Color Removal of Azor A Dye in Aqueous Solution by ZnO and Hydrogen Peroxide Under Solar Irradiation

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Abstract. ZnO nanoparticles was synthesized by sol gel process and investigated using XRD, FT-IR, SEM and AFM analysis. Advanced oxidation processes (AOP) have showed to be very effective in degradation of most pollutants in wastewaters. The effect of initial concentration for Azure A dye was studied for five concentrations from (1×10⁻⁵ M to 5×10⁻⁵ M). The photocatalytic degradation of the dye was observed spectrophotometrically. Also, the study involved effect of initial dye concentration, hydrogen peroxide dosage, pH, temperature and photocatalyst dose under solar light. The study involved to find out the optimum operating conditions of the treatment processes. Generally it has been found that the rates of photodegradation of azure A dye was higher in presence of solar radiation and ZnO photocatalyst. Also, photocatalytic ZnO showed the importance with light lead to enhance the generation of hydroxyl radicals which drive the photo oxidation processes. The kinetic and the percentages of degradation were studied at different intervals for 60 min. The experimental results indicated that the maximum photodegradation 89% of Azor A dyes occurred with ZnO photocatalyst at acidic media. COD analysis of The photodegraded samples showed reduction in COD after 60 min.

1- Introduction
Textile industry is one of the most complicated industries among manufacturing industry[1]. There are numerous classes of organic pollutants such as organic dyes, pharmaceuticals, polychlorinated pesticides, polycyclic aromatic hydrocarbons, polychlorinated dibenzodioxins and biphenyls etc. that by the seriousness of the risks they pose to environment and human health are significant priorities for environmental controlling by the most important environmental agencies [2-3]. The conventional treatment methods are incorporated with the advanced oxidation processes (AOP) leads to the most dyes can be easily treated, which can destroy the structure of the dye and make it more a minable to bio-degradation [4-5]. Major pollutants in textile wastewaters are high acidity, heat and other soluble substances main pollution in textile wastewater came from dyeing and finishing processes [6-7]. Treatment of wastewater is one of the main problems looked by textile. The removal of color from dyestuff manufacturing industry wastewaters and textile industry implies a fundamental ecological concern. Sullied air, soil and water by wastes from the industries are related with substantial disease load [8-9]. The processes of photocatalysis is relatively simple. The Light energy beginning from ultraviolet radiation in the form of photons which below 390 nm can excites the electrons on the surface of titanium atoms suspended in the contaminated of water [10]. Advanced oxidation processes
(AOP) have showed to be very high effective in degradation of different of pollutants in wastewaters [11-12]. The effect of \( \text{H}_2\text{O}_2 \) with irradiation of dye solution in presence of \( \text{H}_2\text{O}_2 \) which lead to generate free hydroxyl radicals. The UV-Vis radiation can be used to break the (O-O) bond in hydrogen peroxide then produce the free hydroxyl radical can be used in the degradation of different organic pollutants in an aqueous solutions [13-15]. ZnO is a commonly studied n-type semiconductor, ZnO photocatalysts have limited applications in catalysis, gas sensor, solar cells and miniaturized sensor. ZnO have large exciton binding energy of 60 meV and direct band gap (3.37 eV) [16-17]. Textile wastewater has sizable pollution hundreds in terms of Chemical Oxygen Demand(COD), Biochemical Oxygen Demand (BOD), Total Dissolved Solids(TDS), Total Suspended Solids(TSS) and heavy metals. Concerning the COD, at commercial wastewater treatment centers as in tannery wastewater treatment and organic concentration are typically reduced both by means of expensive tertiary actions or by adding activated powder carbon through biological treatment [18-19].

The present study was carried out to investigate synthesis of ZnO nanoparticles using sol-gel process. Also, the removal efficiency of color of azure A dye using various advanced oxidation processes in presence solar radiation to achieve complete degradation of azor A dye.

2- Experimental

2-1- Chemicals

In this study, all chemicals were used without further purification. Hydrogen peroxide (\( \text{H}_2\text{O}_2 \) 30% w/v), zinc acetate dehydrate \( \text{Zn(O}_2\text{CCH}_3)_2 \), 99.9% and potassium hydroxide (KOH) from Sigma-Aldrich, hydrochloric acid (HCl), sodium hydroxide (NaOH) were provided from BDH. Azure A dye (product of USA, MSDS) was obtained from Omega, the structure formula was shown in Fig.1. All the other chemicals were prepared with double distilled water.

![Figure 1: Structural formula of Azure A dye](image)

2-2- Instruments

The crystalline character has been recognized by X-ray diffraction (XRD) analysis using a D/Max 2,550 V diffractometer with Cu Kα radiation \( (\lambda = 1.54056 \text{ Å}) \) (Japan) for 20 in a range from 10° to 80°. Scanning electron microscope (SEM) analysis was achieved using Tescan, Vega 3 (Czech) electron microscope with accelerating voltage of 25 kV. The roughness of the surface was noted by angstrom AFM (SPM-AA3000, USA). The functional groups was determined using FTIR (Shimadzu FTIR 8400s, Japan). Centrifugation of ZnO suspensions was performed by using CORP Triup International Italy instrument. Thermoreactor TR 300 COD Thermoreactor. UV-Visible 1650 spectrophotometer (Shimadzu Japan) was used to recording the absorption spectra of dye solutions at (632 nm). UV-Visible 7804C spectrophotometer (Sunny, China) was used to measure of absorbance of dye solutions. The pH was measured by using microprocessor pH meter 211, (Hanna, Romania) instruments.
2-3- Synthesis of ZnO Nanoparticles
ZnO nanoparticles was synthesized by sol gel process, firstly, 2.19 g of zinc acetate dehydrate \(\text{Zn(O}_2\text{CCH}_3)_2\) was dissolving in 100 mL distilled (water/ethanol) then stirred under ambient atmosphere. Solution of KOH prepared by dissolved 1.12 g of KOH in 10 mL distilled water then added to the above solution drop wise with continuous stirring. After several minutes, a milky white solution was gotten. Then the mixture was heated at 60 °C without stirring for 2 h. The resulting suspension was centrifuged, then the mixture was washed with distilled water several times in an ultrasonic bathwater and then the powder was dried at 70 °C overnight [20].

2-4- Irradiation procedures
All dye solutions were irradiated for a period of 60 min (the primary experiments investigated that the most of azor A dye molecules are degraded or convert colorless at the time near to this period). Within this time samples were taken by syringe for measuring the dye absorption at \(\lambda_{\text{max}} = 632\) nm and then determination of dye concentration by using the calibration curve shown in Fig.(2). Fig.(3) shows the UV-Visible absorption spectrum of dye. The effect of \(\text{H}_2\text{O}_2\), ZnO, pH and temperature were studied. Control experiments were approved under UV irradiation with \(\text{H}_2\text{O}_2\) in the solutions. ZnO amount in a range 50-150 ppm were added to aqueous dye solution (concentration of all dyes was \(1\times10^{-5}\) M). The pH was adjusted to the desired value by 0.1 N of NaOH or HCl acid [21]. Samples of azure A dye was treated with Thermo Low-range (0-150) COD test reagent in COD Thermoreactor. In this research 3 mL of dye solution taking then added to 3 mL of the solution of COD. Carried out study the effect of concentration dyes in range \(1\times10^{-5}\) to \(5\times10^{-5}\) M, pH and \(\text{H}_2\text{O}_2\) dosage on the degradation of Azor A dye.

\[y = 0.3378x + 0.0268\]
\[R^2 = 0.9971\]

![](image1.png)

Figure 2: Calibration curve for Azure A dye at pH=6 and T=298 K

![](image2.png)

Figure 3: UV-Visible spectrum of aqueous solution of Azure A dye (\(1\times10^{-5}\) M), pH=6 and T=298 K

3- Results and Discussion
3-1- analysis
3-1-1XRD analysis
The crystalline character has been identified by X-ray diffraction (XRD) analysis which recorded in the range of 20-80°. Fig.4 show the XRD pattern for ZnO nanoparticles, the peaks appeared at 20 = 31.65°, 36.35°, 34.52°, 47.53°, 62.33°, 66.37°, 67.96°, and 69.09°, which correspond to (100), (101),
(002), (102), (103), (110), (112) and (201), reflection planes. Also observed the highest intensity peak (101) and the average size distribution recorded in the range of 46 nm by Debye Sherrer’s formula. All the diffraction peaks can also be well indicate to the hexagonal wurtzite structure of ZnO (JCPDS no: 36-1451)[22].

![Figure 4: XRD pattern of ZnO nanoparticles](image)

### 3-1-2 FT-IR analysis

In Fig. 5, FTIR spectra of ZnO shown that the peak was observed at 467 cm\(^{-1}\) indicates the presence of Zn-O stretching bond [23]. The peak appear at 3427 cm\(^{-1}\) can be attributed to the characteristic absorption of hydroxyl group. In spectra the broad absorption peak observed at 2350 cm\(^{-1}\) which indicates to presence of H\(_2\)O and CO\(_2\) peak that adsorbed on the surface of material.

![Figure 5: FT-IR spectrum of ZnO Nanoparticles](image)

### 3-1-3- SEM analysis

The surface morphology of ZnO nanoparticles has been studied in terms of the size, shape of nanoparticles and clusters among them, in addition to the distribution of these nanoparticles using the SEM analysis. Fig. 6 shows SEM image of ZnO, the nanoparticles have a hexagonal wurtzite shape with a mean size of between 45-63 nm with a lower of agglomerate.

![Figure 6: SEM images of ZnO Nanoparticles](image)
3-1-4- AFM analysis
Atomic force microscopy (AFM) analysis is usually used for the determination of the size and roughness of ZnO nanoparticles[24]. Fig.7 shows the 3D and 2D images of ZnO nanoparticles using tapping mode over a 2×2 μm to investigate the surface morphology and roughness of ZnO nanoparticles. The AFM images show the distribution of ZnO nanoparticles is high uniform and homogenous with a slight agglomerate of zinc oxide on the surface. The parameters such as the concentration of metal ion, temperature, pH and suggestively affected on the size and shape of the nanoparticles.

![Figure 7: 3D and 2D image of AFM for ZnO Nanoparticles](image)

3-2- Photocatalytic Study
3-2-1- Effect of Dye Concentration
The photodegradation of azure A was followed by measuring the absorbance of treatment samples at dye λmax, the results are shown in Fig. 8. A different initial dye concentration were used in the investigation and observed the effect on color removal of azure A dye by using 75 ppm of ZnO photocatalyst in presence solar radiation. The color removal efficiency from azure A was decreased from 43% to 13% with increasing the concentration of dye from 1×10⁻⁵ M to 5×10⁻⁵ M in presence of solar irradiation. The results clear in figures, extremely concentrations of increased the UV absorption radiation, subsequently lowering the UV light availability for the reaction, so decreasing the formulation free radicals of hydroxyl in the solution [21], due to reducing penetration of photons which entering through the solution and lowering the creation of hydroxyl free radicals[25]. The degradation rate was first-order with respect to dye concentration. Fig. 8(c) refer the relationship between Log C and Log R to produce the order reaction.

![Graphs](image)
3-2-2- Effect of ZnO Mass

The effect of ZnO nanoparticles concentration was investigated on the photodegradation of azure A dye. Accordingly the color removal rate was increased significantly by increasing the amount of ZnO. To optimize the concentration of ZnO nanoparticles needed for the highest degradation rate the following quantities were used starting from 50-150 ppm depending on the nature of the compounds [26]. The increasing in the amount of ZnO photocatalyst increases the number of active sites of the ZnO surface that leading to increase the number of O$_2$ and OH free radicals [27]. Several authors [28-29] related this observation when the solution induced by light proof suspended catalyst, the light scattering and consequent reduction in light penetration. This increase in the rate of bleaching may be attributed to increase in the exposed surface area of the semiconductor. The results indicated that the maximum photodegradation 89% using 100 ppm of ZnO photocatalyst was achieved in presence of solar irradiation, the electrons in the semiconductor are excited from the valence band to the conduction band leaving positive holes in the valence band. Fig.9 illustrated that the photodegradation of dye increases as increasing of catalyst concentrations.

3-2-3- Effect of Initial Concentration of Hydrogen Peroxide

A number of experiments has been carried out including the effect of adding H$_2$O$_2$ in the range 100-500 mg/L on azure A dye photo degraded. The results is shown in Fig.10. Hydrogen peroxide low concentrations cannot produce enough hydroxyl radicals and the oxidation rate is logically very slow but higher hydrogen peroxide concentration was produced more OH leading to a faster oxidation rate [30]. Photolysis of H$_2$O$_2$ by (H$_2$O$_2$/solar) generates hydroxyl free radical according to following[31]:

$$\text{H}_2\text{O}_2 + \text{hv} \rightarrow 2 \cdot \text{OH}$$

……..(1)
\[ \text{OH} + \text{H}_2\text{O}_2 \rightarrow \text{H}_2\text{O} + \text{HO}_2 \]  \hspace{1cm} \text{......(2)}

\[ \text{HO}_2 + \text{H}_2\text{O} \rightarrow \text{H}_2\text{O}_2 + \text{O} + \text{OH} \] \hspace{1cm} \text{......(3)}

\[ 2\text{HO}_2 \rightarrow \text{H}_2\text{O}_2 + \text{O}_2 \] \hspace{1cm} \text{......(4)}

Also, the effect of \( \text{H}_2\text{O}_2 \) concentration on COD removal was investigated, through the results observed that with increase of the hydrogen peroxide concentration the value of COD removal increase. The results are clarified in Fig.9.

![Graph 1](image1.png)

**Figure 10:** Effect of different initial \( \text{H}_2\text{O}_2 \) concentration on color removal efficiency and COD of Azure A dye=\( 1\times10^{-5} \)M, pH=6 at \( T=298\text{K} \) by using solar/\( \text{H}_2\text{O}_2 \) method

3-2-4 Effect of initial pH

The higher ratio color removal of azure A dye was obtained under acidic media at pH=4 under solar light and decreasing ratio in basic media. The experiments are conducted at \( 1\times10^{-5} \) M dye concentration in presence of ZnO photocatalyst and different pH media were investigated 2,3,4,5,7,9,10 and 12 for 60 min at \( T=298\text{K} \). The results deduced the high color removal under acidic medium due to more OH radicals generation and changes in the structure of the molecule because of having a free hydrogen atom which makes the dye molecule exposed for attack by the hydroxyl radical in acidic conditions[32]. The decomposition in alkaline medium formed oxygen and water relatively than producing hydroxyl free radicals in presence UV-Visible irradiation[33]. The effect of different pH media on the COD and color removal efficiency of azure A dye was studied as show in Fig.11.

![Graph 2](image2.png)

**Figure 11:** Effect of different pH value on COD and color removal efficiency of Azure A dye =\( 1\times10^{-5} \) M as function of irradiation time using solar/ZnO=100 ppm method at \( T=298\text{K} \)

3-2-4 Effect of Temperature

Temperature has obvious effect on color removal efficiency. At high temperature increases the generation rate of ’OH radicals or directly effects on the reaction rate with the dye molecules [34].
The experiments were carried out in a range between 298 to 318K in presence of ZnO photocatalyst, the results are clarified in the Fig.12. The Arrhenius equation in eq. 5 may be used to refer to the relationship between temperature and rate constants, this the relationship as show in Fig.12.

\[ k = A e^{(-Ea/RT)} \]  

Where: \( k \) : Rate constant, \( Ea \) : Activation energy, \( A \) : Frequency factor, \( T \) : The absolute temperature and \( R \) : Ideal gas constant [35].

The activation energy calculated from the diagram was equal to 42.23 kJ/mole for azure A dye in the presence of solar irradiation.

**Figure 12:** (a) and (b) Show the effect of the temperature degree on color removal efficiency of the Azure A conc. =1X10^{-5} M, ZnO=100 ppm (c): Arrhenius plot of color removal of Azure A conc. = 1x10^{-5} M and pH=6

4- Conclusions

The photooxidation of azure A dye by using the ZnO nanoparticles is more effective than using initial dosage of H_{2}O_{2}. The size of prepared ZnO nanoparticles were recorded using SEM analysis to be in the range of 45-63 nm. Different techniques such as XRD, FTIR, SEM, AFM techniques were used for the identification of prepared ZnO nanoparticles. The photodegradation was strongly influenced by numerous parameters, mainly the initial H_{2}O_{2} dosage, dye concentration, pH and temperature as well as irradiation time and photocatalyst used ZnO nanoparticles. The rate of photodegradation was recorded higher efficiency at temperature 308K.
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