

Homogeneous Photocatalytic Degradation of Aqueous Solutions of RR84 Dye Under Wide Experimental Conditions at 25 °C

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ABSTRACT

This study deals with the homogeneous photocatalytic degradation of an example of a reactive textile azo dye, namely Reactive Red 84 (RR84). The measurements were carried out under wide experimental conditions. The variable parameters included the absence and presence of the UV source. H₂O₂ was used as a photocatalyst in the presence of UV with variable concentrations (1.96 x 10⁻⁵ - 1.96 x 10⁻⁴)M, UV lamp emitting at 254 nm and the temperature was maintained at 25 ± 1°C. Also the pH was varied from (1 - 10) and the dye concentration ranged from (25 - 150) ppm. The optimum dye concentration, pH and H₂O₂ concentration were found to be 25 ppm, 1 and 3.91 x 10⁻⁵ M, respectively. The degradation was almost completed within 4 min. for the above mentioned optimum conditions. The results are discussed in terms of advanced oxidation process (AOP) of the dye under investigation.

Key words: Reactive Red 84; UV/H₂O₂ process; decolorization; degradation; advanced oxidation process.

Introduction

The residual dyes from different sources (e.g., textile industries, paper and pulp industries, dye and dye intermediates industries, pharmaceutical industries, tannery, and Kraft bleaching industries, etc.) are considered as wide variety of organic pollutants introduced into the natural water resources or wastewater treatment systems. One of the main sources with severe pollution problems worldwide is the textile industry and its dye containing wastewaters (Baban *et al.*, 2010).

The textile industries also use significant amounts of dyes and chemicals. Kant (2012) stated that a normal sized textile industry uses about 0.51-0.58 kg of chemicals per one cubic meter of water per day. Significant portions of the textile dyes are found in a high concentration in textile effluents due to the large amount of unfixed dyes on the fabrics that gets washed away during the textile finishing processes.

Soloman *et al.*, 2009 showed that 10-25% of textile dyes are lost during the dyeing process, and 2-20 % are directly discharged as aqueous effluents in different environmental components.

Reactive dyeing has been the most common method for cotton dyeing in the past years (RAJKUMAR *et al.*, 2006). Dyes in wastewater cause aesthetic problems as they absorb and scatter sunlight and thus affect the aquatic ecosystem (Devi *et al.*, 2009).

Ananthashankar *et al.*, 2013 reported that textile effluents are capable of preventing photosynthesis of aquatic plants by blocking the penetration of sunlight into water. Nese *et al.* 2007 stated that textile effluents could prevent the growth of aquatic organism by increasing COD/BOD contents of the receiving and depleting dissolved oxygen.

Azo compounds represent the largest group of colorants with respect to both number and production volume. These compounds contain one or more azo groups (-N=N-), mostly linked to benzene or naphthalene rings (Devi *et al.*, 2009). Some azo dyes, via metabolic cleavage of the azo linkage, can produce potentially carcinogenic aromatic amines (Zaharia *et al.*, 2009). This is one more reason for the open concern about the release of azo dyes to the environment.

The color of textile wastewater is mainly due to the presence of textile dyes, pigments and other colored compounds. A single dyeing operation can use a number of dyes from different chemical classes resulting in a complex wastewater (Correia *et al.*, 1994). Moreover, the textile dyes have complex structures, synthetic origin and recalcitrant nature, which makes them obligatory to be removed from industrial effluents before being disposed into hydrological systems (Anjaneyulu *et al.*, 2005).

Several treatments are employed to remove contaminant from textile effluents. Suspended solids in textile effluents are removed by primary treatments such as coagulation, sedimentation and screening (EPA, 2004).

The organic substances in the effluent are removed by secondary treatment mechanisms including activated sludge, aerated lagoons and trickling filters (Lajeunesse *et al.*, 2012). Dissolved salts and metal ions are removed using tertiary treatment processes such as electrodialysis or reverse osmosis (Babu *et al.*, 2007). However, these primary, secondary and tertiary treatment processes were found to be ineffective as some of the

textile dyes, which have complicated structure, are nearly impossible to be degraded by these methods (Ghoreishi and Haghighi, 2003).

Recently, AOPs have been used as techniques which are alternative to physico-chemical phase transfer methods. These techniques are successfully applied for the complete mineralization of organic pollutants in water (Ahandani *et al.*, 2014 ;Huanosta *et al.*, 2012).

AOPs involve different processes, such as H₂O₂/UV, O₃/UV, H₂O₂/O₃/UV, TiO₂/UV, H₂O₂/ Fe²⁺, UV/H₂O₂/Fe²⁺, H₂O₂/Fe³⁺, Fe²⁺/oxalate/UV, H₂O₂/Fe³⁺/oxalate, H₂O₂/Fe³⁺/oxalate/UV, Mn²⁺/ oxalic acid/O₃ and H₂O₂/Fe²⁺/Fe³⁺/UV.

AOPs are divided into two types (homogeneous and heterogeneous) according to the catalyst and pollutant phase , Emara *et al.* , 2009 showed that the structural modification of catalyst has an effective rule on the rate of degradation of pollutants during advanced oxidation processes.

AOP has the ability to take place under any temperature or pressure without the production of secondary components or sludge. AOPs make use of oxidants like UV, O₃, H₂O₂, Fe²⁺, TiO₂, ultra-sound and electron beam irradiation to generate OH[•] radicals, which have the potential to oxidize organics in the wastewater effluents (Stasinakis, 2008). These OH[•] radicals: (a) have an oxidizing potential of 2.33 V (Kdasi *et al.*, 2004), (b) have an affinity towards electrons in the system and hence are called electrophiles and (c) have one unpaired electron on their outer shell which makes them unstable and as such try to attain a more stable configuration by reacting with the organics.

Some of the advantages of using UV/H₂O₂ in comparison to other AOPs are a considerably safe and easy operation, a reduction of the chemical oxygen demand (COD) and a short reaction time. The reaction of hydroxyl radicals, generated by photolysis of H₂O₂, with organic contaminant includes three different mechanisms: hydrogen abstraction, electrophilic addition and electron transfer (Durán *et al.*, 2009).

The aim of this work is to study some important factors influencing the advanced oxidation process (AOP) of Reactive Red 84 using H₂O₂ as a photocatalyst.

Experimental:

1-The photochemical reactor:

The photochemical degradation was carried out in a specially designed reactor which shown in (Fig.1).

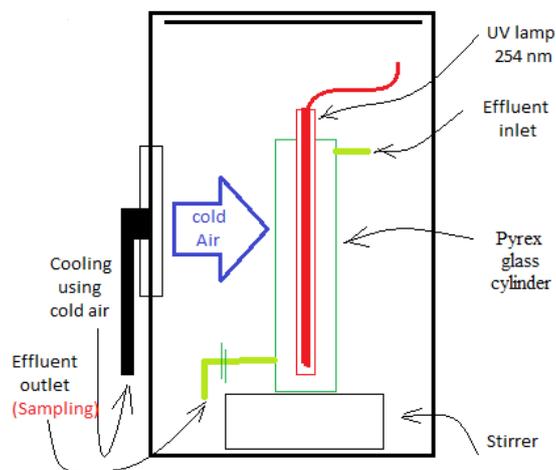


Fig. 1: Photo-chemical reactor.

Stock solutions of the pollutants containing the desired concentration were prepared in deionized water. A UV lamp- isolated by a small quartz tube – was fixed at the center of a Pyrex glass cylinder that has an inlet opening at the top and an outlet opening at the bottom .This glass cylinder was equipped with a magnetic stirrer to prevent settling of organics. A continuous cold air flow current generated by an air blower and connected with a thermostat was used to maintain the reaction temperature at 25±1 °C. The light source was 11 W UV lamp (Japan) having a wavelength of 254 nm. The spectra were taken with UV-Vis spectrophotometer (UV-2900).

2-Chemicals:

The chemicals used in this study were (RR84) dye (from Sigma Aldrich) , 30% hydrogen peroxide (from Fisher Scientific), sodium hydroxide and hydrochloric acid. Where, The sodium hydroxide (NaOH) and

hydrochloric acid (HCl) were used to adjust the pH range of reaction medium between 1 and 10 . The properties of the RR84 dye are as shown in Table 1.

3-Solutions of dye:

Aqueous solutions of reactive red 84 dye of different concentrations ranging from 25 ppm to 150 ppm were prepared by dilution of a stock solution (1000 ppm) which was prepared by dissolving the desired amount of dye in deionized water.

4-Procedure:

For the degradation experiments, A fixed amount of photo catalyst was added to 250 ml of dye solution in each trial at the desired pH. The homogeneous solution was subjected to irradiation under UV light. An aliquot was taken out with the help of a pipette at different time intervals. Then absorption spectra were recorded and the rate of decolorization was calculated in the terms of change in intensity at 495 nm. The percentage of decolorization was calculated as follows:

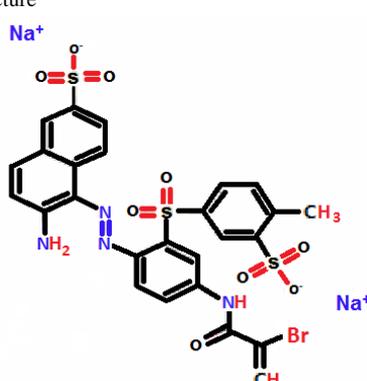
$$\% \text{ Decolorization} = 100 \times (C_0 - C)/C_0$$

Where,

C_0 = initial concentration of dye solution.

C = concentration of dye solution after photo irradiation.

Table 1: The properties of RR84 dye.

Properties Value
Chemical formula $C_{26}H_{19}BrN_4Na_2O_9S_3$
Molecular weight 753.529
Functional group Azo
Color Red
□ max (nm)495
Structure


In order to determine the effect of catalyst loading, the experiments were performed by variation of catalyst concentration at a fixed dye concentration and at a definite pH . For studying the effect of dye concentration the best catalyst concentration (which achieved the highest DD% in the smallest interval) was used with definite pH and variation of dye concentration . Finally, for studying the effect of pH the best concentration catalyst and dye concentration were used with variation of pH .

Results and Discussion

1- Ultraviolet treatment:

The degradation degrees (DD%) of different concentrations of dye solution as a result of exposure to UV alone at natural pH are shown in (Fig.2). The maximum DD% achieved with the small concentration (25 ppm) which give the largest degree of degradation after 30 min. where it was 42.1 % and the DD% decrease with increasing dye concentration . At 150 ppm dye concentration the DD% was 6.79% .

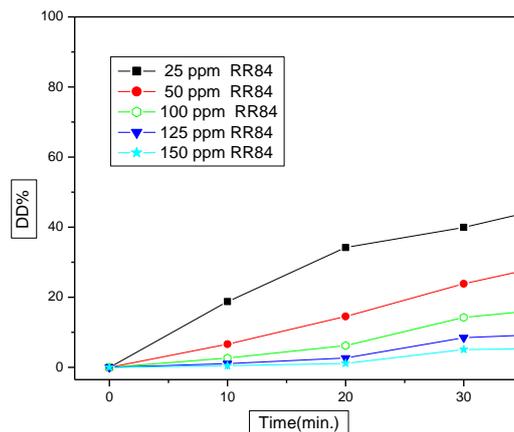


Fig. 2: The degradation degree of different initial concentrations of RR84 in presence of UV at normal pH and Temp. 25 ± 1 °C.

The degradation of textile effluent when exposed to UV could be explained by the following reactions (SET, 2012; EPA US, 2012).



Eqn. (1) shows the production of atomic oxygen radicals as a result of exposure to UV. The oxygen radical (O°) in turn reacts with other oxygen molecules resulting in the production of ozone (O_3) as shown in Eqn. (2).

Eqn. (3) shows the reaction between ozone and water which results in the production of hydrogen peroxide (H_2O_2). Ozone then interacts with hydrogen peroxide to form hydroxyl radicals (OH°) as shown in Eqn. (4).

Finally, the produced hydroxyl radicals interact with the dye molecules causing the destruction of the dye. The low removal efficiency observed in this study was due to the low quantity of hydroxyl radicals produced in the photoreactor and/or the very short life span of ozone and hydrogen peroxide as reported by Kesselman *et al.*, 1996.

Several researchers indicated that the use of UV alone as photo-oxidant was not very effective in destroying textile dyes.

Ghaly *et al.*, 2008 stated that degradation of textile dyes with UV alone was not effective unless it was accompanied with other photocatalysts.

2- H_2O_2 / UV treatment:

The experiments were carried out to study the degradation of RR84 employing H_2O_2 as a catalyst under UV light. Various parameters which affect the DD% such as catalyst loading (1.96×10^{-5} to 1.96×10^{-4} M), pH range from (1 to 10), initial concentration of dye (25 ppm to 150 ppm) and time of decolorization were assessed under UV light.

2-1 Effect of the initial H_2O_2 Concentration:

The concentration of hydrogen peroxide is an important parameter that influences the efficiency of dye removal by the UV/ H_2O_2 process (Devi *et al.*, 2009). An excess of H_2O_2 is theoretically required to produce sufficient OH° radicals. Numerous authors have reported that the concentration of H_2O_2 may either enhance the photoreaction rate or inhibit it due to the scavenging action of peroxide, depending on the concentration (Sadik and Nashed, 2008).

Therefore, an optimum concentration of H_2O_2 in the reaction must be reached. (Fig.3).shows the effect of H_2O_2 concentration on the degradation of RR84 at natural pH of dye. It can be seen that the degree of decolorization increase by increasing H_2O_2 concentration from 1.96×10^{-5} up to 3.91×10^{-5} M ; further increase in the dose of catalyst had a bad effect on degradation of the dye. The photocatalytic destruction of other organic pollutants has also exhibited the same dependency on catalyst dose which reported by numerous authors such as the results recorded by Emará *et al.*, 2002.

The rate constants of degradation (K) vs. different initial concentrations of H_2O_2 are summarized in (Fig. 4).

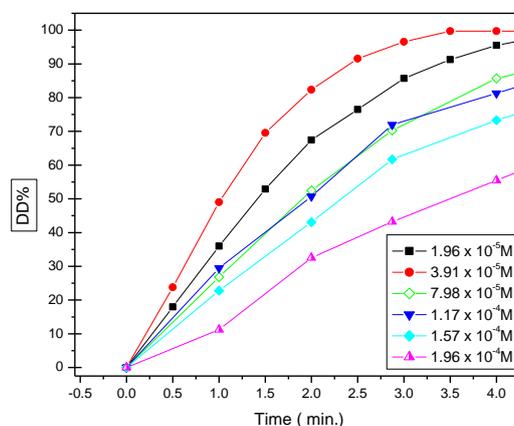


Fig. 3: The photo-catalytic degradation of 25 ppm RR84 at different initial concentrations of H_2O_2 , normal pH and Temp. 25 ± 1 °C in the presence of UV.

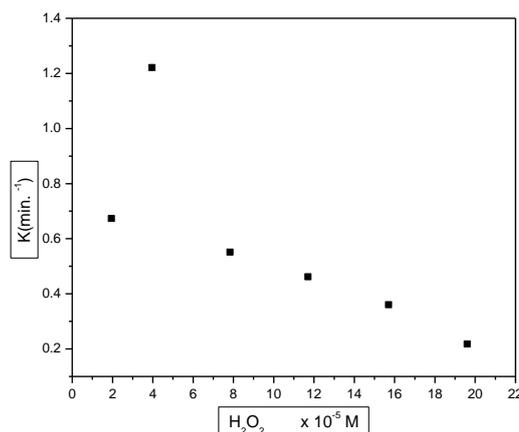


Fig. 4: Plot of $K(\text{min.}^{-1})$ VS. different initial concentrations of H_2O_2 in presence of UV & 25 ppm RR84 at normal pH and Temp. 25 ± 1 °C in the presence of UV.

This plot shows that k is considerably increased from 0.673 to 1.22 min^{-1} with increasing hydrogen peroxide concentration from 1.96×10^{-5} to 3.91×10^{-5} M. At the low concentration of H_2O_2 , a relatively low concentration of $^{\circ}\text{OH}$ radicals was formed for dye oxidation, which resulted in a low decolorization rate. However, with increasing peroxide concentration, more hydroxyl radicals were generated upon its photo-dissociation according to the eqn. (6); this step is known as hydroxyl radical initiation.



At an initial peroxide concentration above 3.91×10^{-5} M, the degradation rate constant decreased from 1.22 min^{-1} gradually by increasing H_2O_2 concentration and reaches 0.218 min^{-1} at 1.96×10^{-4} M. When the initial peroxide concentration was very high, the generated $^{\circ}\text{OH}$ radicals mostly reacted according to one of the next equations which is known as hydroxyl radical propagation .

1) with excess H_2O_2



2) with hydroperoxyl radicals.

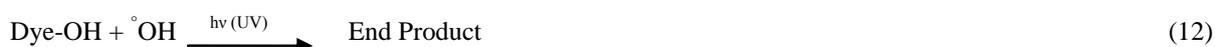


3) Dimerize to form.



Therefore, the optimum range of hydrogen peroxide concentration giving the maximum RR84 degradation rate is approximately 3.91×10^{-5} M.

Hydroxyl radical termination



This mechanism was proposed by Daneshvar *et al.*, 2005 for the decolourization of organics with the $\text{H}_2\text{O}_2/\text{UV}$ process.

Shu and Chang, 2005 compared the decolourization effects of six different azo dyes and found that more than 95% degradation efficiency was achieved when the dye solution was treated with $\text{UV}/\text{H}_2\text{O}_2$ in less than 11.5 min, which was higher than the degradation rate achieved with UV/O_3 for the same time period.

Modirshahla *et al.*, 2007 reported that the improvement in decolourization was not found to be significant above a certain amount of H_2O_2 due to the recombination of hydroxyl radicals in the solution or the reaction between hydroxyl radicals and H_2O_2 creating hydroxyl radical scavenging capacity.

2-2 Effect of the initial pH of dye solution:

The most important parameter that influences the photocatalytic degradation is the solution pH. The efficiency of the catalyst is affected by pH of the solution (Neppolian *et al.*, 2002).

The influence of pH on the rate of degradation of RR84 azo dye by $\text{UV}/\text{H}_2\text{O}_2$ process was investigated at ten different pH values: (1.0, 2.0, 3.0 9.0 & 10.0) at concentration of 25 ppm of RR84, temp. 25 ± 1 °C, 3.91×10^{-5} M of H_2O_2 ; pH was adjusted using HCl and NaOH. Fig.5. shows the effect of pH on the DD% of RR84 at different pH values.

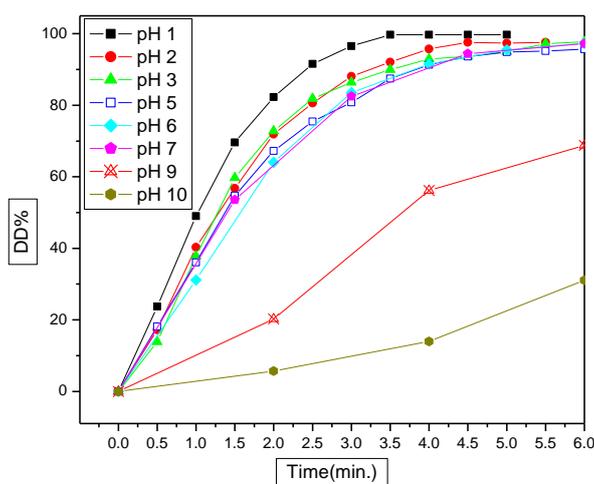


Fig. 5: The photo-catalytic degradation of 25 ppm RR84 at different initial pH, H_2O_2 concentration 3.91×10^{-5} M and Temp. 25 ± 1 °C in the presence of UV.

The DD% of pH 1 achieved the maximum value at min. 3 (96.52%) and this value decreased gradually by increasing pH value. At pH 6 the DD% at min. 3 was equal 82.9% & sharp decrease in DD% at pH 9 and 10 which dropped to 40% and 10.1%, respectively.

Several researchers indicated that photo-oxidation is more efficient under acidic conditions than alkaline conditions. Ganesan and Thanasekaran, 2011 reported that the effect of UV/H₂O₂ degradation decreases as the pH increases. They have also noted that the DD% increased as the concentration of H₂O₂ was increased due to the additional production of hydroxyl radicals. Kavitha and Palanisamy, 2011 stated that the photocatalytic activity was maximum under acidic condition and was found to decrease when the pH range was increased above 6.

At alkaline pH, the concentration of the conjugate base of H₂O₂ increases:

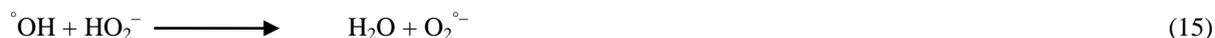


This anion (HO₂⁻) reacts with a non-dissociated molecule of H₂O₂, which leads to oxygen and water, instead of producing hydroxyl radicals under UV radiation:



Therefore, the instantaneous concentration of [∘]OH is lower than expected:

Furthermore, the deactivation of [∘]OH is more important when the pH of the solution is high. The reaction of [∘]OH with HO₂⁻ is approximately 300 times faster than its reaction with H₂O₂.



The reactivity of O₂^{∘-} and HO₂[∘] with organic pollutants is much lower than that of [∘]OH. They preferentially disproportionate and produce hydrogen peroxide and oxygen gas.



On the other hand, several authors have reported that the peroxide self-decomposition rate, which is strongly pH dependent, partly contributes to the decrease of the rate of degradation values in alkaline medium.



Eqn. (18) represents the main reaction which occurs when H₂O₂ exposed to UV at highly alkaline pH (Muruganandham and Swaminathan 2004; Habibi *et al.*, 2006; Shu, Chang, 2005).

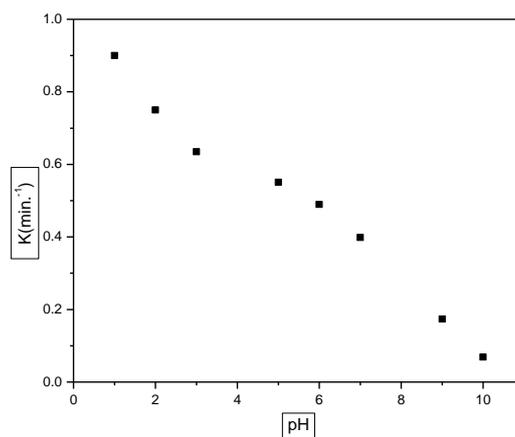


Fig. 6: Plot of K(min.⁻¹) Vs. PH in presence of 25 ppm RR84, H₂O₂ (3.91 X 10⁻⁵) M and Temp 25 ± 1 °C in the presence of UV.

2-3 The effect of initial dye concentration:

The effect of the initial RR84 concentration on the DD% was investigated in the concentration range of 25 ppm and 150 ppm results are presented in (Fig.7).

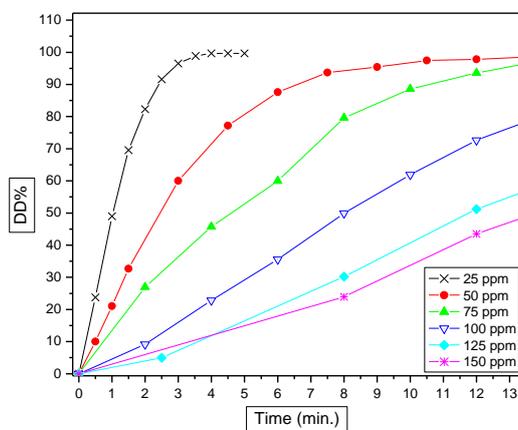


Fig. 7: The photo-catalytic degradation degree of different concentrations of RR84 in the presence of (3.91×10^{-5}) M H_2O_2 , pH 1 and temp. 25 ± 1 °C in the presence of UV.

It is seen that with increasing initial dye concentration, the DD% of dye decreases. The results indicate that the degradation rate constant decreases sharply from 0.90 min^{-1} for a dye concentration of 25 ppm to be 0.32 min^{-1} for a dye concentration of 50 ppm, Then the rate constant of degradation decreases gradually to be 0.072 min^{-1} at a dye concentration of 150 ppm.

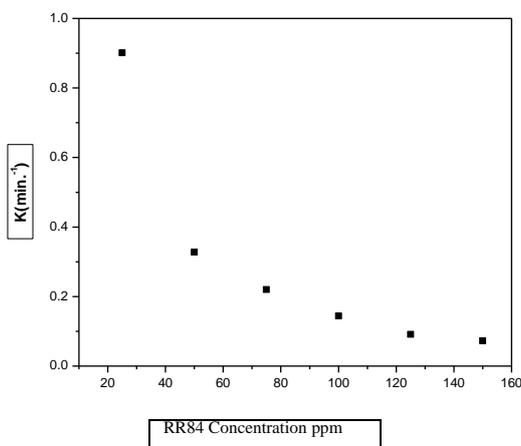


Fig. 8: Plot of $K(\text{min}^{-1})$ Vs. dye concentration in presence of H_2O_2 (3.91×10^{-5}) M, pH 1 and Temp 25 ± 1 °C in the presence of UV.

At high dye concentrations, most of the UV light was absorbed by the dye instead of peroxide. The dye solution becomes increasingly impermeable to UV light, which inhibits photolysis of the peroxide. The UV/ H_2O_2 system would be more effective for relatively dilute dye solutions. Emara *et al.*, 2010 reported the same results similar to the results obtained in this study during the photocatalytic degradation of other pollutants using other catalysts.

Acknowledgement

This research project was supported by the Science Center for Detection and Remediation of Environmental Hazards (SCDREH).

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