Preparation of Azo dye and study of the photo activity of zinc oxide

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ABSTRACT

This work include two parts, the first part preparation of 3-[(4-Phenyl azo-phenylimino)-methyl]-phenol. In the second part Photocatalytic degradation of prepared azo dye has been studied using zinc oxide ,the suspended solution include different concentration of prepared azo dye with fixed value of 0.13gm/100cm3 zinc oxide was irradiated by mercury lamp 160 Watts inside a Pyrex photoreaction cell of 100 cm3 with 10 cm3/min of flow rate air babble and at room temperature 298 K. Several experiments are carried out in various conditions to create the best Photocatalytic degradation of prepared azo dye, the effect of concentration of prepared azo dye, the effect of mass loaded of zinc oxide, and the effect of light intensity. UV-Vis spectrophotometer has been used to investigate the products.

KEYWORDS: photo degradation, Azo dye, zinc oxide, Advance Oxidation process, catalyst.

1. INTRODUCTION

In the worldwide environmental pollution have been problem for the human and animals because increase uses the dye industry in all filed of life and this lead to pollute the surface water and groundwater (Cheng , 2016; Abouzlam, 2015; Suzuki, 2015). To solve this problem the scientist have been uses advanced oxide processes (AOP) method for wastewater

(Jeirani, 2015; Maria, 2016; Hu, 2016). Advanced oxide processes (AOP) method starting with produce hydroxyl radicals (˙OH), this reactive radical capable of mineralizing organic pollutants (Hazim, 2016; Hazim, 2015). When the catalyst (in this work zinc oxide) directly irradiated by UV lamp from external source, the electrons will promote from valance band to conduction band, producing electrons in conduction band undergo photo reduction will leave a positive hole in valance band inter photo oxidation processes.

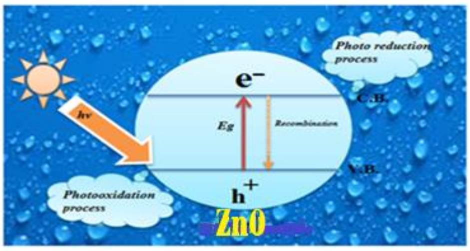

Figure.1.General mechanism of the photo catalysis on zinc oxide

The synthesis of 3-[(4-phenyl azo-phenylimino)-methyl-phenol classifide as the Imines, known even as azomethines or Schiff bases compounds. The schiff bases compounds that are represented by the general formula $R_3R_2C=NR_1$. The substituents R_2 and R_3 may be alkyl, aryl, heteroaryl, hydrogen.The substituent at the N-imino (C=N) may be alkyl, aryl, heteroaryl, hydrogen or metallo (usually Si,Al, B, Sn) (Ahmed HassenAlsryfy , 2015).

2. MATERIALS AND METHODS

A-Chemicals:

1-Zinc oxide: purity (99%), particle size (100) mesh, supplied by Fluka AG.
2- 4- aminoazobenzene, supplied by Fluka AG.
3-Ethanol, sigma – Aldrich.
4-m- hydroxyl benzaldehydesupplied by Fluka AG.

B-Preparation of 3-[(4-Phenylazo-phenylimino)-methyl]-phenol: A mixture of 4- aminoazobenzene (1.97 g, 10m.mole), 15 mL of Ethanol was stirred in the round bottom flask for 5-10 minutes at room temperature and Degases the reaction mass. When got on the clear solution, m- hydroxyl benzaldehyde (1.22 g, 10m.moles) was added to the mixture, and continued the stirring for 4 h. At the end of this period, the resulting mixture was filtered and washed with 2 mL of Ethanol, the filtrate was evaporated to obtain the residue.

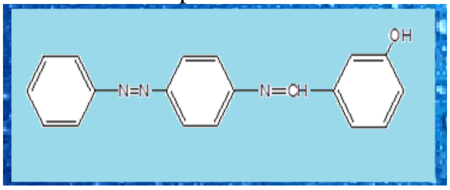

Figure.2. 3-[(4-Phenylazo-phenylimino)-methyl]-phenol

C - Photo reactor and Procedure: Experiments were carried out in glass photochemical reactor. The cylindrical annular – type reactor consisted of two parts. The first part was an outside thimble; running water was passed through the thimble to cool the reaction solution. Owing to the continues cooling, the temperature of the reaction solution was maintained of room temperature. The second part was an inside thimble and the reaction solution ($100 cm^3$) was put in the reaction chamber.

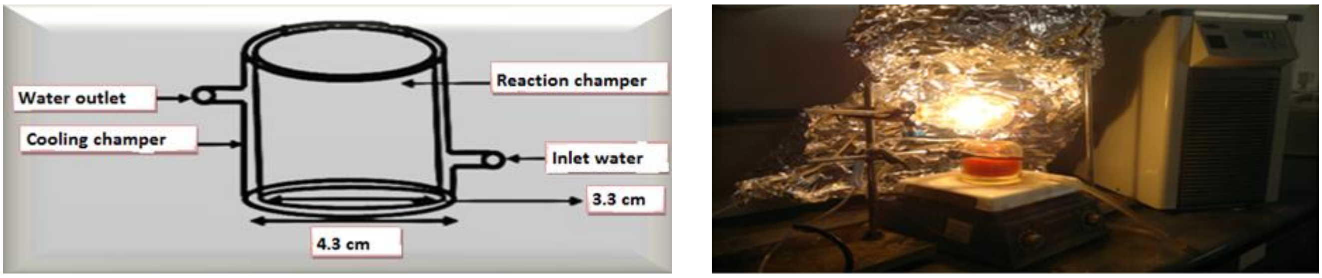

Figure.3. Main parts of the photocatalytic cell used in Photocatalytic degradation of prepared azo dye

3. RESULT AND DISCUSSION

The Effect of dosage mass zinc oxide on photo catalytic degradation of prepared azo dye: Different experiments has been perform for investigated The effect of mass of zinc oxide on Photocatalytic degradation of prepared azo dye, using 20 ppm of reactive blue, flow rate of air 10 cm^3/min, room temperature 298 K .

Table 1 and Figure 4 represent photo catalytic degradation processes of prepared azo dyeat different loaded mass ofzinc oxide rang (0.03 – 0.5gm/100cm^3). Photocatalytic degradation of prepared azo dye. When thezinc oxidemasses gradually increases photocatalytic degradation increases until reach to 0.13gm/100cm^3 which represent optimum value of zinc oxide, then gradually decreases.semiconductorzinc oxide can be provide the highest absorption of light at 0.13gm/100cm^3. The decrease in the efficiency of photo catalytic degradation process at the masses ofzinc oxide higher than 0.13gm/100cm^3due to the light absorption will be limited only to the first layers of prepared azo dyeand the other layers of solution do not receive light photons. Moreover light scattering at high zinc oxide loading , this lead to decrease the photon intensity, so the strong absorption of light through the first successive layers of solution and prevent light from passing through all other layers in the reaction vessel (Hazim, 2015; Chen, 1995).

At the loading mass of zinc oxide below the optimum value 0.13gm/100$cm^3$the rate of photocatalytic degradation of prepared azo dyealso decrease due to the quantity of mass of zinc oxide decrease that mean the surface area decrease which lead to decrease of light absorption of light by zinc oxide which cause lower photocatalytic degradation rate of prepared azo dye.

Table.1.The change of A_t /A_0 with irradiation time using different masses of zinc oxide

Catalyst Mass gm /100 cm^3	**0.03**	**0.06**	**0.12**	**0.3**	**0.5**
Irradiation Time/min	A / A_0				
0	1	1	1	1	1
10	0.67	0.76	0.52	0.82	0.91
20	0.37	0.48	0.25	0.58	0.75
30	0.20	0.28	0.13	0.38	0.52
40	0.10	0.20	0.05	0.26	0.36
50	0.09	0.15	0.05	0.21	0.31
60	0.08	0.13	0.04	0.18	0.29

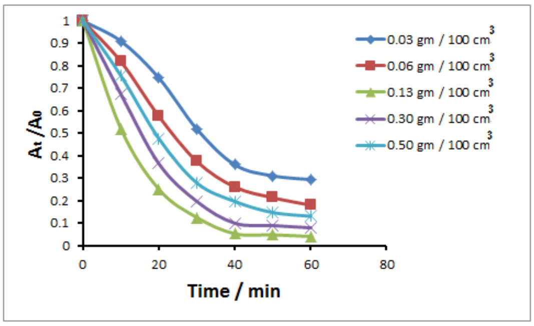

Figure.4.The effect masses of zinc oxide on Photcatalytic degradation of prepared azo dye.

The Effect of initial prepared azo dye concentration on photo catalytic degradation processes : A serious of experiments a has been carried out , the effect of initial range (20 – 80 ppm) on photocatalytic degradation process of prepared azo dyewas studied using 0.13gm / 100 cm^3 , temperature equal to 298 K and flow rate of air 10 cm^3/min . The results are listed in Table 2 and plotted in figure 5. It has been observed that the rate of photocatalytic degradation gradually decreases with the increasing of initial prepared azo dye concentration. The concentration 20ppm was the optimum concentration to cover the largest area of the zinc oxide particles, therefore absorbed maximum exciting photons to generate higher concentration of the activated zinc oxide semiconductor. Another reason for this behaviour is the strong absorption of light by the prepared azo dyein the sample which contain high concentration that 20ppm ,prepared azo dyeon 0.13 gm /100cm^3 of zinc oxide. The excess of prepared azo dyeprevent the penetration of light through the successive layers of prepared azo dyeon the zinc oxide surface is weak to generate the required excited state of the reactive prepared azo dyeon zinc oxide (Madhusudhana, 2012; Chen, 2013; Ting, 2013).

Table.2.The change of A_t /A_0 with irradiation time using different concentration of prepared azo dye.

Conc. prepared azo dye	**20 ppm**	**30ppm**	**40 ppm**	**60 ppm**	**80 ppm**
Irradiation Time/min	**A / A_0**				
0	**1**	**1**	**1**	**1**	**1**
10	**0.52**	**0.68**	**0.79**	**0.86**	**0.92**
20	**0.31**	**0.45**	**0.61**	**0.73**	**0.80**
30	**0.20**	**0.30**	**0.45**	**0.61**	**0.71**
40	**0.11**	**0.20**	**0.30**	**0.48**	**0.58**
50	**0.05**	**0.14**	**0.23**	**0.38**	**0.52**
60	**0.04**	**0.13**	**0.21**	**0.35**	**0.51**

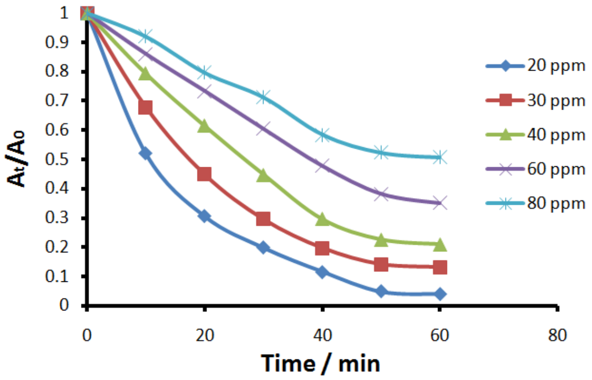

Figure.5.The change of (A_t / A_0) with irradiation time at concentration of prepared azo dye.

The effect of light intensity on photodegradation of prepared azo dyeusing zinc oxide: Several experiments has been investigated change light intensity range (2.15 – 8.22) mW/cm^2.At fixed mass of zinc oxide 0.13 gm/100cm^3 with 20ppm of prepared azo dye,and10cm^3/min flow rate of air babble the rate of photo degradation of prepared azo dye, was measured . Table 3 and Fig.6, illustrate the effect of light intensity on the photocatalytic degradation of prepared azo dye (Hazim, 2015; Qassim, 2015). The results indicate that the photocatalytic degradation of prepared azo dye, increases with the increase of light intensity, because increasing in the number of photons cause to generate of electrons in the conduction band of zinc oxide. The light intensity 8.5 mW / cm^2gives the optimum photo degradation efficiency which is equal to 95.87 %. The results of the change in photocatalytic degradation efficiency (P.D.E) with light intensity plotted in Fig.7.

Table.3.The change of A_t /A_0 with irradiation time using different light intensity.

light intensity/ mW/cm^2	**2.30**	**3.00**	**4.30**	**8.50**
Irradiation Time/min	A_t /A_0			
0	1	1	1	1
10	0.87	0.77	0.66	0.52
20	0.65	0.51	0.35	0.25
30	0.46	0.34	0.22	0.13
40	0.35	0.24	0.16	0.05
50	0.32	0.21	0.13	0.05
60	0.28	0.20	0.11	0.04

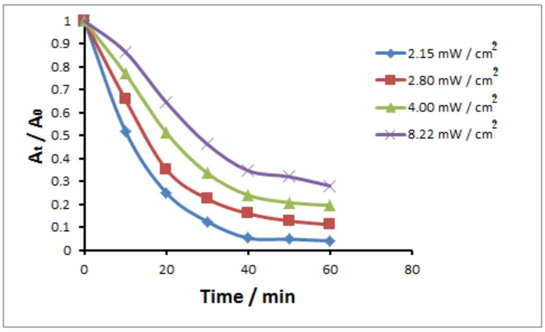

Figure.6. The change of (A / A_0)with irradiation time at different light intensity with 0.13 gm/100cm^3 zinc oxide on photocatalytic degradation of prepared azo dye

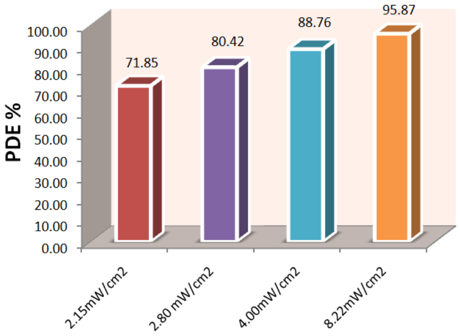

Figure.7. The change of Photocatalytic Degradation Efficiency with irradiation time of different light intensity

4. CONCLUSION

- The compound has been not degraded in case of absent of catalyst.
- The compound has been successfully degraded when used the catalyst with the light.
- The optimum condition for the Photocatalytic degradation of reactive blue equal 0.13 gm / 100cm^3 mass of zinc oxideand 20ppm concentration of prepared azo dyeand 8.22 mW/cm^2.
- The percentage efficiency of degradation reactive blue equal 95.87%.

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