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UV-A Activated ZnO Mediated Photocatalytic Decolorization of Nigrosine (Acid Black 2) Dye in Aqueous Solution

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Abstract

The present study deals with studied the essential requirements for photo-decoulorization of nigrosine dye with suspension solution of photocatalyst (ZnO), under 250 Watt UV-A light (I_o = 1.47 × 10^{-7} ensien s^{-1}). A kinetics study of photo-decolourization for this dye was obeyed to pseudo-first order. The best initial pH of decolorization at 25 mg/L of dye solution with 300 mg of ZnO was given a fast reaction at 8.17. The calculated activation energy for this photoreaction was found to be 31.549 kJ·mol^{-1}. Thermodynamically, the reaction is exothermic and spontaneously. The efficiency of decolorizatio E% was 97.077 at 15 min that decreased with addition oxidant reagents such as H_2O_2, Fe^{2+} and mixture from them.

Keywords

Nigreson Dye, Acid Black 2 Dye, Photocatalytic, Decolorization, Oxidant Reagents

1. Introduction

There are more than 100,000 varieties of dyes available in the market, which used in the textile, cosmetics, food, paper, leather industries and Medications industries. Whereas over 700,000 tons per were produced in worldwide. About 10% of the above amount of used dye is lost as wastewater, which affected on the environment by leading to Poisoning of living organisms [1] [2] [3].

During the Industrial Revolution, the synthetic dyes were quickly replaced with the commercial textiles manufactured and unlike the natural dyes, because of these dyes were suitable for synthetic fibers [4].

Nigrosin dye is a mixture of artificial black dyes (CI 50415, Solvent black 5)

made by heating a mixture of aniline, nitrobenzene and aniline hydrochloride in found the existence of catalyst like copper or iron. The advantage of sulfonation of nigrosine dye is yielded it ability for solubling in water as anionic dye. The main industrial employed for nigrosin dye is done as a marker-pen inks and colorant for varnishes. In biology field, the nigrosin dye is employed for negative Staining bacteria, as well as the capsule-containing fungus and *Cryptococcus neoformans* [5] [6].

Heterogeneous photocatalysis has performed an adevastating mission technology that caused to the total mineralization of most of the organic pollutants like organic dyes. Titania (TiO_2) is the most commonly employed effective photocatalyst for a wide range of organic chemical degradation. In recent years, ZnO is another important semiconductor investigated. In some cases, ZnO, has been more effective than TiO_2. ZnO is regarded as a representative of the metal oxide class, which is played an important role to the study of electrochemistry and catalysis. The ZnO nanoparticle has received much attention because it has a low cost of production, responded to UV light to have a band gap range 3.2 - 3.3 eV, and high photoactivity in several photochemical and photoelectron-chemical processes. All these characteristic of it that lead to use it in eliminating environmental pollution [7] [8] [9] [10].

The aim of this work was focused on the studied of the effect of initial pH , temperatures and oxidation reagent on photodecolorization of Nigresoine dye in suspension solution of ZnO.

2. Materials and Method

All used chemicals in this work were employed without further purification. 99.5% purity of commercial ZnO was supplied by Fluka. Nigrosin dye has molecular weight equal to 616.49 $g \cdot mol^{-1}$, IUPAC name is mono (4,4'-((5-phenyl-9-(phenylamino) phenazine-5-ium-2,3 diyl) bis(azanediyl)) dibenzenesulfonate), synonym is Acid Black 2, molecular formula ($C_{22}H_{14}N_6Na_2O_9S_2$), the chemical family beyonds to Aniline derivatives, classification as acid dye and supplied by High Purity Laboratory Chemicals (HPLC), Mumbai. The chemical structure for Nigrosin dye is illustrated in (**Figure 1**).

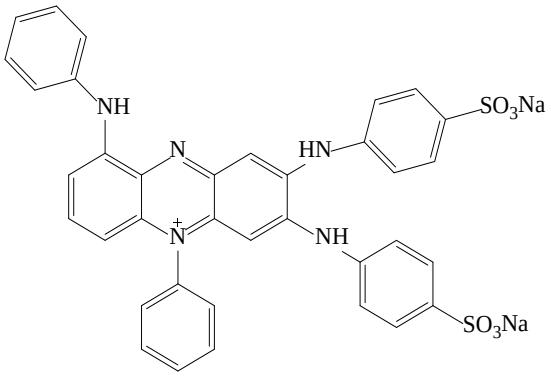


Figure 1. The chemical structure for Nigrosin dye.

The photocatalytic decolorization by using ZnO experiments were done by employing home made photoreactor that consists of 250 Watt high pressure mercury lamp at light intensity I_o equal to 1.47×10^{-7} ensien s^{-1} that calculated by chemical actinometer (ferrioxalate actinometric) [11], magnetic stirrer, wooden box that used to prevent the escape of harmful radiation [12], fan, Pyrex glass beaker and Teflon bar.

25 ppm of Nigrosin dye was mixed with 300 mg of ZnO to generate a suspension solution by employing a magnetic stirrer. The mixing process was continued without irradiation in dark reaction for 30 min, and then the mixture was activated via the light irradiation in photocatalytic process. About 3 mL of suspention solution was collected at 5 min as a sequence time by using a plastic test tube. The withdrawn suspention was centrifuged at 4000 rpm for 10 minutes and filtered. The centrifuged process was repeated to ensure the completed of removing the catalyst before found the residual concentration of dye . To find the residual concentration of Nigrosin dye was measured using spectrophotometry at 570 nm.

The rate constant for this reaction was determined by depending upon the Langmuir-Hinshelwood (L-H) model that modified to investigate the order of the studied photoreaction, from the following equations [13] [14] [15]:

$$C_t = C_o \exp^{\left(-k_{app} \cdot t\right)} \tag{1}$$

where: C_o is an initial concentration of Nigrosin dye in (dark reaction) at time of irradiation = 0, C_t is a concentration of the same dye at time t of irradiation.

$$\ln \frac{C_o}{C_t} = k_{app} \cdot t \tag{2}$$

Equation (2) was applied, at assumption the initial concentration of dye is low, hence, the rate of photo reaction follows pseudo first-order kinetics [14]. The effeciency of decolorization E% was assessed [14] [16] in Equation (3).

$$E\% = \frac{C_o - C_t}{C_o} \times 100 \tag{3}$$

3. Results and Discussion

3.1. Effect of pH

The initial pH is regarded as a vital parameter to study the photoreaction for decolorization any dye from suspension solution of photocatalyst. The effect of pH from 4 to 10 on the photocatalytic decolorization of Nigrosin dye are appeared in (**Figure 2(a)** and **Figure 2(b)**) and **Figure 3**.

The results demonstrated that the best initial pH for decolorization of Nigrosin dye is 8.177. This value is near from the zero point charge of ZnO that equal to 9 [12] [17]. This effect depended upon the characteristics of studied dye, the surface charge of catalyst and generation of hydroxyl radicals via photocatalysis process [18] [19]. In acid medium, the surface of photocatalyst is positive chargly and caused photocorrosion via self oxidation, therebye the efficiency of

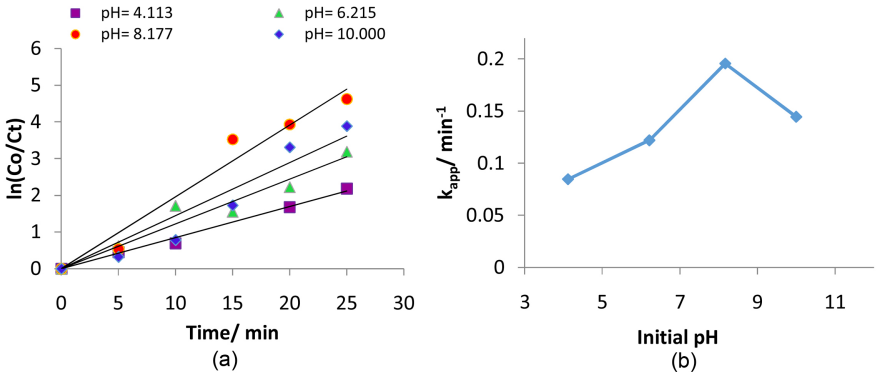


Figure 2. Effect of initial pH on the apparent rate constant of Nigrosin dye decolourization, dye concentration 25 ppm with ZnO dosage 300 mg/100mL, at initial pH range (4.113 - 10.000), Temp. 288.15 K and I_o of UV-A is 1.47×10^{-7} Ens s^{-1}). (a) $\ln(C_o/C_t)$ vs time and (b) k_{app} vs initial pH of solution.

decolorization depresses. Moreover, In basic medium, the surface of photocatalyst is negative charge, that will lead to dissolution of the photocatalyst according to the following Equations (4)-(7) [19] [20].

$$ZnO + 2h^+ \rightarrow Zn^{2+} + \frac{1}{2}O_2 \qquad (\text{acidic pH}) \tag{4}$$

$$\text{or} \quad ZnO + 2H^+ \rightarrow Zn^{2+} + H_2O \qquad (\text{acidic pH}) \tag{5}$$

$$Zn-OH + OH^- \rightarrow Zn-O^- + H_2O \qquad (\text{basic pH}) \tag{6}$$

$$\text{or} \quad ZnO + H_2O + 2OH^- \rightarrow \left[Zn(OH)_4\right]^{2-} \qquad (\text{basic pH}) \tag{7}$$

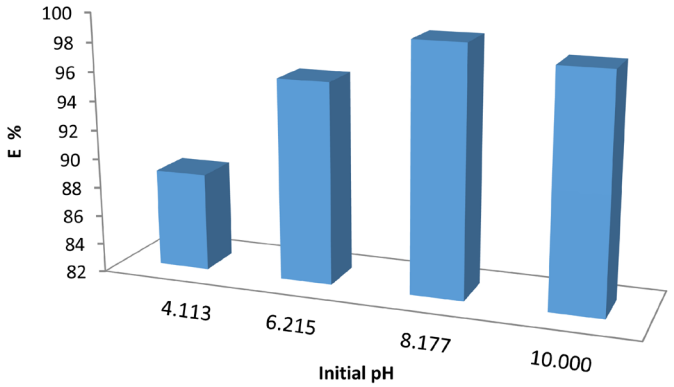


Figure 3. Effect of initial pH of Nigrosin dye solution on E% for decolourization of 25 ppm from this dye with 300 mg/100mL of ZnO, at initial pH range (4.113 - 10.000), Temp. 288.15 K and I_o of UV-A is 1.47×10^{-7} Ens s^{-1}).

Figure 3 was proved, at initial pH equal to 8.170, the maximum value of the efficiency (E%) of removal Nigrosin dye in presence of ZnO is reached to 99.014 at 25 min and 288.15 K.

3.2. Effect of Temperature

The effect of temperature on photodecolorization of Nigresone dye from sus-

pension solution of ZnO was estimated in the range (283.15 - 303.15) K. This effect plays an important role to determine the apparent activation energy (E_a) that used Arrhenius equation (Equation (8)), and some thermodynamics parameters such as ($\Delta H^{\#}$, $\Delta S^{\#}$ and $\Delta G^{\#}$) that employed Eyring-Polanyi equation (Equation (9)) and Gibbs equation (Equation (10)). These parameters were calculated by the following Equations [21] [22] [23] [24].

$$\ln k_{app} = \frac{-E_a}{RT} + \ln A \tag{8}$$

Whereas: k_{app} is apparent rate constant, E_a is apparent activation energy, A is a frequency constant, R is gas constant and T is temperature of photoreaction.

$$\ln\left(\frac{k_{app}}{T}\right) = \frac{-\Delta H^{\#}}{RT} + \left(\ln\frac{k_B}{h} + \frac{\Delta S^{\#}}{R}\right) \tag{9}$$

Whereas: k_B is a Boltzmann's constant, h is a Plank's constant, R is a gas constant and T is the temperature of reaction.

$$\Delta G^{\#} = \Delta H^{\#} - T\Delta S^{\#} \tag{10}$$

From (**Figure 4(a)** and **Figure 4(b)**), it was observed the decolorization of the studied dye declines with increasing temperature that due to the exothermic nature in reaction ($\Delta H^{\#}$). This result is in agreement with information that reported in references [21] [25].

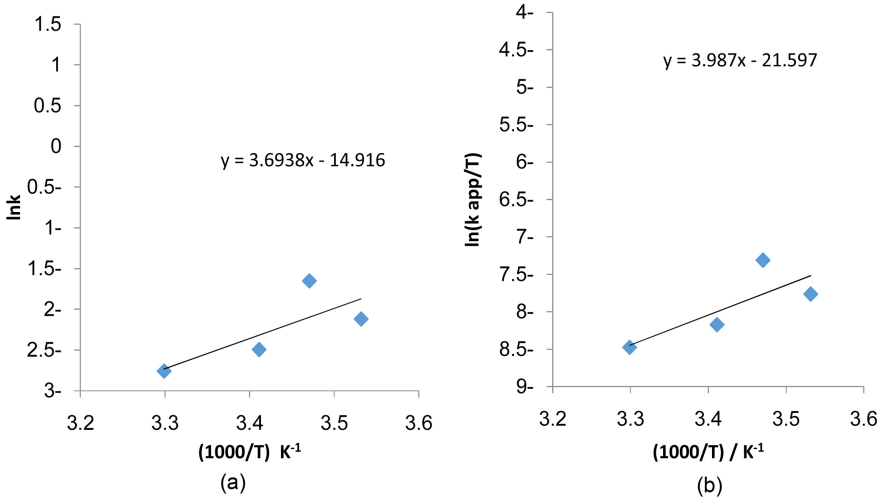


Figure 4. Effect of temperature on photodecolorization of 25 ppm Nigresone dye, with 300 mg/100mL ZnO, at normal initial pH of solution (8.177), range in temperature (283.15 - 303.15) K and I_o of UV-A equal to 1.47×10^{-7} Ens s^{-1}). (a) Arrhenius equation plot of (ln*kapp*) vs. 1/*T*. and (b) Eyring plot of (ln(*kapp*/*T*)) vs.1/*T*.

On basis on calculated data from **Figure 4(a)** and **Figure 4(b)**, the values of activation energy and the essential thermodynamics parameters that are listed in **Table 1**.

The negative value of $\Delta G^{\#}$ for this photoreaction indicates the reaction spontaneous in nature. The negative value of $\Delta S^{\#}$ proved the depress randomness.

Table 1. The activation kinetic and thermodynamic parameters that calculated for photodecolorisation of Nigresine dye with suspension solution of ZnO.

E_a kJ·mol^{-1}	$\Delta H^{\#}$ kJ·mol^{-1}	$\Delta S^{\#}$ kJ·mol^{-1}·K^{-1}	$\Delta G^{\#}_{303.15}$ kJ·mol^{-1}
30.71025	−33.1479	−0.00546	−31.4927

The moderate value of activation energy refer to the rate for decolorization of this dye is low and reach to final at 25 min.

3.3. Effect of Addition Oxidant Reagents

Figure 5 displays the effect of addition of varies types of oxidant reagent such as H_2O_2 and Fe(II) on the decolorization of Nigrosein dye in suspension solutuion of ZnO.

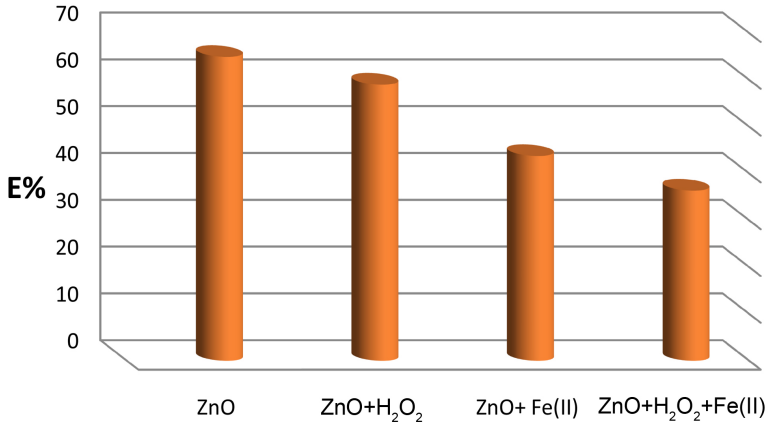


Figure 5. Effect of addition oxidant reagents on E % for decolourization of 25 ppm from this dye with 300 mg/100mL of ZnO, at initial pH 8.177, Temp. 303.15 K, 1% H_2O_2, 1×10^{-4} M Fe (II) and I_o of UV-A is 1.47×10^{-7} Ens s^{-1}).

In case of used ZnO alone as photocatalyst, The E% for decolorization of above-mentioned dye is equal to 64.9122 at 30˚C (Lab temperature) and 15 min. The hydroxyl radical acts as a power for starting the photoreaction with presence of photocatalyst. This progress of photoreaction was monitored by the following equations [26] [27].

$$\mathrm{ZnO} + h\nu \rightarrow \mathrm{ZnO}\left(\mathrm{e}^{-}_{\mathrm{CB}} + \mathrm{h}^{+}_{\mathrm{VB}}\right) \quad (11)$$

$$\mathrm{H_2O} + \mathrm{h}^{+}_{\mathrm{VB}} \rightarrow \mathrm{H}^{+} + \mathrm{HO}^{\bullet} \quad (12)$$

$$\mathrm{O_2} + \mathrm{e}^{-}_{\mathrm{CB}} \rightarrow \mathrm{O}^{\bullet -}_{2} \quad (13)$$

$$\mathrm{O}^{\bullet -}_{2} + \mathrm{H}^{+} \rightarrow \mathrm{HO}^{\bullet}_{2} \quad (14)$$

$$\mathrm{HO}^{\bullet}_{2} + \mathrm{H}^{+} \rightarrow \mathrm{H_2O_2} \quad (15)$$

$$\mathrm{H_2O_2} + h\nu \rightarrow 2\mathrm{HO}^{\bullet} \quad (16)$$

The decolorization of Nigresine dye will suggest in the following equation

$$\mathrm{Dye} + \mathrm{HO}^{\bullet} \rightarrow n\mathrm{CO_2} + n\mathrm{H_2O} + \mathrm{Inorganic\ ions}\left(\mathrm{NH}^{+}_{4}, \mathrm{SO}^{2-}_{4} + \mathrm{Na}^{+}\right) \quad (17)$$

When addition 1% of H_2O_2 to suspension solution of dye and ZnO, that depress the E% to 58.969, that attributable to the high concentration of H_2O_2 that acts as scavenger for •OH radical to form Perhydroxyl radical $HO_2^{\bullet}$ [12] [26].

$$H_2O_2 + HO^{\bullet} \rightarrow H_2O + HO_2^{\bullet} \quad (18)$$

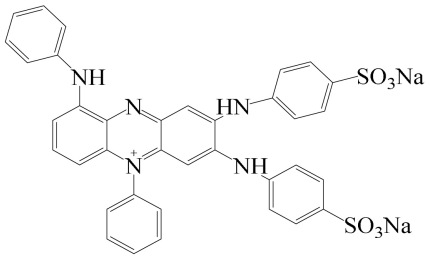
$$HO_2^{\bullet} + HO^{\bullet} \rightarrow H_2O + O_2 \quad (19)$$

In this work, the process for addition of Fe(II) in concentration 1×10^{-4} M to the dye solution with presence ZnO has a negative effect. So, *E*% of this reaction declines to be 43.750. That belongs on the reacted of Fe^{2+} with Perhydroxyl radical, according to the following equation [28] [29].

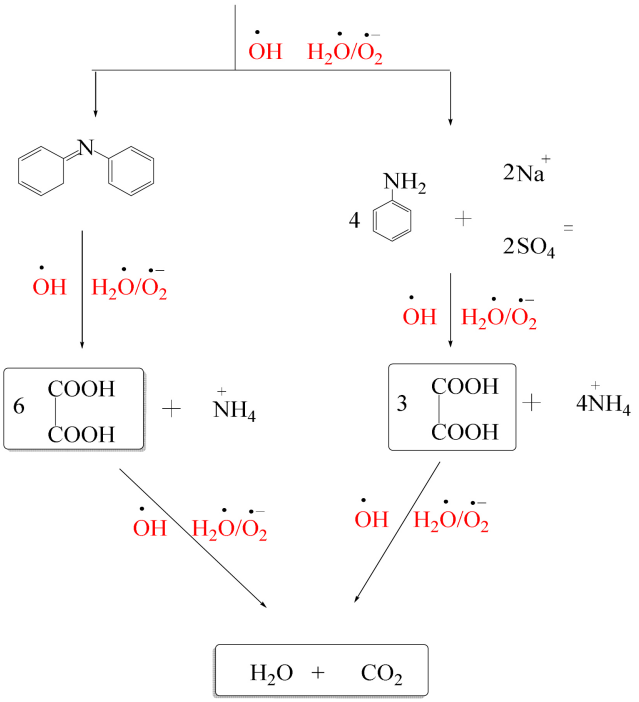
$$HO_2^{\bullet} + Fe^{2+} \rightarrow HO_2^{-} + Fe^{3+} \quad (20)$$

$$HO_2^{\bullet} + Fe^{2+} \rightarrow O_2 + Fe^{3+} + H^{+} \quad (21)$$

From the other hand, the used of fenton's reaction in the conditions of the photoreaction, that leads to depress the *E*% to 36.363. The certain concentration of H_2O_2 and Fe^{2+} causes to produce H_2O and O_2 [30] [31].



disodium mono(4,4'-((5-phenyl-9-(phenylamino)phenazine-5-ium-2,3-diyl)bis(azanediyl))dibenzenesulfonate)



Scheme 1. Suggested mechanism for decolorization of Negrisone in suspension solution of ZnO.

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + HO^{\bullet} + HO^{-} \quad (22)$$

$$H_2O_2 + HO^{\bullet} \rightarrow H_2O + HO_2^{\bullet} \quad (23)$$

$$HO_2^{\bullet} + HO^{\bullet} \rightarrow H_2O + O_2 \quad (24)$$

3.4. Proposed Mechanism

The suggested mechanism for decolorization of Nigreson in presence ZnO under UV-A light was summarized in (Scheme 1).

3.5. Conclusions

The essential conclusions for this work were proven the following results:

1) The photoreaction of decolorization of Nigresoin dye in suspension solution of ZnO obeys the pseudo first order kinetics.

2) The Rate of photocatalytic decolorization of the above dye is followed the next sequences:

Rate of decolorization in presence ZnO alone > Rate of decolorization in presence ZnO with H_2O_2 > Rate of decolorization in presence ZnO with Fe(II) > Rate of decolorization in presence ZnO with H_2O_2 and Fe(II).

3) The decolorization reaction is fast, exothermic and spontaneously at initial pH 8.177 of dye solution.

4) At 25 min and 288.15 K, the efficiency (*E*%) of removal Nigrosin dye in presence of ZnO was reached to maximum value and to be 99.014.

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