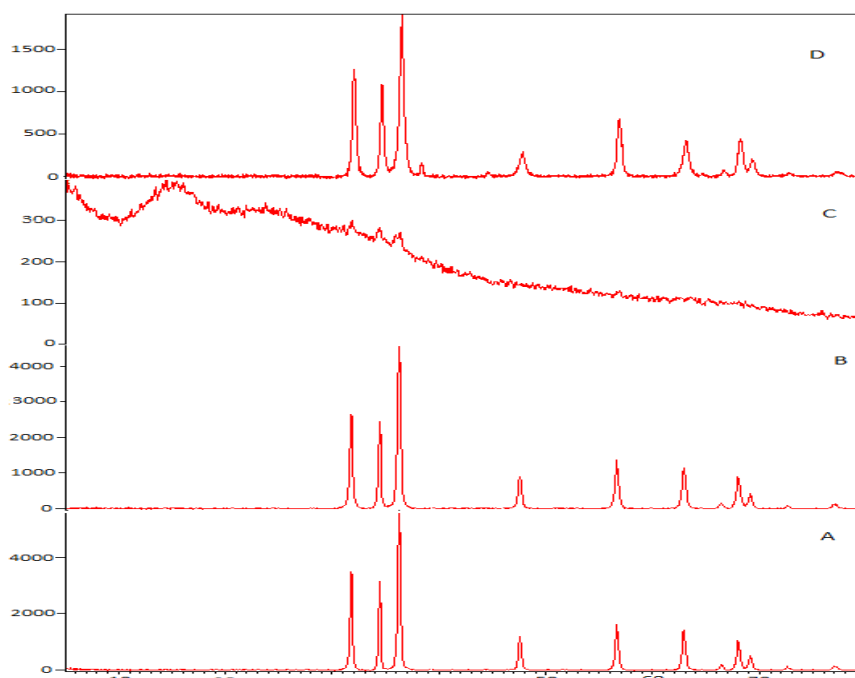


FIG (1). XRD SPECTRA. A) Calcined Zinc Oxide (ZC), B) Silver-doped Zinc Oxide (AZ), C) Nitrogen doped Zinc Oxide (NZ), D) Silver-Nitrogen co-doped Zinc Oxide (ANZ)

3.2 Uv-vis diffuse absorption

The optical absorption spectra of photocatalysts i.e. (Zc, AZ, NZ and ANZ) are depicted in Figures 2(A-D). UV-Visible absorption spectra for Zc, AZ, NZ and ANZ are: 379, 402, 531, and 543 nm, respectively. UV-Vis absorption spectra of NZ and ANZ photocatalysts are well extended to visible region spectrum compared to Zc and AZ. This may be due to the modification of electronic levels of zinc oxide by nitrogen-doping. The band gap energies (E_g) of photocatalysts Zc, AZ, NZ, and ANZ were found as 3.27, 3.09, 2.34, 2.28 eV, respectively. Nitrogen-doping in zinc oxide resulted significant decrease of band gap energy of NZ and ANZ. ^[17] Incorporation of silver in Zc caused comparatively smaller change relative to nitrogen incorporation. It may be because of doped silver adheres near the surface of zinc oxide and does not modify the band gap energy of zinc oxide. ^[18] Silver only traps the electrons and prevented from recombination of electrons in the conduction band and holes in the valence band. ^[19]

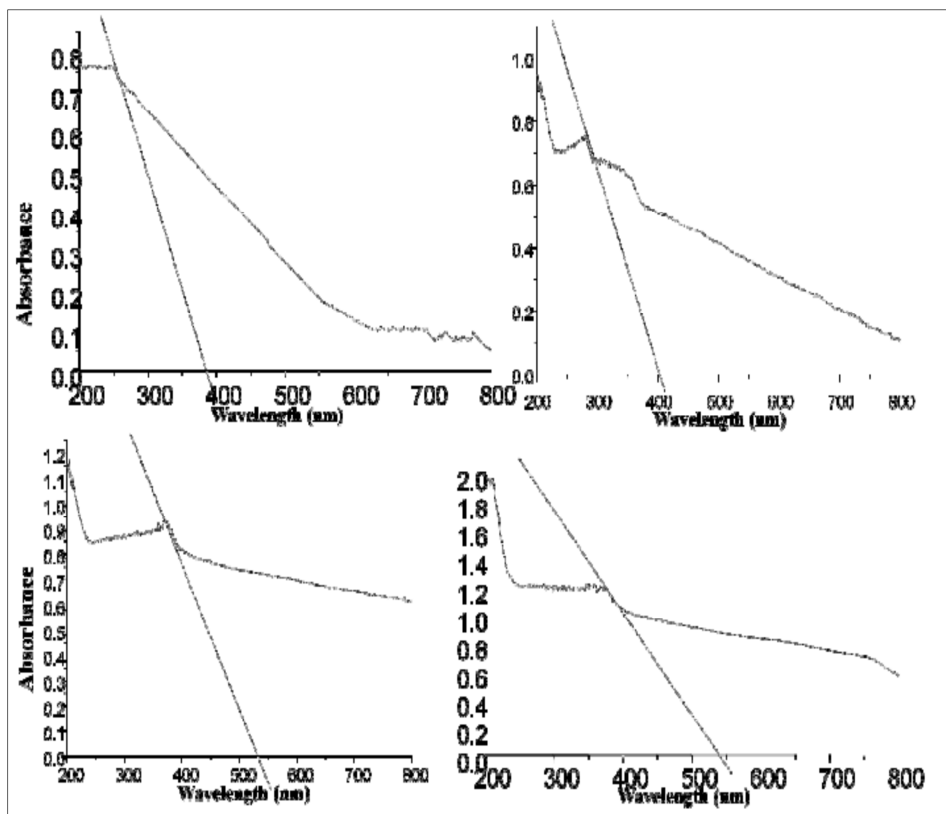


FIG (2). Uv-visible absorption spectra calcined zinc oxide (379 nm) (a), silver-doped zinc oxide (402 nm) (b), nitrogen-doped zinc oxide (531 nm)(c) & silver-nitrogen co-doped zinc oxide (543 nm) (d) respectively.

3.3 Photocatalytic degradation studies

Absorbance of the sample as a function of time under no-irradiation, solar irradiation and UV irradiation were recorded. Plots of absorbance of the sample at 622 nm as a function of time under no-irradiation, solar irradiation and UV irradiations are given Figures 3-5. The percentage adsorption of the sample without irradiation using the adsorbents: Znc, Zc, AZ, NZ, and ANZ at 180 minutes were 4.6%, 7.96%, 3.12%, 2.3%, and 7.13%, respectively (Figure 3). The percent of photo-degradation of the sample under solar and UV irradiations using these photocatalysts, Znc, Zc, AZ, NZ, and ANZ at 180 minutes were 17.04%, 47.06%, 74.12%, 84.92%, 91.65% and 11.08%, 37.11%, 56.62%, 50.95%, and 60.96% as depicted in Figures 4 and 5, respectively.

For a comparative study the photocatalytic degradation of sample solution was also carried out using uncalcined and commercial zinc oxide. As can be seen from the Figures 4-6, the photocatalytic activity of Znc is lowest among studied photocatalysts under both UV and solar irradiations. This may be due to its small specific surface area or amorphous nature.

Photocatalytic activity of AZ is higher than Zc both under solar as well as UV irradiations. It may be because doping of a noble metal, such as silver, in a semiconductor which can entrap the photo-generated electron thereby diminishing the recombination of electron-hole pairs, and thus resulting in higher photocatalytic activity of AZ. It is worthwhile to compare the photocatalytic activity of NZ and Zc under both solar as well as UV irradiations. Nitrogen-doped (NZ) zinc oxide shows more photocatalytic activity than Zc. This may be due to narrowing of band gap energy on doping nitrogen to zinc oxide. It is also possible to compare the photocatalytic activity of AZ and NZ under both solar as well as UV irradiations. It is observed that under solar irradiations the percentage degradation of sample is higher for NZ than AZ as seen in Figure 4. Under UV irradiations the percentage degradation of the sample solution is higher for AZ than NZ as depicted in Figure 5.

The nitrogen incorporated in the crystal of zinc oxide, may modify the electronic levels of zinc oxide resulting in its band gap narrowing, thus, rendering it more responsive to sunlight.^[13, 17] In case of AZ under UV irradiations, since silver can trap the photo-generated electron the recombination of electron-hole pair is inhibited and thus enhancing its photocatalytic activity.^[20] However, in case of NZ, under UV irradiations although there is equal probability of formation of electron-hole pairs since there is no other species which can trap the photo-generated electrons and hence there is lower photoactivity of NZ than AZ.

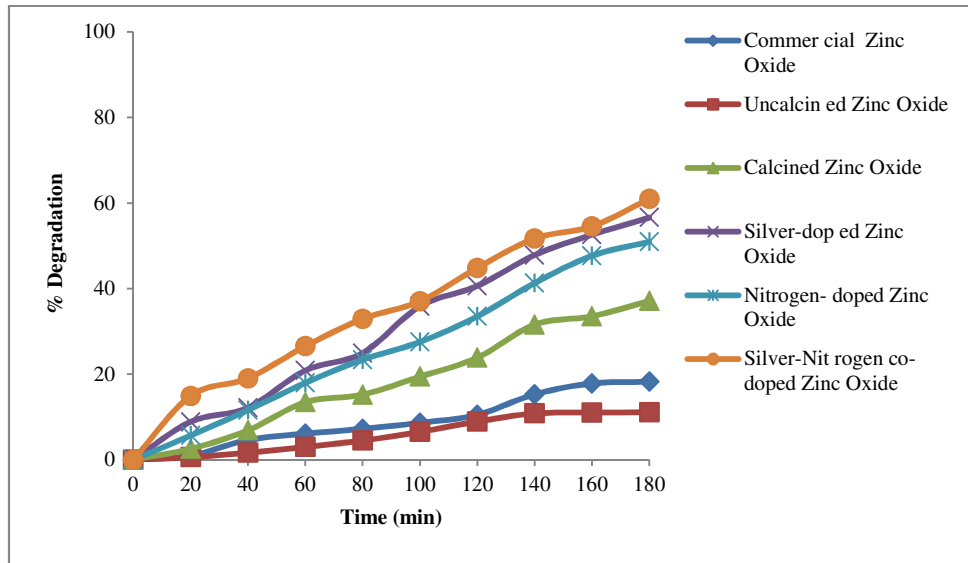


Figure (3). Plots of percentage adsorption of methyl orange (MO) as a function of time (without irradiation)

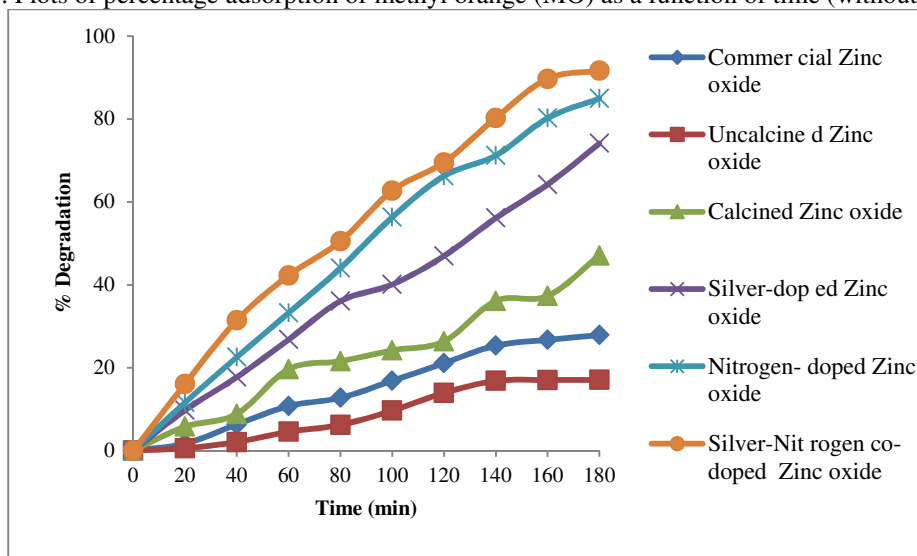


Figure (4). Plots of percentage degradation of methyl orange (MO) as function of time (under solar irradiation)

^[18] stated that silver doping may not modify the band gap of zinc oxide because it has similar wavelength with calcined zinc oxide but enabled the degradation of methyl blue under solar irradiations due to scavenging of photo-generated electrons by silver which retards carrier recombination. The photocatalytic activity of ANZ is highest among all the studied photocatalysts under both solar as well as UV irradiations. It may be due to the synergetic cumulative effect of silver and nitrogen co-doping that enhances the photocatalytic activity. In case of NZ, however, there will be greater probability of formation of photo-generated electron-hole pairs resulted from the narrow band gap increasing the number of species participating in photocatalytic degradation of waste sample under solar irradiations. ^[17]

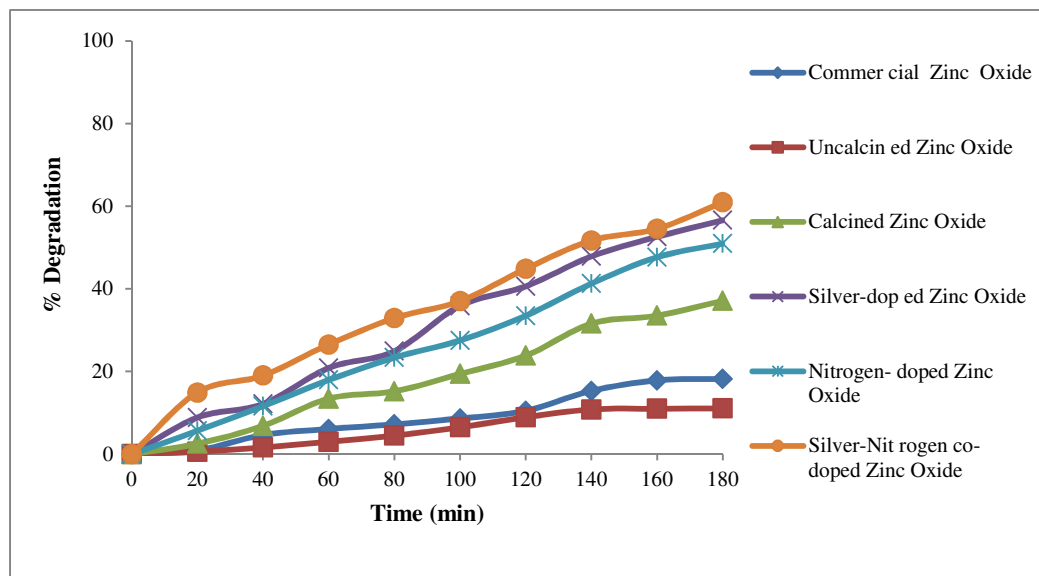


Figure (5). Plots of percentage degradation of methyl orange (mo) as function of time (under uv irradiation)

3.4 Kinetic studies of photocatalytic degradation of sample

Kinetics of adsorption of waste sample (with an initial concentration of 25 mg/l) under no radiation, photocatalytic degradation under solar and UV radiation follow pseudo first order kinetics. It is evident from the observed linear plots of $\log(C_0/C_t)$ versus time depicted in Figures 6-8.

TABLE- 1 . Values of adsorption rate constant (k) of waste sample using different photocatalysts without radiations ($k \times 10^{-4} \text{ min}^{-1}$)

Time (min)	k_{Znc}	k_{Znc}	k_{Zc}	k_{AZ}	k_{NZ}	k_{ANZ}
0	0	0	0	0	0	0
20	2.29	1.19	3.42	1.15	0.40	3.02
40	1.77	1.29	3.43	0.63	0.55	3.03
60	2.36	1.52	4.19	1.17	0.37	3.75
80	2.31	1.69	4.92	1.41	0.74	4.59
100	2.32	2.09	5.12	1.59	1.09	4.86
120	2.57	2.52	5.01	1.72	1.25	4.87
140	2.61	2.35	4.96	1.63	1.16	4.71
160	2.59	2.49	4.93	1.71	1.24	4.39
180	2.71	2.57	4.57	1.80	1.27	4.14

The rate constants (k) of waste sample adsorption (photocatalytic degradation) have been calculated using the relation $2.303 \log(C_0/C_t) = kt$. Where C_0 is absorbance at initial stage and C_t is the waste sample absorbance at time, (t).^[21] The rate constant (k) is obtained from the slope of linear plot of $\log C_0/C_t$ versus time. The rate constant for adsorption of sample using photocatalysts of Znc, Zc, AZ, NZ, and ANZ without irradiation are $(2.56, 3.84, 1.79, 1.27 \text{ and } 4.1) \times 10^{-4} \text{ min}^{-1}$ respectively.

The observed rate constants (k) for the photocatalytic degradation of sample using different photocatalysts of Znc, Zc, AZ, NZ and ANZ are $1.36 \times 10^{-3}, 4.44 \times 10^{-3}, 8.2 \times 10^{-3}, 1.14 \times 10^{-2}$ and $2.23 \times 10^{-2} \text{ min}^{-1}$ under solar irradiations at 180 minutes respectively. The rate constants for the photocatalytic degradation of sample solution using photocatalysts of Znc, Zc, AZ, NZ and ANZ are $9.47 \times 10^{-4}, 3.33 \times 10^{-3}, 5.07 \times 10^{-3}, 4.2 \times 10^{-3}$ and $6.35 \times 10^{-3} \text{ min}^{-1}$ under UV irradiations at 180 minutes respectively. All the graphs are straight line with slope of $k/2.303$.

TABLE- 2. Values of photocatalytic degradation rate constant (k) of sample using different photocatalysts under solar irradiations ($k \times 10^{-4} \text{ min}^{-1}$)

Time (min)	k_z	k_{Znc}	k_{Zc}	k_{AZ}	k_{NZ}	k_{ANZ}
0	0	0	0	0	0	0
20	10.5	4.59	32.9	52.7	61.9	80.3
40	17.9	6.92	28.3	50.3	64.5	99.7
60	21.3	9.59	42.0	53.6	68.0	97.9
80	19.6	10.0	36.8	58.0	73.8	97.6
100	21.0	12.8	35.0	54.0	85.1	111.8
120	23.4	15.4	33.1	55.9	93.9	116.4
140	24.4	15.9	40.2	62.4	93.0	144.1
160	23.2	14.9	37.9	69.2	107.9	211.0
180	22.0	13.7	44.5	82.6	114.3	223.2

TABLE- 3. Values of photocatalytic degradation rate constant (k) of sample using different photocatalysts under uvirradiations ($k \times 10^{-4} \text{ min}^{-1}$)

Time (min)	k_z	k_{Znc}	k_{Zc}	k_{AZ}	k_{NZ}	k_{ANZ}
0	0	0	0	0	0	0
20	4.61	6.91	16.0	48.4	29.9	84.0
40	5.76	13.8	21.9	33.4	31.7	56.0
60	6.91	13.1	29.2	40.3	33.4	56.0
80	7.77	11.8	26.5	37.7	34.5	57.0
100	9.20	11.5	27.9	47.0	34.5	54.0
120	10.6	12.1	29.4	46.0	36.5	58.0
140	10.9	15.0	34.4	49.8	40.0	62.0
160	10.1	15.5	33.0	50.5	43.0	59.0
180	9.47	14.5	33.3	50.7	42.0	63.5

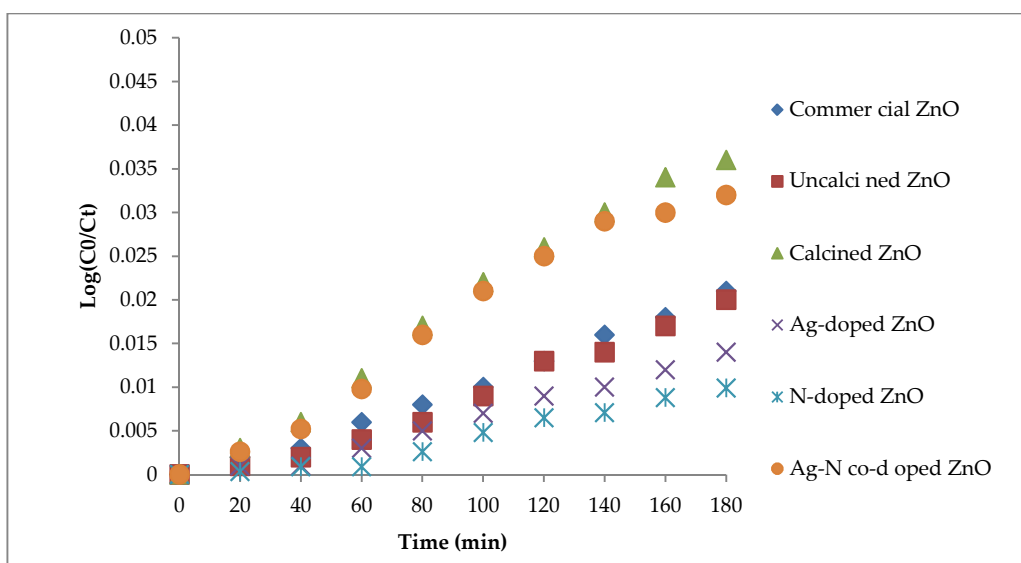


Figure (6). Log (c_0/c_t) vs. Time curves of photocatalytic adsorption of methyl orange (MO) using zinc oxide & modified zinc oxide photocatalysts (without irradiation)

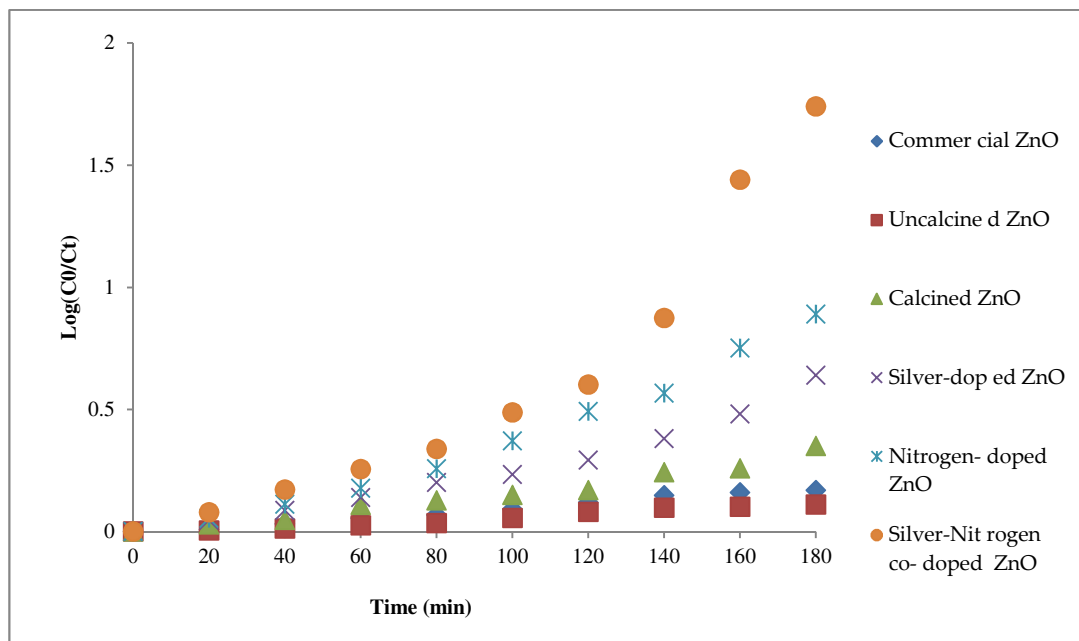


Figure (7). Log (c_0/c_t) vs. Time curves of photocatalytic degradation of methyl orange (MO) at zinc oxide and modified zinc oxide photocatalysts (under solar irradiation)

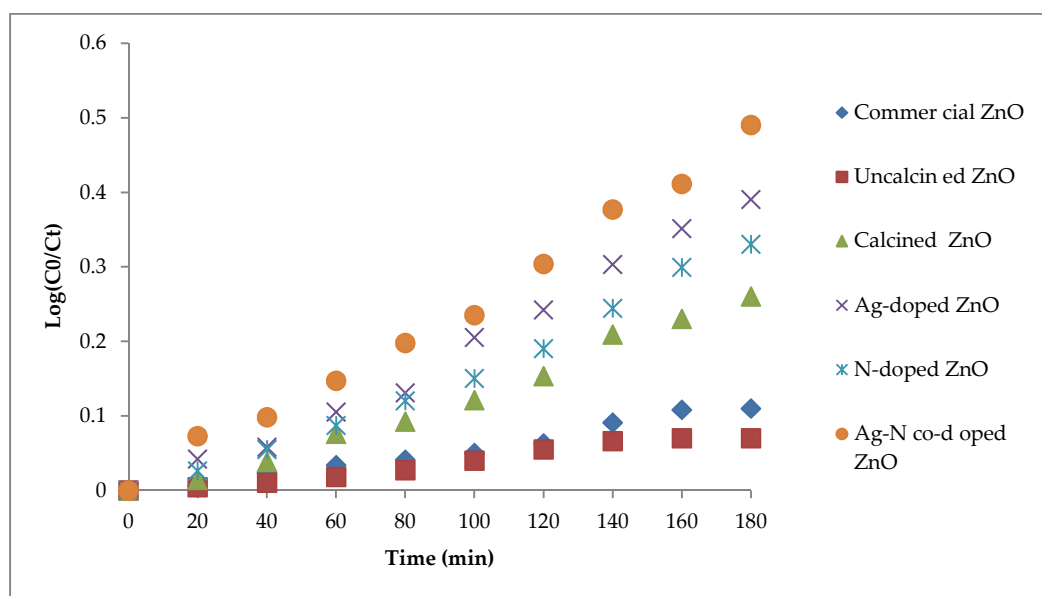


Figure (8). Log (c_0/c_t) vs. Time curves of photocatalytic degradation of methyl orange (mo) using zinc oxide & modified zinc oxide photocatalysts (under uv irradiation)

4. CONCLUSIONS

In present study, modified zinc oxide nanoparticle photocatalysts by co-doping silver and nitrogen have been synthesized with an ultimate goal of improving the photocatalytic efficiency of zinc oxide for waste sample photo-degradation under solar and UV irradiations.

Doping of nitrogen into zinc oxide modifies the electronic properties of zinc oxide leading to the optical absorption of zinc oxide well extended to the visible light region. This leads to harvesting of more photons of solar irradiations; thus, improving the photocatalytic activity of the semiconductor. Silver-doping of zinc oxide showed a positive influence on its photocatalytic degradation of waste sample by facilitating electron-hole pair separation due to its trapping ability of photo-generated electron. As-synthesized silver and nitrogen co-doped zinc oxide showed higher photoactivity than the silver-doped or nitrogen-doped zinc oxide as well as calcined zinc oxide in both UV and solar irradiation. However, photocatalytic activity of nitrogen-doped zinc oxide is higher in solar irradiation compared to UV irradiation.

ACKNOWLEDGMENTS

First of all, we wish to express our gratitude to research & publication center, Aksum University for their precious guidance, encouragement, commitment and support throughout our entire research work. We appreciate the chance that gave us to work on the intriguing research field of nanomaterials and photocatalysis.

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