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Solar Photocatalytic of Reactive Blue Dye in Aqueous Suspension of V_2O_5

Abstract- Decolorization of blue azo dye (Cibacron Blue FN-R) in simulated wastewater has been investigated as a function of solar photocatalytic process. Different parameters that affected on the removal efficiency, such as initial concentration of dye, pH of the solution, catalysis dosage, and H_2O_2 concentration, were evaluated to find the optimal operation conditions. From the experimental results, the desired pH value for solar photocatalytic V_2O_5 was 2 and the best catalysis dosage was 500mg/L. In addition, the most efficient H_2O_2 concentration was 100 mg/L for V_2O_5 . The mathematical models that describe the photocatalytic process have been predicted by using response surface methodology (RSM). The decolorization efficiency by using V_2O_5 as solar photocatalytic can be reached to 97%.

Keywords- azo dye, photocatalysis, vanadium oxide.

Received on: 19/06/2016

Accepted on: 19/01/2017

How to cite this article: R.H. Al-Anbari, A.H. Al-Obaidy and E.A. Mohammed, "Solar Photocatalytic of Reactive Blue Dye in Aqueous Suspension of V_2O_5 ," *Engineering and Technology Journal*, Vol. 35, Part A, No. 1, pp. 1-8, 2017.

1. Introduction

The dyes are common industrial residues present in wastewater of different industries, ordinarily in the textile dyeing process, inks, and photographic industries, among others [1]. Textile industry produces large volume of colored dye effluent, which are toxic and non-biodegradable [2].

Dyes contained two types of group, namely chromophore (chromophore is an electron withdrawing group) and auxochromes (are electron releasing groups) which are responsible for their color. The most important chromophores are the azo ($-N=N-$), carbonyl ($C=O$), methine ($-CH=$), and nitro (NO_2) groups [3].

Azo dyes represent the major types of dyes that utilized in textile industry [2]. Azo dyes have been classified to different types, one of these essential types are reactive dyes which are typically consist of azo-bond as chromophore group linked with a variety of reactive groups. These types of dye have a property that made them resistance to biodegradation and as a result made their elimination severely from effluent wastewater by means of conventional treatment methods, like adsorption, ion exchange, and biological treatment [4]. These dyes have several effects on human health due to their containment an aromatic compound. Some of these effects are: carcinogenic effect, allergies, skin irritation or dermatitis.

Furthermore, these dyes have an acute and chronic toxicity. These health effects came from the high tendency of these dyes to adsorb through the gastrointestinal tract, lungs, skin, and also formation of hemoglobin adducts and disturbance of blood formation [5].

There are several available methods to treat this wastewater: adsorption, coagulation & precipitation, aerobic and anaerobic process, and others [6]. One difficulty with these methods is that they are not destructive but only transfer the contamination from one phase to another. Therefore, a new and different kind of pollution is faced and further treatments are deemed necessary [7,8]. An alternative to conventional methods, "advanced oxidation processes" (AOPs) have been developed based on the generation of very reactive species such as hydroxyl radical. The generated hydroxyl radical can oxidize a broad range of organic pollutants quickly and non-selectively [9,10]. Among the (AOPs), heterogeneous photocatalysis appears as an emerging solution to the environmental pollution of aquatic system [10].

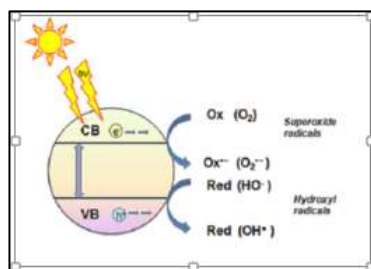
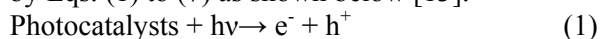


Figure 1: Schematic of semiconductor excitation [12]

A photocatalyst is defined as a substance, which is activated by adsorbing a photon and is capable of accelerating a reaction without being consumed; these substances are invariably semiconductors [11]. The photocatalytic reaction mechanism shown in Figure 1 can be described by Eqs. (1) to (7) as shown below [13]:



Several compounds such as metal oxides (TiO_2 , ZnO , ZrO_2 , V_2O_5 , Fe_2O_3) and metal sulfides (CdS , ZnS) have been studied as potential semiconductor photocatalysts [11,14]. Among these semiconductors, TiO_2 is more applicable because of its low cost, consistency, self-reduction, and good performance in atmospheric condition [15]. The main problem with TiO_2 is that it can absorb only a small portion of solar spectrum in the UV region, and has a high recombination rate of photoinduced electron-hole pairs at or near its surface [16,14,17].

Many researchers study the removal of textile dye by using semiconductors. Kansal et al. (2009) showed that the photocatalytic activity of ZnO under UV light was better than TiO_2 to remove black and orange reactive dyes. Jiang et al. (2008) studied removal of basic blue dye under solar radiation by using bicomponent TiO_2 - ZnO powder. 100% color removal was achieved in about 1 hr.

The present study was carried out to monitor the decolorization of the reactive blue dye (Cibacron Blue FN-R), the variation in ORP (oxidation-reduction potential), Ec (Electrical conductivity) and pH of the solution by using V_2O_5 as a photocatalysis under solar light irradiation instead of UV-lamp. The effects of important parameters such as pH, initial concentration of reactive dye, catalyst loading and volume of H_2O_2 were examined with respect to the highest efficiency of decolorization of reactive blue dye. The reaction kinetics has been established and a mathematical model that describes the process has been created.

2. Experimental Set-up and Materials Used

I. Materials

The blue reactive dye RB had been supplied from AL-Kut textile factory south of Baghdad, (Department of Dying and Printing). Simulated solutions were prepared by dissolving a defined amount of dye in the required volume of distilled water. The chemical formula for this dye is $\text{C}_{29}\text{H}_{23}\text{CuFN}_9\text{O}_{12}\text{S}_3 \cdot 3\text{Na}$ and the color index number is CI Reactive Blue 235 [18-21]. Analytical grade reagents of V_2O_5 , H_2SO_4 , NaOH and H_2O_2 were used as such without further purification.

II. Experimental set-up

Different oxidation processes was carried out by preparing a suspension of simulated synthetic solution containing blue reactive dye and the selected photocatalysis, this suspension was transferred to a 1000 ml Pyrex beaker on a magnetic stirrer. This suspension was kept in dark for about 15 min to achieve the equilibrium of adsorption after that the degradation was accomplished under sunlight and the average intensity of sunlight was measured by using UV-radiometer (UV-340A, Lutron, USA). The heat up of the solution was not much and the temperature varied within $\pm 6^\circ\text{C}$ between starting and ending point of reaction.

Samples were withdrawn from beaker at regular time interval and filtered by using $0.45\ \mu\text{m}$ membrane filter then the dye concentration was measured by UV-spectrophotometer (UV-1800 Shimadzu) and ORP, Ec and pH of these samples were also measured.

The adsorption of reactive dye on the surface of catalyst considers as one of the essential factors that effect on the photocatalytic reaction. So, the time required for adsorption of reactive dye on V_2O_5 was estimated.

The amount of catalysis taken were 100, 200,300,400,500 and 600 mg/L and the concentration of H_2O_2 added were 0, 100, 200, 300,400 mg/L. the initial concentration of reactive blue dye chosen were 25, 50,75 and 100 mg/L. To study the effect of pH 2,4,6,8 and 10 were selected as initial pH value of the solution.

3. Kinetic analysis

The most suitable model that has been used to illustrate the photocatalytic process is the Langmuir-Hinshelwood (LH), which is represented by the following equation [22]:

$$r = -\frac{dc}{dt} = k_{\text{LH}} \frac{KC}{1+KC} \quad (8)$$

Where: r [$\text{mg L}^{-1}\text{s}^{-1}$] represents the reaction rate, C [mg L^{-1}] the pollutant concentration at time t during degradation, K [$\text{mg}\cdot\text{L}^{-1}$] the equilibrium constant for pollutant adsorption onto catalyst and k_{LH} [$\text{mg L}^{-1}\text{s}^{-1}$] the reaction rate constant at maximum coverage. Under certain experimental conditions, $KC \ll 1$, so that the LH model is usually closer to first-order reaction kinetics and Eq. (8) will be simplified to:

$$r = \frac{dc}{dt} = -k_{\text{LH}} \cdot KC = -K_0 C \quad (9)$$

Where, K_0 [min^{-1}] $= k_{\text{LH}} K$ is the pseudo-first-order rate constant [22].

The intensity of solar irradiance was certainly fluctuated as a result of several environmental variables (i.e., time of day, cloud, season, etc.) and for the purpose of normalizing the data that obtained at various solar light intensity, Eq. (10) [23] was used:

$$t_{30W,n} = t_{30W,n-1} + (t_n - t_{n-1}) \frac{UV}{30} \frac{V_i}{V_T} \quad (10)$$

In Eq. (10), t_n represent the exposure time for each sample, UV is the average solar ultraviolet radiation that measured through t_n , and t_{30W} is the "normalized illumination time" and refers to the value of time at constant solar UV irradiation of 30 W/m^2 (the typical solar UV irradiation on a fully sunny day around noon), V_i is the irradiate volume in the reactor and V_T the total volume in the reactor [24]. In that way, photocatalytic process could be evaluated as a function of t_{30W} which take the environmental conditions into account [23]

4. Results and Discussion

I. The adsorption time

The adsorption of dye on V_2O_5 was carried out in dark (without exposure to sunlight) by mixing the dye solution with catalyst in a 500 ml beaker. The samples were withdrawn at regular intervals from this beaker and the dye concentration was determined. Figure 2 illustrates the variation of decolorization rate with time due to adsorption onto V_2O_5 surface. From this Figure, it is clearly seen that the time required for adsorption was about 15 min. So, the adsorption time for all subsequent experiments will be fixed at 15 min.

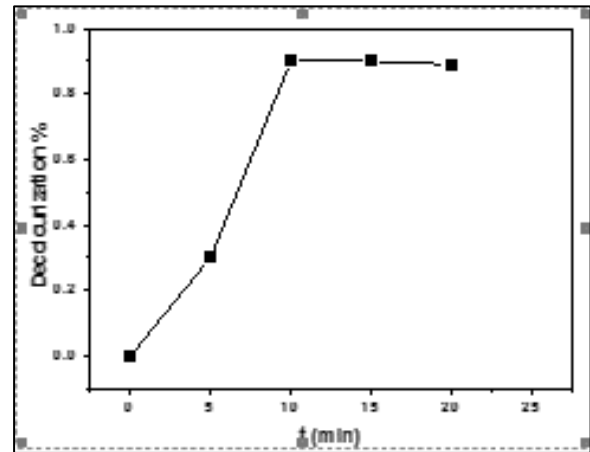


Figure 2: The Adsorption of blue dye onto V_2O_5

II. Effect of pH of dye solution

The pH value of aqueous solution is a key parameter for photocatalytic process. The aqueous solution of dye having 25 mg/L concentration and 100 mg/L of V_2O_5 were treated by varying the initial pH of solution from $\text{pH}=2$ to 10 . pH value was justified by using $0.1\text{N H}_2\text{SO}_4$ and 0.1N NaOH . Figure 3 shows the effect of pH on the removal efficiency of blue reactive dye ORP values. It can be concluded from this figure that the photocatalytic process was higher in acidic media ($\text{pH}=2$) for V_2O_5 . These results can be interpreted by the change of surface charge of catalyst with pH value. Each catalyst has point of zero charge (pH_{pzc}), at this pH the net charge on catalyst surface is equal zero [25]. Catalyst surface is positively charged at $\text{pH} < \text{pH}_{\text{pzc}}$ whereas it is negatively charged for $\text{pH} > \text{pH}_{\text{pzc}}$ [26]. Point of zero charge (pzc) for V_2O_5 is approximately at $\text{pH } 2.5$. For $\text{pH} \leq 2.5$, the V_2O_5 surface will be positively charged, while it will be negatively charged for $\text{pH} \geq 2.5$ [27]. Figure 3 shows the variation in oxidation reduction potential (ORP) of the solution with pH value. It is clearly noticed that the ORP was affected by the initial pH of solution.

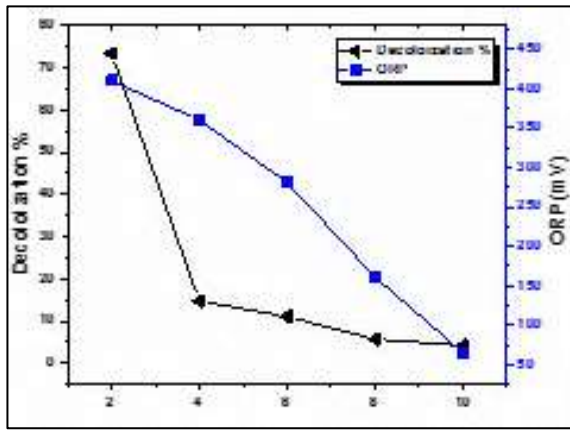


Figure 3: The decolorization rate and ORP values of reactive blue dye change with different initial pH

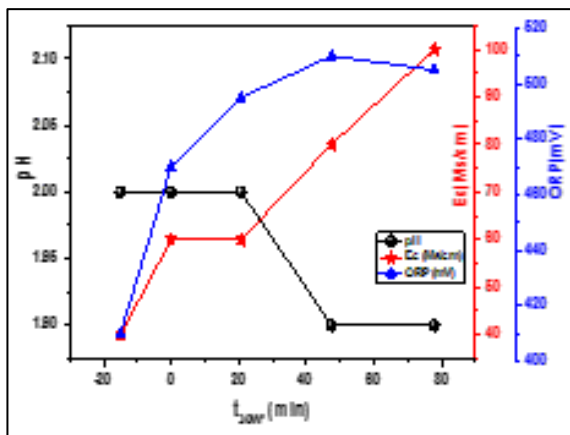


Figure 4: Change of ORP, pH and EC values of solution with illuminating time

The ORP values were decreased when pH of solution was increased and maximum ORP value notices at pH=2, the same results were obtained by Wu and Wang, 2012 [32]

For pH=2, the variation of ORP, Ec (electrical conductivity) and pH of the solution through all the experimental time was presented in Figure 4.

It is easily to observe that the pH value was dropped at the end of the experiment this behavior can be attributed to the degradation of dye to an organic acid. On the other hand, the ORP value was increased from about 410 mV to 510 mV which indicates that the oxidation activity increased throughout the reaction time. The electrical conductivity of solution was also increased at the end of the reaction time due to emergence of inorganic ions (like Na^+) from degradation of reactive dye.

III. Effect of photocatalyst loading

The photocatalyst mass have an evidential effect on the photocatalysis process of reactive blue dye. The mass of the photocatalyst used was changed from 100 to 600 mg/L.

From Figure 5, it is clearly observed that the decolorization processes was increased when the mass of catalyst increased up to 500 mg/L. This increment in decolorization can be attributed to the increment in the active sites on the catalyst surface, which accelerated the removal process. Further increases in photocatalyst loading over 500 mg/L causes decrease in the decolorization rate. These results can be rationalized with the increasing of solution turbidity when increasing the photocatalyst load, which subsequently reduces the penetration of the light into the solution. The variation of ORP, EC and pH of the dye solution with time are shown in Figure 6, the explanation of these variation are same as results in Figure 4.

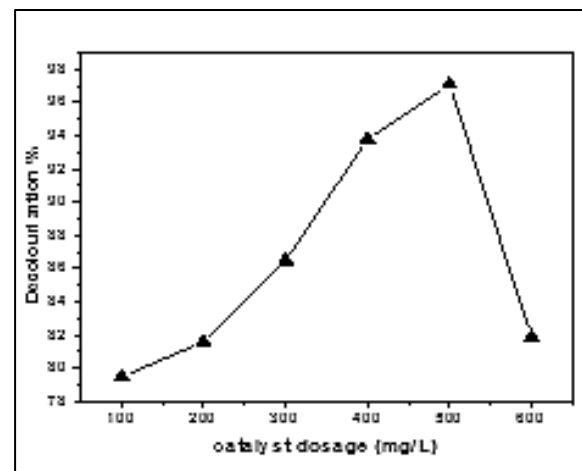


Figure 5: The effect of V_2O_5 load on the decolorization percent of blue dye

IV. Effect of H_2O_2 concentration

The addition of H_2O_2 has a major effect on the photocatalytic process; this additive increases the rate of reaction by improvement the formation of hydroxyl radical. This is due to inhibition the recombination of the electron-hole in present of H_2O_2 . The decolorization rate of reactive blue dye can be accelerated by increasing the concentration of OH radicals by the addition of oxidant like H_2O_2 [27].

As illustrated in Figure 7, the results show that the decolorization percent increases when the concentration of H_2O_2 increased, the percentage removal becomes maximum at 100 mg/l and after that begin declining with further increase in H_2O_2 concentration. This effect is due to the fact that at a higher H_2O_2 concentration, scavenging of OH radicals will take place, which can be expressed by Eq. (11). The result is the formation of perhydroxyl radicals which are significantly less reactive than hydroxyl radicals and thus influence the degradation of dyes [28-30]:

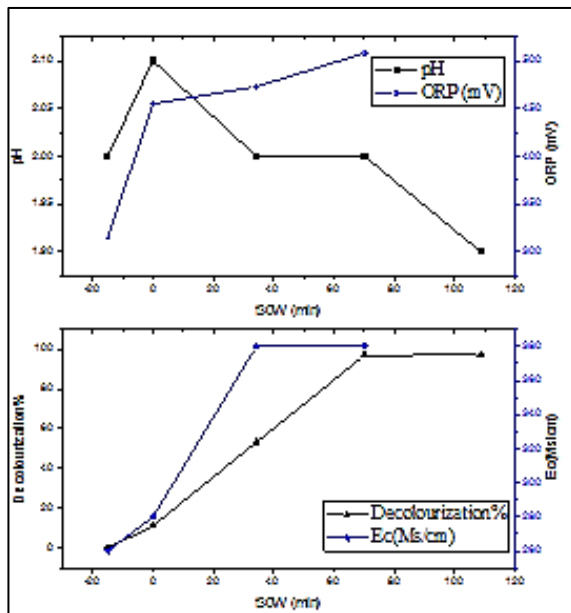
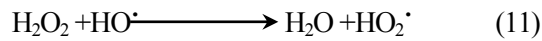


Figure 6: Variation of ORP, pH & EC values of solution with illuminating time for 500 mg/L catalyst dosage

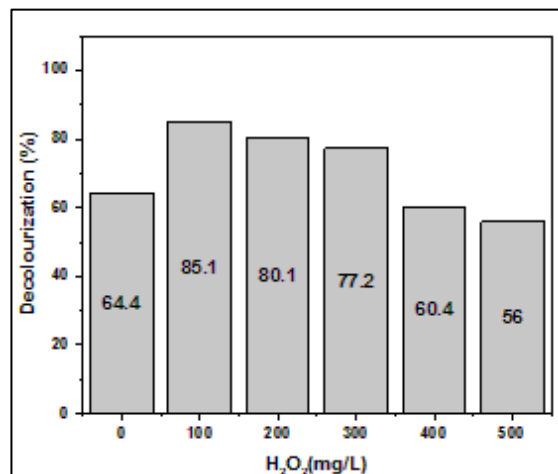


Figure 7: The effect of H_2O_2 concentration on decolorization percent of reactive blue dye (number inside column represent decolorization %)

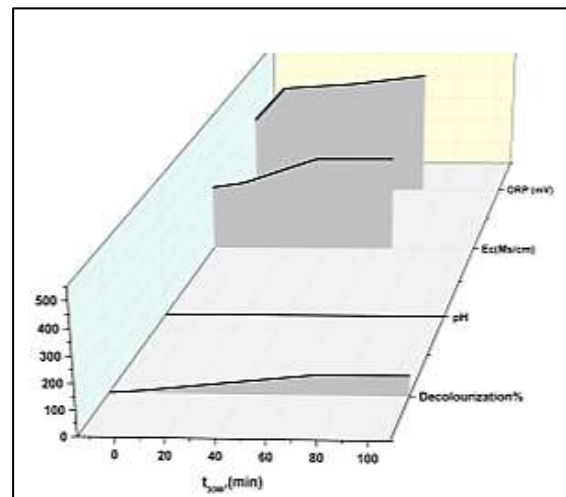


Figure 8: Variation of ORP, pH & EC values of solution with illuminating time for H_2O_2 conc. mg/L

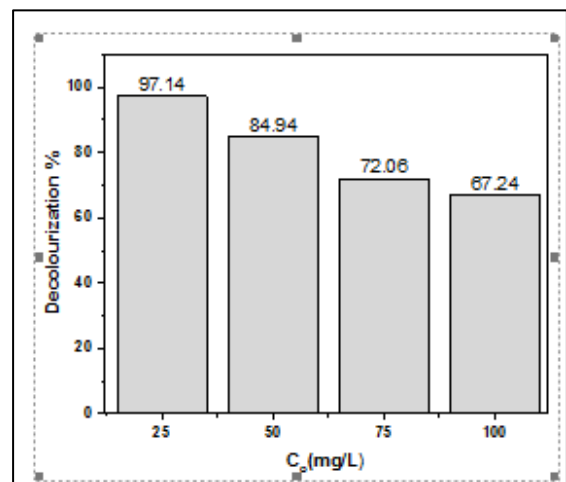


Figure 9: The LH models for reactive red dye

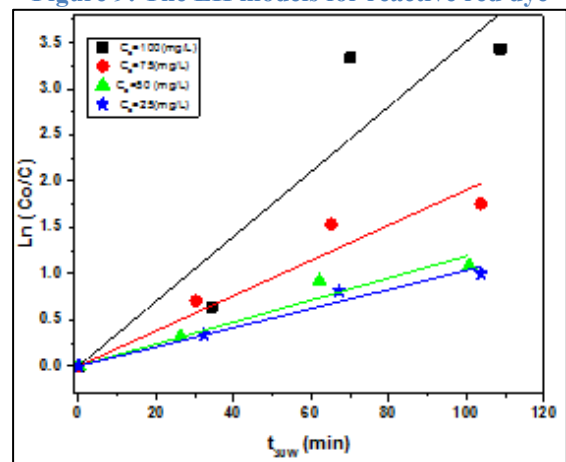


Figure 10: The LH models for reactive blue dye

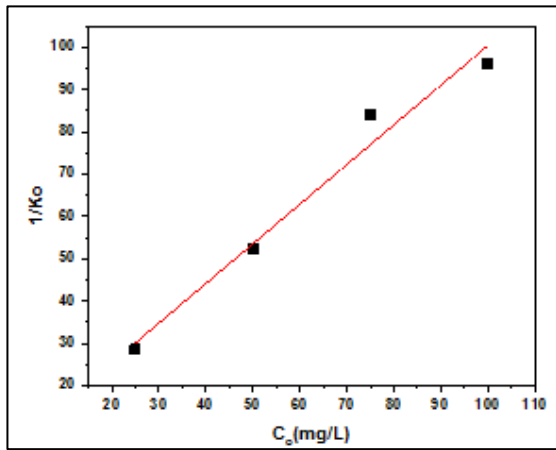


Figure 11: Variation of 1/K vs C_0 of reactive dye

Table 1: The kinetic analysis data

Catalysis	Eq. for linear fitting	k_{LH} [mg L ⁻¹ min ⁻¹]	K [mg L ⁻¹]	Correlation coefficient R^2
V ₂ O ₅	$Y = 6.625 + 0.938X$	1.066	0.142	0.962

The regression coefficients and the kinetic constants were estimated and tabulated in Table 1.

The variation in the spectrum of the solution was monitored by measuring the absorption of light with UV-Vis spectrophotometer. Figure 12 represents the reduction in color of reactive blue dye over time at the optimum operation conditions (pH=2, H₂O₂ conc.=100 mg/L, V₂O₅ dosage=500 mg/L, and C_0 =25 mg/L)

5. Mathematical Model

Response surface method (RSM) is a collection of mathematical and statistical techniques that are useful for modeling, analysis and optimizing the process in which response of interest is influenced by several variables and the objective.

From the experimental runs that carried out, mathematical relation of the response function (Y) and impact of the independent factors is specified as a polynomial model Eq.(12). The regression coefficients estimated by response surface regression analysis were estimated by fitting experimental data to the polynomial model [31]

$$Y = \beta_0 + \sum_{i=1}^k \beta_i X_i + \sum_j \sum_{i=2}^k \beta_{ij} X_i X_j + \sum_{i=1}^k \beta_{ii} X_i^2 \quad (12)$$

The regression coefficients for intercept, linear, quadratic and interaction terms are represented by β_0 , β_i , β_{ii} , β_{ij} , respectively. While the

independent variables that affect the response Y are denoted by X_i and X_j .

The mathematical models that adequately describe the photocatalytic of reactive blue dye under solar light irradiation were developed. The dependent variable of our process is the decolorization percentage of reactive dye and the independent variables namely X_1 , X_2 , X_3 and X_4 represent pH, H₂O₂, catalysis dosage and influent concentration of dye. Mathematical models that represented the process are given in Eq. (13) with regression coefficient of 0.987

$$Y = -176.6 - 383.9X_1 - 1854.2X_2 + 2782.8X_3 - 3576.4X_4 + 2.4X_1^2 - 16 \times 10^{-5}X_2^2 - 18 \times 10^{-5}X_3^2 + 21 \times 10^{-5}X_4^2 - 3781.9X_1X_2 + 1986.68X_1X_3 - 48.7X_2X_3 - 7932.8X_1X_4 + 571.5X_2X_4 - 75.4X_3X_4 \quad (13)$$

Figure 13 demonstrated the a valuable regression between the actual and the predicted results thus, establishing the model's accuracy under the independent variables were investigated .

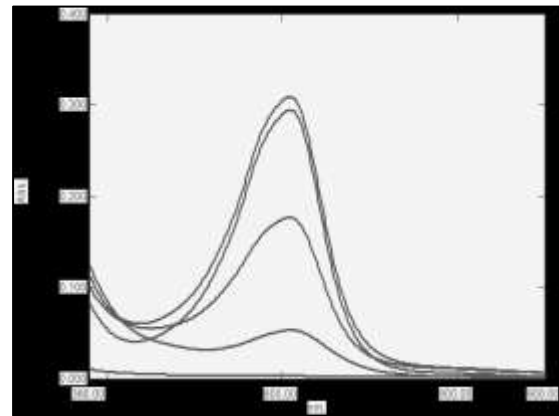


Figure 12: Variation in the spectrum of reactive blue dye at regular time intervals

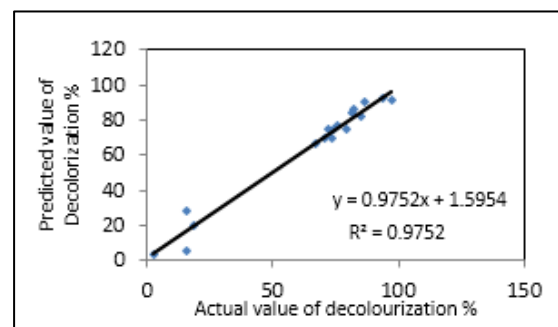


Figure 13: Relation between actual responses and predicted values of decolorization percent of reactive blue dye

6. Conclusion

Photocatalytic process of reactive blue dye was carried out by using V₂O₅ under solar light irradiation.

The optimum conditions for decolorization were obtained to be: pH=2, H₂O₂ concentration =100mg/L, catalyst load=500mg/L and initial concentration of dye=25 mg/L.

The ORP, pH and Ec value of the dye solution was monitored throughout the reaction period. It was observed that the pH is decreased at the end of reaction time this is due to degradation of reactive blue dye and producing organic acid. The ORP value at the end of reaction time is greater than its value at the beginning; this can be considered as a reliable indicator on the powerful of the oxidation process. Furthermore, Ec value was clearly increased through the reaction time as a result of emergence of inorganic ions during degradation of reactive blue dye.

The decolorization process could be fitted to L-H model and the reaction kinetics was estimated.

The photocatalysis process was appropriately represented by the developed model equations. The suitability of the model was found to be satisfactory. In addition, data generated from the quadratic polynomial fitted the experimental data well.

Acknowledgement

We greatly acknowledge the kind support obtained from the Environmental Research Center, University of Technology and Environmental Engineering Department, Collage of Engineering, Al-Mustansiriyah University during the experimental work.

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