

Optimization of Degradation of Trypan Blue Dye using Advanced Oxidation Processes and Photocatalysis under UV Light

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Abstract

In textile wastewater industry, Advanced Oxidation Processes (AOP'S) are used for degrading and removing color from dye baths. . In this study, the effects of initial dye concentration, hydrogen peroxide dosage, pH, Fenton reagent, temperature, sodium perchlorate and photocatalyst used TiO_2 were examined to find out the optimum operating conditions of the treatment processes (dark and photo). Complete degradation was achieved in the relatively short time of 75 minutes irradiation. Faster decolourization was achieved at low pH and high temperature. The removal rate increased with increasing initial concentration of H_2O_2 . The degradation with $\text{H}_2\text{O}_2 / \text{Fe}^{2+} / \text{UV}$ was the fastest , the best results were obtained from photo Fenton's reagent with efficiency more than 98 % at pH=6, $[\text{H}_2\text{O}_2]= 500\text{mg/L}$, $[\text{Fe}^{2+}]=100 \text{ mg/L}$. The concentrations of undegraded dye were detected by spectrophotometer at $\lambda_{\text{max}} =590\text{nm}$.The photodegradation reaction of dye under UV obeys the first order kinetics. The results of photocatalytic degradation illustrate that the rate of degradation process depends on the mass of TiO_2 , the best efficiency was 46 % by using 1gm/L of TiO_2 .

1- Introduction

The environmental risks by effluents of textile wastewater industry are the major source of water and ground water pollution. These risks include highly colored compounds, high level of COD, wide range of pH natural, and resistance to the natural degradation. For instance textile dyeing disposed 100-170 L of colored effluents per Kg of cloth processed⁽¹⁻³⁾ .The degradation of these synthesised dyes have been extensively investigated by different technological and chemical processes such as photocatalytic degradation (SiO_2 , TiO_2 , ZnO ,.....) ⁽⁴⁻⁶⁾ , chemical methods: ozonation ⁽⁷⁻⁹⁾ , chlorination ⁽¹⁰⁻¹²⁾ ,biodegradation⁽¹³⁾ , adsorption^(14,16) and oxidation process⁽¹⁷⁻²⁵⁾ .The present study was carried out to investigate the

removal of color of trypan blue dye Fig(1) using various advanced oxidation processes and photocatalyst(TiO_2) and various reaction conditions and UV light.

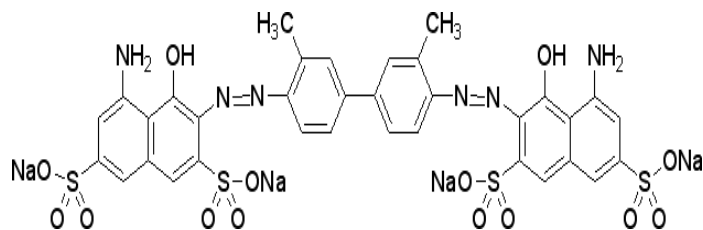


Fig (1): structural formula of Trypan Blue dye.

2- Experimental

1. Materials

All chemicals were used without further purification. Hydrogen peroxide (H_2O_2 30% w/v), sodium perchlorate monohydrate ($\text{NaClO}_4 \cdot \text{H}_2\text{O}$ 100%), Ferrous chloride (FeCl_2), sodium hydroxide (NaOH) and hydrochloric acid were supplied from BDH. Titanium dioxide (TiO_2) was purchased from GCC. Trypan Blue dye ~ 60% was purchased from Aldrich. All the other chemicals and solutions were prepared with distilled water.

2. Instruments

UV-Visible 1650 spectrophotometer (Shimadzu) was used to recording the absorption spectra of aqueous solutions of dye. UV-Visible 7804C spectrophotometer (SUNNY) was used to measured absorbance of dye solutions at ($\lambda_{\text{max}} = 590\text{nm}$). The pH was measured by using microprocessor pH meter 211, HANNA instruments. The Temperature was adjusted by used regulator water bath WB 710M (Optima). Centrifugation of TiO_2 suspensions was performed by using CORP Triup International, Italy instrument.

3. Photoreactor setup

The photoreactor shown in Fig (2), was fitted with low pressure mercury lamp (6 W with the light emitted at 253.7 nm). The UV lamp was putted in the side of reactor and the quartz sleeve was enclosed. The photoreactor was fitted with regulator water bath to maintain the temperature and circulation pump to continuous feed and collection of dye solution in a 1L tank. A syringe was fitted with photoreactor to withdrawn of dye solution samples at meaning time.

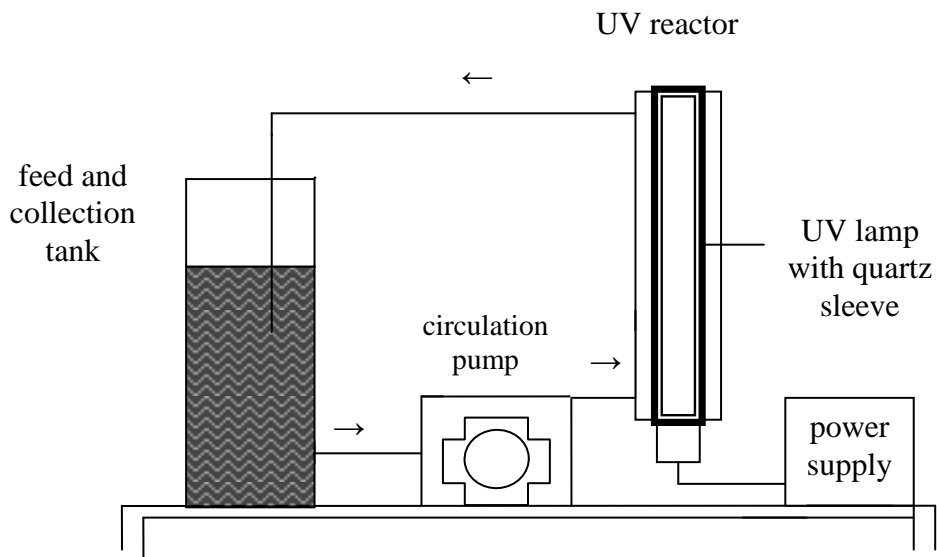


Fig (2): Photoreactor setup

4. Irradiation of dye solutions

All dye solutions were irradiated for a period of 75 minutes (the primary experiments indicated that the most of dye molecules are degraded or become colorless at the time near to this period). Within this time samples were taken by syringe for measuring the dye absorption at $\lambda_{max} = 590\text{nm}$ and then determination of dye concentration by using the calibration curve shown in Fig(3), Fig(4) shows the UV-Visible absorption spectrum of dye.

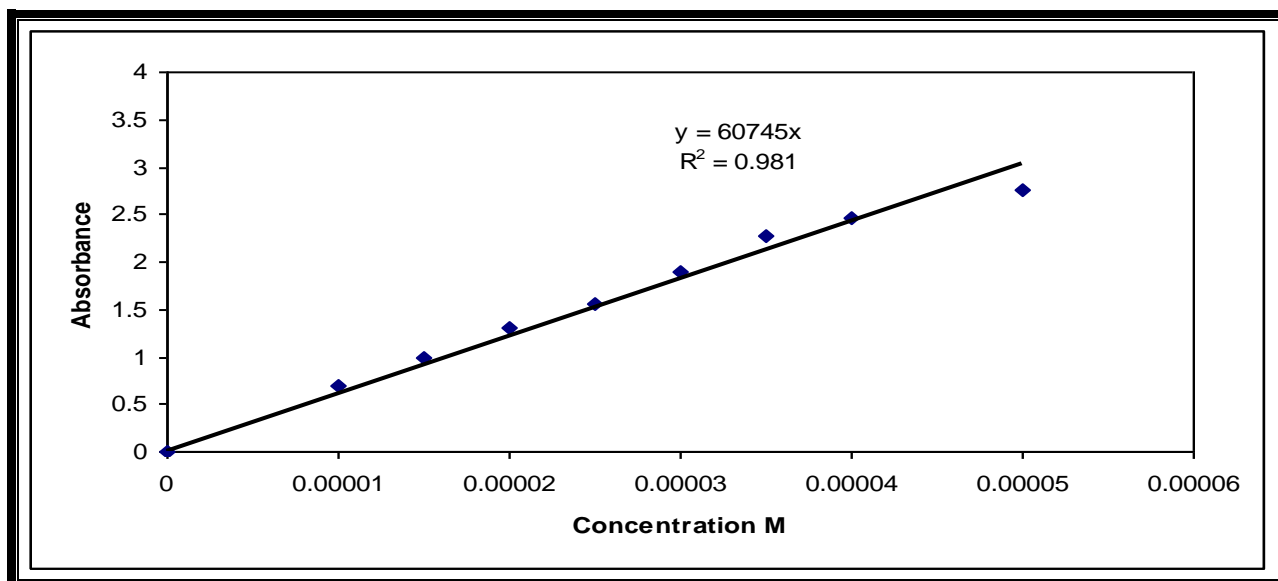


Fig (3): Calibration curve for Trypan Blue dye.

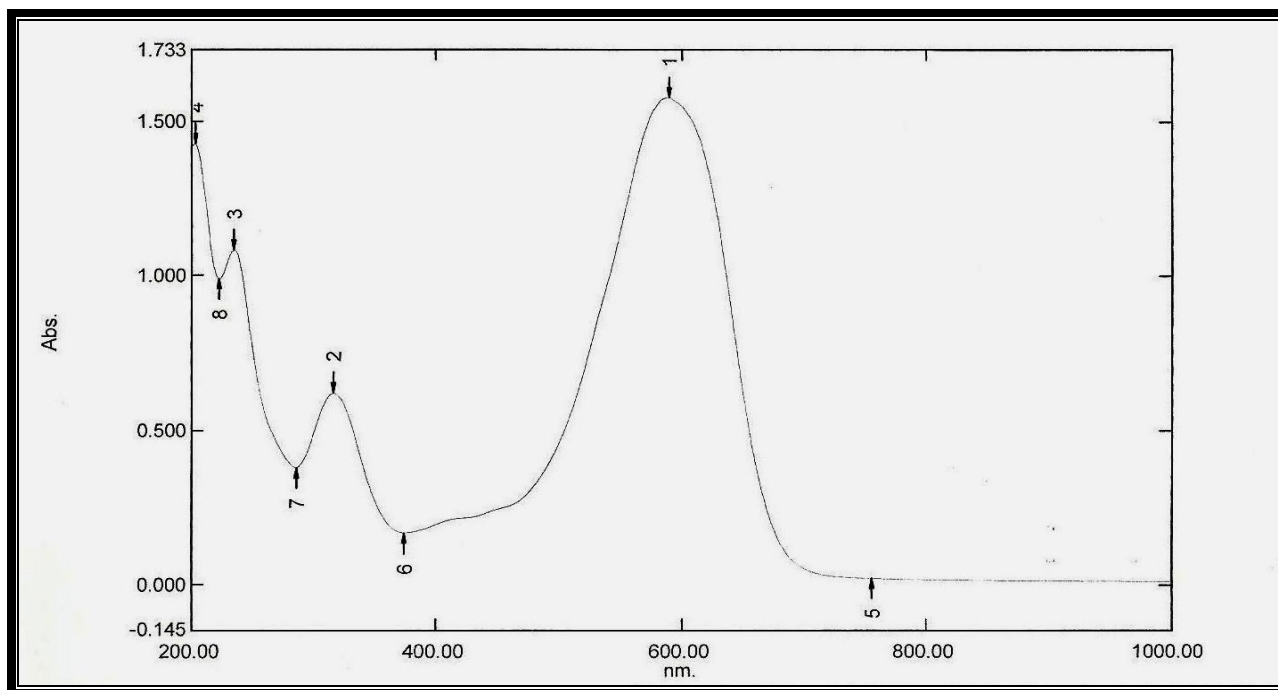


Fig (4): UV-Visible spectrum of aqueous solution of Trypan Blue dye concentration 3×10^{-5} M, pH=6, T=293K.

5. Effect of initial dye concentration

The 1L solutions of dye were prepared by using different concentration in the range (1×10^{-5} - 5×10^{-5} M) in distilled water, and then the solution was irradiated by using UV light in the photoreactor for 75 minutes⁽¹⁹⁾. The concentration of dye was measured by spectrophotometer at $\lambda_{max} = 590\text{nm}$. Other experiments were done in the dark at the same time to check the dye concentration.

6. Effect of initial Hydrogen peroxide concentration

Hydrogen peroxide in the range (150-500mg/L) was added to aqueous solution of dye (3×10^{-5} M) at pH=6, T=293K, then the solutions were irradiated for 75 minutes⁽²⁷⁾. Other samples performed in the dark at the same time, and concentrations were measured by spectrophotometer at $\lambda_{max} = 590\text{nm}$.

7. Effect of initial pH

The effect of pH was evaluated at pH media 1,3,5,7 and 8. H_2O_2 Concentration was 500mg/L. The pH of solutions was adjusted by using NaOH and HCl. All the experiments were performed at 293 K for 75 minutes. Other experiments were done in the dark.

8. Fenton's system

A series of Fenton's solutions were prepared by mixing H_2O_2 (100mg/L) and Ferrous ion at the range (20-100mg/L) at the pH equal of 6 and 293K. Other series of solutions were prepared by mixing H_2O_2 (100-500mg/L) and Fe^{+2} (100mg/L) at pH equal of 6 and 293K. A third series of solution were prepared by using H_2O_2 (100mg/L), Fe^{+2} (100mg/L) at pH=(1-8) and 293K. In all the above solutions, the dye concentration was $3 \times 10^{-5} \text{M}$. All the solutions were irradiated for 75 minutes⁽²⁹⁾, then measuring the concentration was determined by measuring the dye absorbance at $\lambda_{\text{max}}=590\text{nm}$. Other experiments were done in the dark

9. Effect of Temperature

The degradation of dye was performed by irradiation of TB dye solutions ($3 \times 10^{-5} \text{M}$) at temperature range (290 - 308K) and pH 6. The temperature was maintained by using regulator water bath. Some experiments were done in the dark, then the concentration was determined by measuring the absorbance at $\lambda_{\text{max}}=590\text{nm}$.

10. Effect of TiO_2

The degradation of dye solutions were performed by irradiating 1Liter of the solution of dye concentration ($3 \times 10^{-5} \text{M}$) in the presence TiO_2 in the range (0.2-1.2gm/L) at $T=293\text{K}$ for 75 minutes and suspension were magnetically stirred, some experiments were performed in the dark⁽⁶⁾. The concentration of dye was measured in the $\lambda_{\text{max}}=590\text{nm}$ after removing TiO_2 by using centrifugation.

11. Effect of initial sodium perchlorate concentration

Sodium perchlorate in the range (10-100mg/L) were added to ($3 \times 10^{-5} \text{M}$) aqueous dye solution at 293K and pH equal of 6, then the solution was irradiated by using photoreactor for 75 minutes. Other samples performed in the dark. Samples were taken by syringe through the reaction to measure the concentration by using calibration curve.

3-Results and Discussion

1. Effect of initial dye concentration

The effect of different dye concentrations on the degradation of TB dye using UV irradiation for 75 minute. The results are shown in Fig (5) for TB dye at pH=6 , T=293K. The percent of color removal decrease from 42% to 16% with increasing the concentration from $1 \times 10^{-5} \text{M}$ to $5 \times 10^{-5} \text{M}$. Higher dye concentrations increased the absorption of UV radiation, therefore decreasing the availability of UV light for reactions , thus lowering the formation of hydroxyl free radicals in the solution⁽¹⁹⁾ . The degradation rate was first-order kinetic, the TB contain two azo bonds and degradation of this dye was due to the initial electrophilic cleavage of its chromophoric azo (-N=N-) bond attached to naphthalene ring⁽²⁶⁾ . The kinetic constant can also be calculated by Eq. (1).

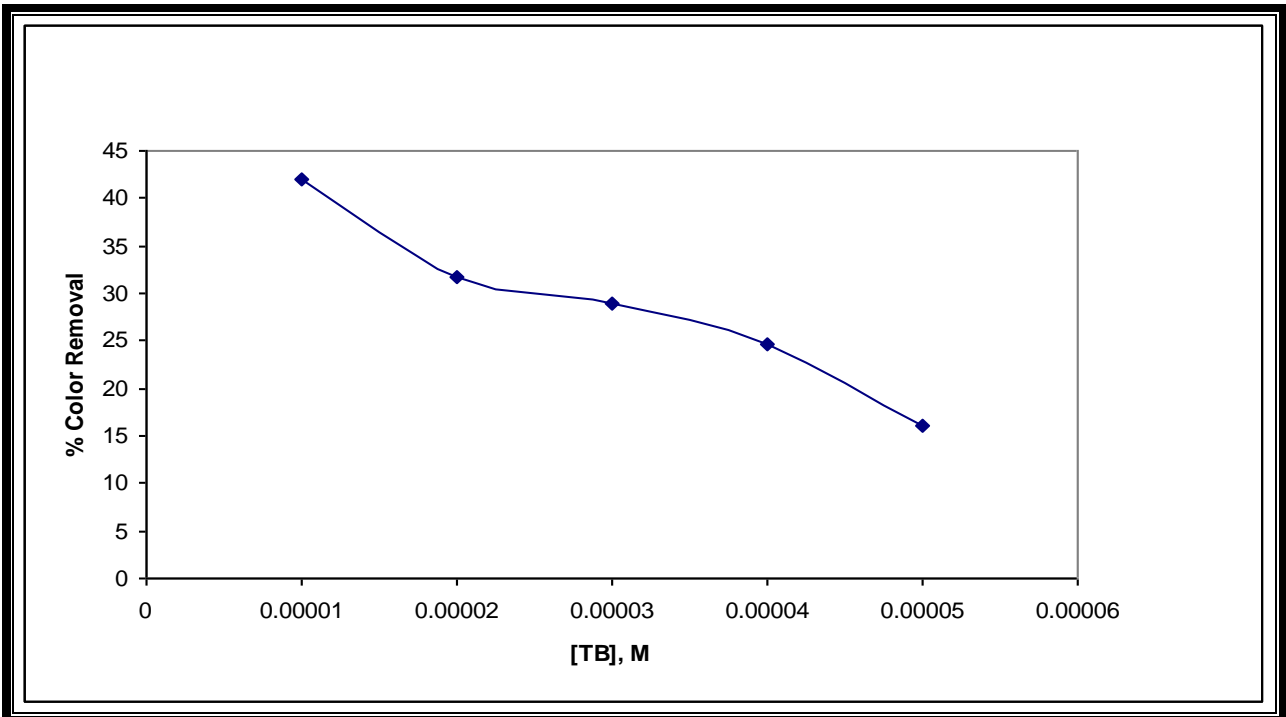
$$\ln \frac{A}{A_0} = -kt \quad \dots\dots\dots(1)$$

Where :

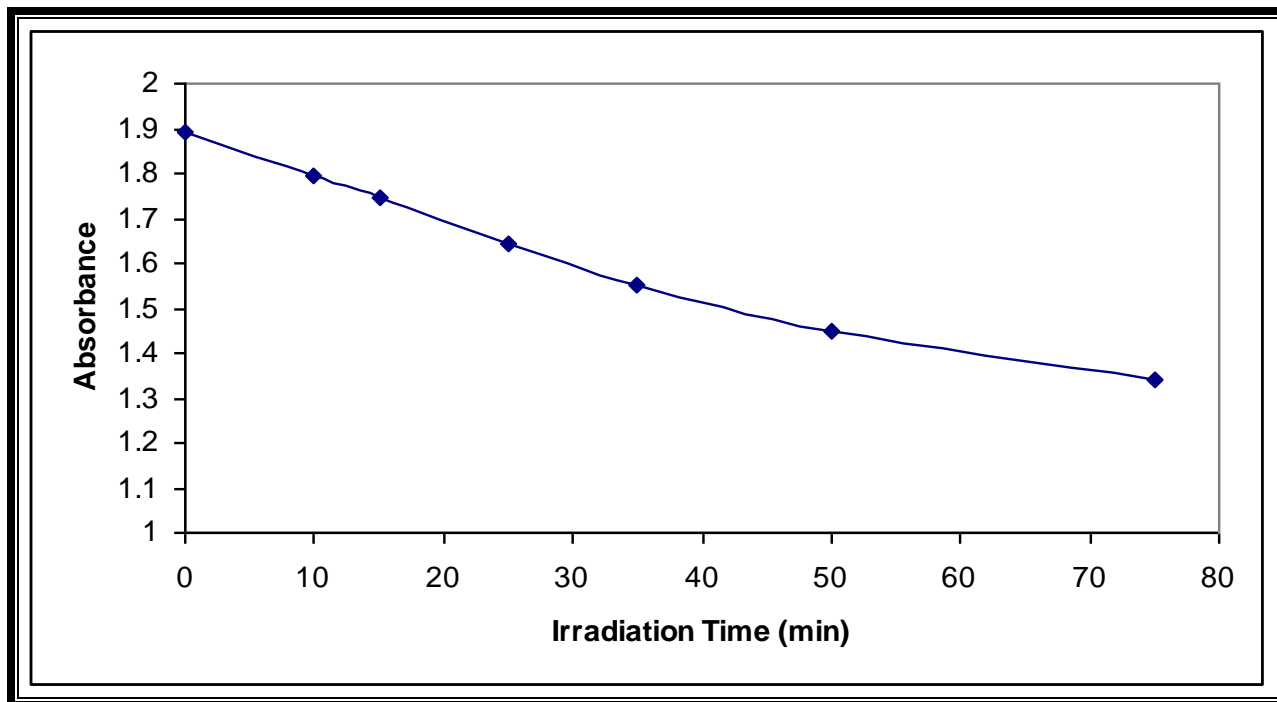
A_0 and A are the initial and final absorbance before and after irradiation.

k : reaction rate constant.

Fig (6) shows the relation ship between absorbance and time irradiation. It is clear the dye concentration is decreases as long as the time increased.



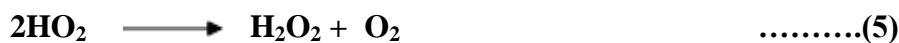
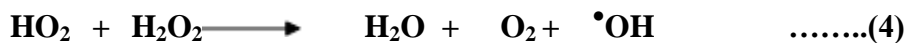
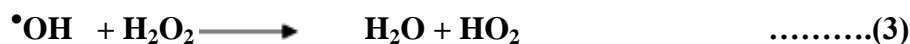
Fig(5):Effect of dye concentrations on color removal at pH=6,T=293K.



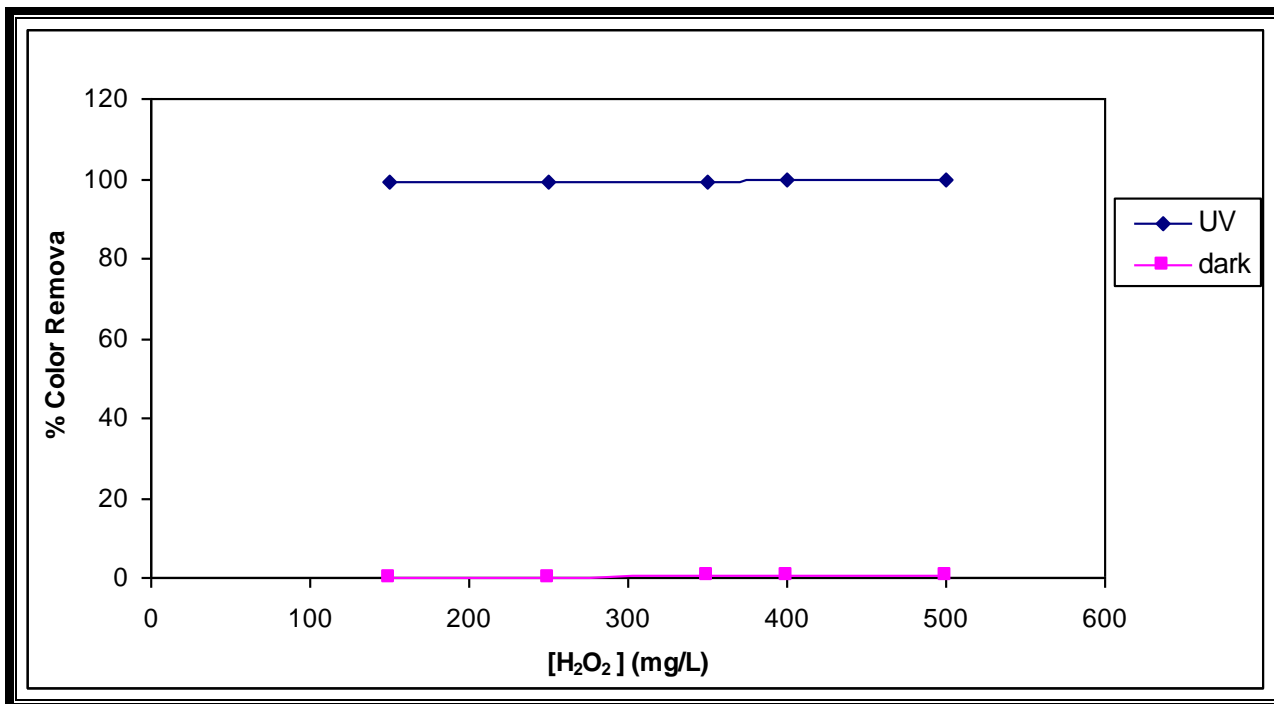
Fig(6): Effect of Irradiation time on Color removal , [TB]= 3×10^{-5} M , pH=6, T=293K.

2. Effect of initial Hydrogen peroxide concentration

The effect of varying the initial H_2O_2 concentration was studied in the range(100-500 mg/L) and TB dye concentration 3×10^{-5} M at pH=6 , T=293K , and the results is shown in Fig(7). At low hydrogen peroxide concentrations, formation of $\cdot\text{OH}$ is the kinetic determining step. Hydrogen peroxide cannot generate enough hydroxyl radicals and the oxidation rate is logically slow. At higher hydrogen peroxide concentration more $\cdot\text{OH}$ was produced leading to a faster oxidation rate. However, these free radicals preferred to react with the excess of H_2O_2 rather than with the dye chromophore ^(27,28) . Photolysis of H_2O_2 by ($\text{H}_2\text{O}_2/\text{UV}$) generates hydroxyl free radical according to following ⁽¹¹⁾ :



The results show that the higher color removal in presence light was 99.6% for 500mg/L of H_2O_2 .The dark experiments without irradiation showed no degradation was detected because that H_2O_2 generate very high active $\cdot\text{OH}$ only under UV or visible light .



Fig(7):Effect of different H₂O₂ concentration on color removal,[TB]=3x10⁻⁵ M, pH=6 at T=293K.

3. Effect of initial pH

The effect of pH on photodegradation is very important , experiments are conducted at 3 x 10⁻⁵M dye concentration in presence of 500mg/L H₂O₂dose at different initial pH values ranges from 1.0 to 8.0, for 75 minutes at T=293K, the result is shown in Fig(8). The results show that, high color removal 99.5% are observed at pH 5.0 and decrease slightly in alkaline media in presence UV,and 43% in pH=1 in dark at the same conditions. The degradation rate under UV/ H₂O₂ should decrease with increasing pH⁽²⁸⁾ because the concentration of •OH will decreased . Hydrogen peroxide undergoes decomposition in alkaline medium produced oxygen and water rather than producing hydroxyl free radicals under UV-Visible irradiation ⁽²⁷⁾.

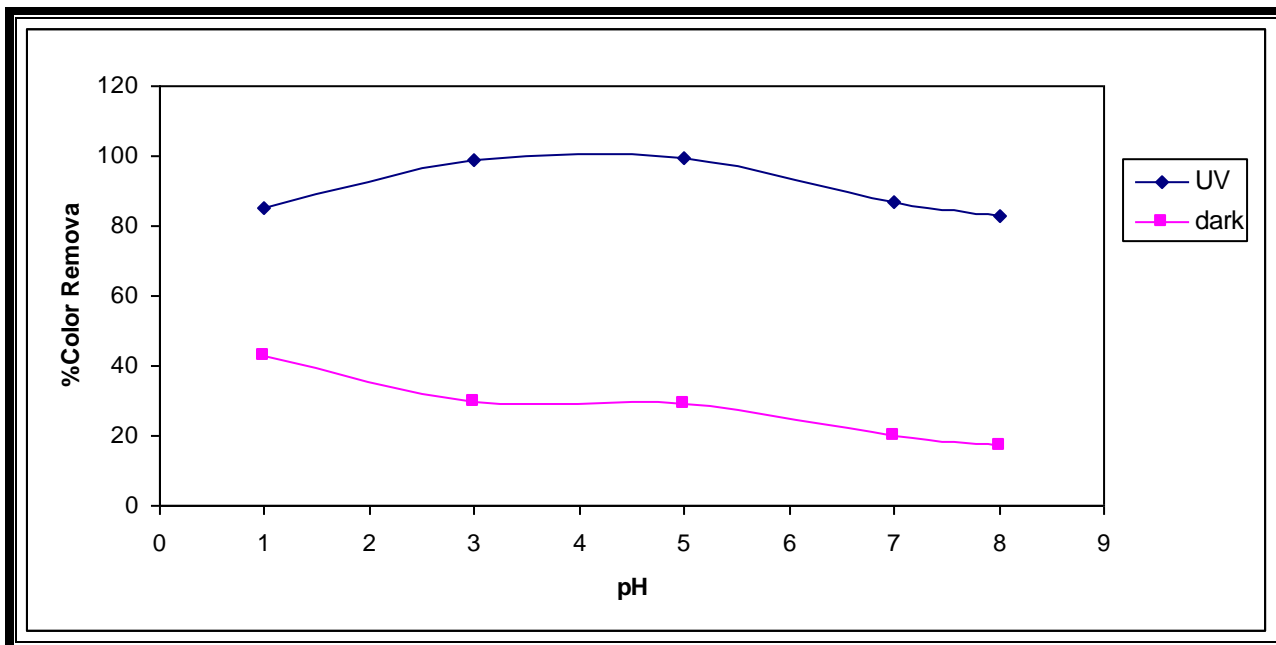
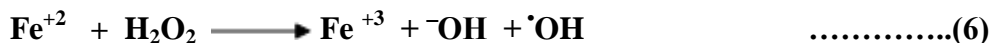


Fig (8):Effect of different pH media on the degradation of dye.[TB]= 3×10^{-5} M, $[H_2O_2]=500\text{mg/L}$, T=293K.

4. Fenton's system

The rate of the degradation by H_2O_2 alone is extremely low for many dyes ,especially azo dyes, addition of Fe^{+2} accelerate the generation of hydroxyl radical ⁽²¹⁾.Figs(9,10) shows the effect of photo and dark Fenton’s reagent at T=293K for 75 minutes. The best result was reached 98% of color removal with $Fe^{+2} / H_2O_2 / UV$ used $[H_2O_2]= 500\text{mg/L}$, $[Fe^{2+}]=100 \text{ mg/L}$, with increasing the concentration of Fe^{+2} , H_2O_2 the percent of color removal increased after 75 minutes of reaction time. In Fenton system under acidic conditions, a Fe^{+2} / H_2O_2 mixture produces hydroxyl radicals as following:



The high color removal under acidic conditions is due to more $^{\cdot}OH$ generate, and low color removal at basic conditions because Fe^{+2} ions rapidly oxidized to Fe^{+3} ions formation $Fe(OH)_3$. The Fenton process requires strict pH control because the best yields are obtained at pH values ranging between 3.0 and 5.0 ⁽²⁹⁾ ,The pH value was adjusted either by HCl or NaOH and the results shown in Fig (11).The best result was reached 97.6% at pH=3 under UV light and 88.5% at pH=5 in dark ,this showed that photo Fenton have more efficiency than dark which is attributed to the effect of absorption of UV light on the increasing of concentration of hydroxyl radicals which are more generated in acidic medium.

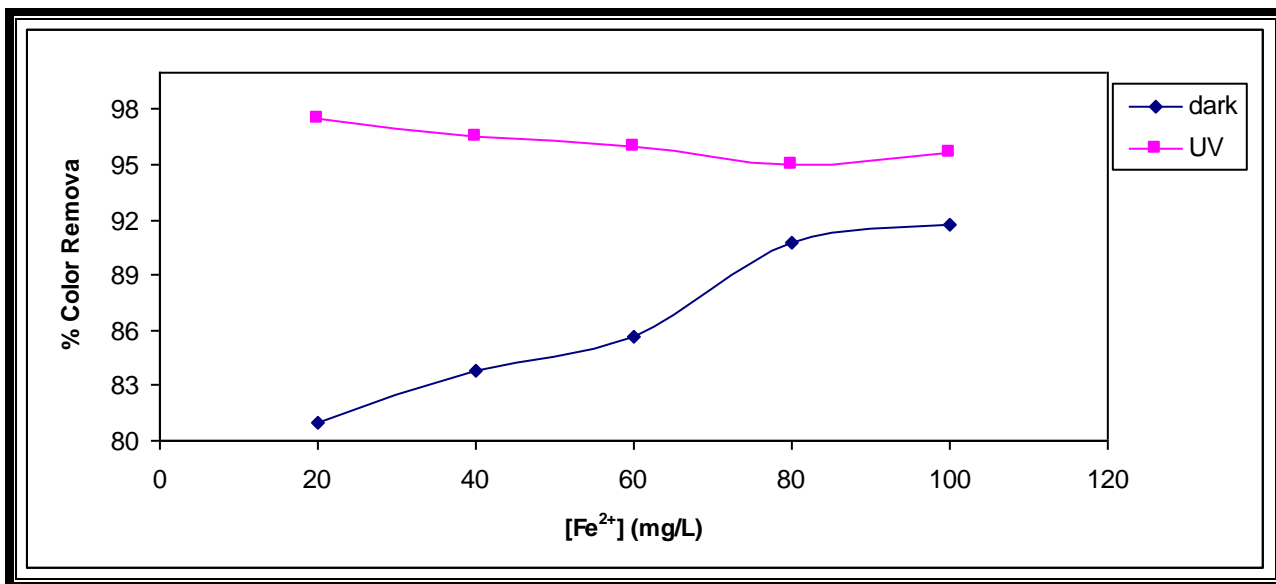


Figure (9): Effect of $\text{H}_2\text{O}_2/\text{Fe}^{+2}$ system on the degradation of dye $[\text{TB}]=3 \times 10^{-5} \text{ M}$, $[\text{H}_2\text{O}_2]=100 \text{ mg/L}$, $\text{pH}=6$ at $T=293 \text{ K}$.

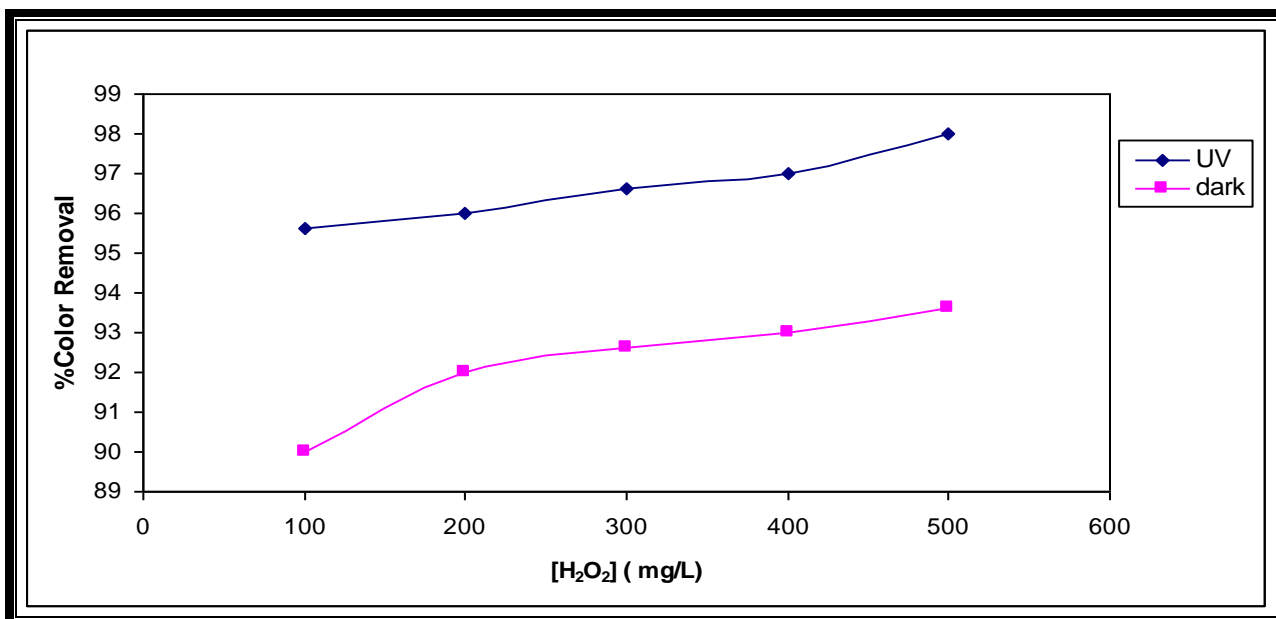


Figure (10): Effect of $\text{H}_2\text{O}_2/\text{Fe}^{+2}$ system on the degradation of dye $[\text{TB}]=3 \times 10^{-5} \text{ M}$, $[\text{Fe}^{+2}]=100 \text{ mg/L}$, $\text{pH}=6$ at $T=293 \text{ K}$.

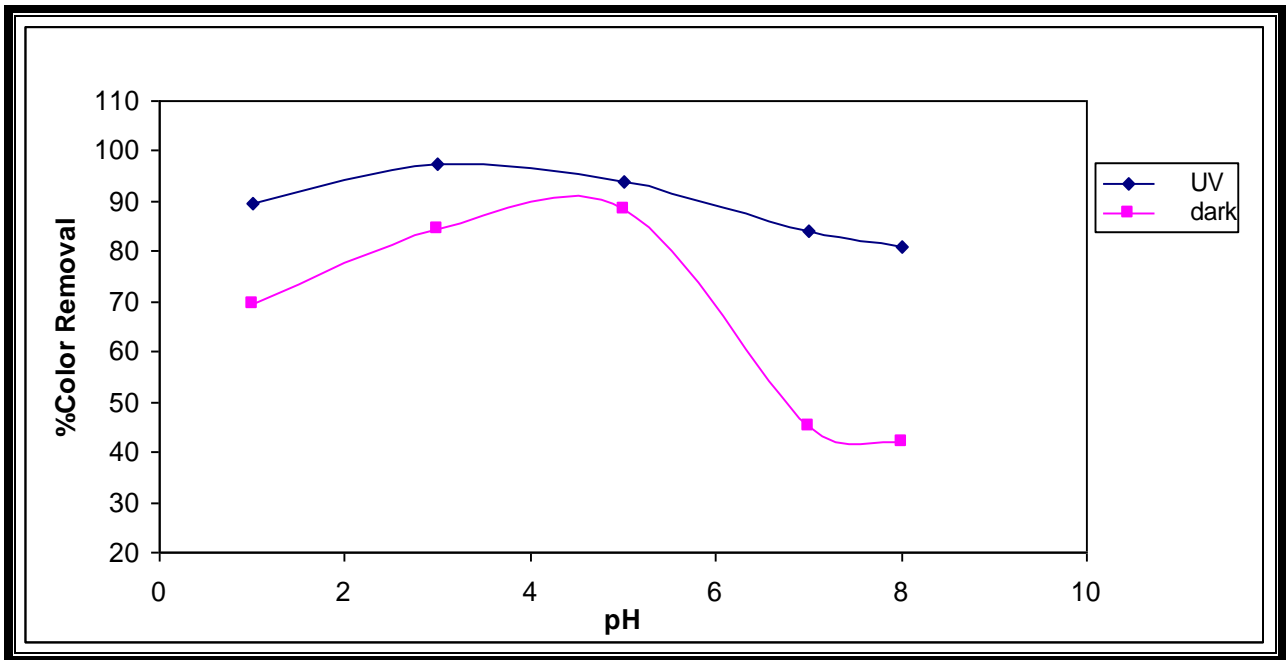


Figure (11): Effect of H₂O₂/Fe⁺² system on the degradation of dye. [TB]=3x10⁻⁵ M, [Fe⁺²]=100mg/L, [H₂O₂]=100mg/L at T=293K.

5. Effect of Temperature

The effect of temperature on the dark reaction rate of TB dye in temperature range of (290 -308 K) , as shown in Fig(12) at pH=6 , for 75 minutes . The degradation efficiencies were about 36.6% under UV light at 308K and 1.5% at 308K in dark . It is indicated that azo-dye degradation efficiency increased with increasing temperature in the reaction. The Arrhenius equation may be used to describe the relationship between rate constants and temperature:

$$k = A e^{(-E_a/RT)} \dots\dots\dots(8)$$

where:

k : rate constant.

A : frequency factor.

E_a : activation energy.

R : ideal gas constant.

T : the absolute temperature.

The estimated activation energy for the degradation of TB is approximately 49.86 KJ/mol under UV light, 59.86 KJ/mol in dark. High temperature either increases the generation rate of ·OH or directly affects the reaction rate with the dye molecule. Given that the overall rate seems to be depended on availability of ·OH, that the higher temperature reduced the

activation energy for the hydrogen peroxide reaction to hydroxyl radicals. The difference between activation energy values in dark and photo is due to fact that UV light enhanced the generation of $\cdot\text{OH}$ and reducing the energy demands in decolorization process from dye solution.

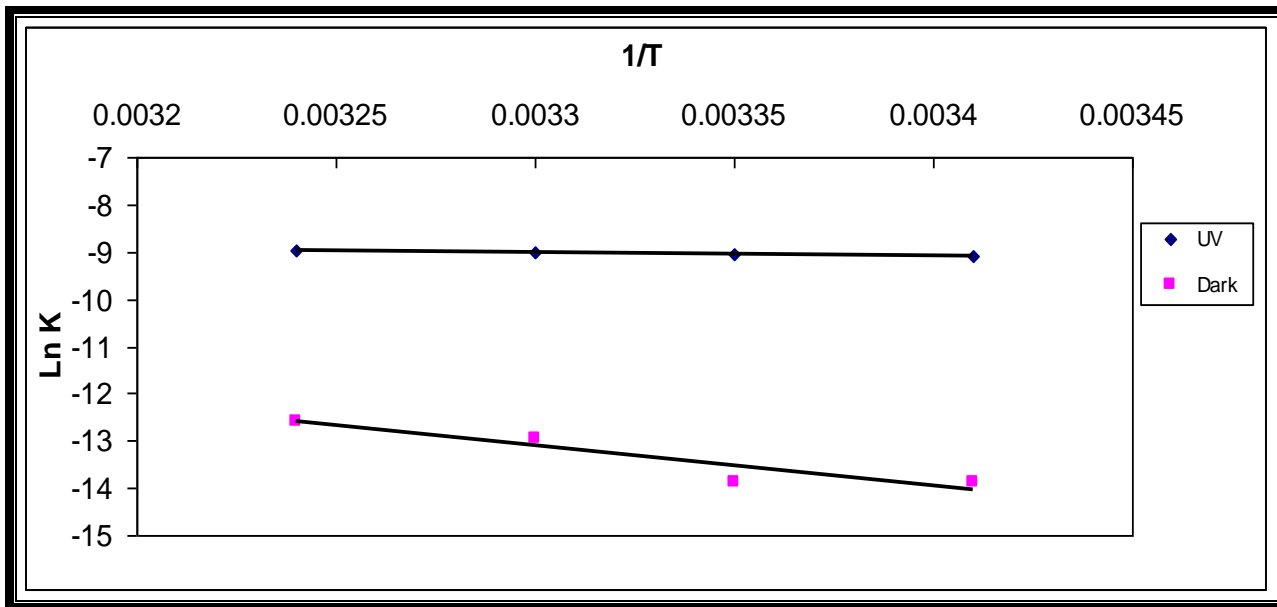
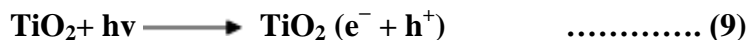


Fig (12): Effect of different temperature on the degradation of dye.[TB]= 3×10^{-5} M, pH=6

6. Effect of TiO_2

The efficiency of degradation of TB dye by TiO_2 in absence of UV radiation is shown in Fig(13) at pH=6 . A maximum degradation efficiency of 2.6% was attained at the residence time of 75 min. This low degradation efficiency could be due to the absence of light source. Activated by light source, TiO_2 has been reported to be capable of degrading various organic pollutants completely according to the following reaction⁽¹⁾ :



Where:

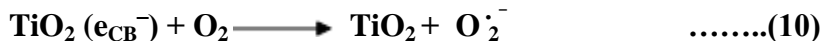
$h\nu$ = radiation energy (mW-sec)

e^- = electron

h^+ = hole

The higher color removal under UV light was 46% by used 1gm/L of TiO_2 . The factor which effect on the degradation efficiencies used TiO_2 included : quantity of TiO_2 , dye

concentration, intensity of UV source used and reactor configuration. The degradation efficiency process increase as the photocatalyst TiO₂ concentration is increase. In the photocatalytic reaction where U.V irradiation is used, electrons in the semiconductor are excited from the valence band to the conduction band leaving positive holes in the valence band. The electrons in the conduction band react with the adsorbed oxygen molecules to form O₂⁻ species, while the positive holes react with the adsorbed hydroxyl ions to form hydroxyl radicals. These processes could be represented in the following equations⁽⁶⁾ :



The highly reactive hydroxyl radicals oxidize the dye molecules as follows:

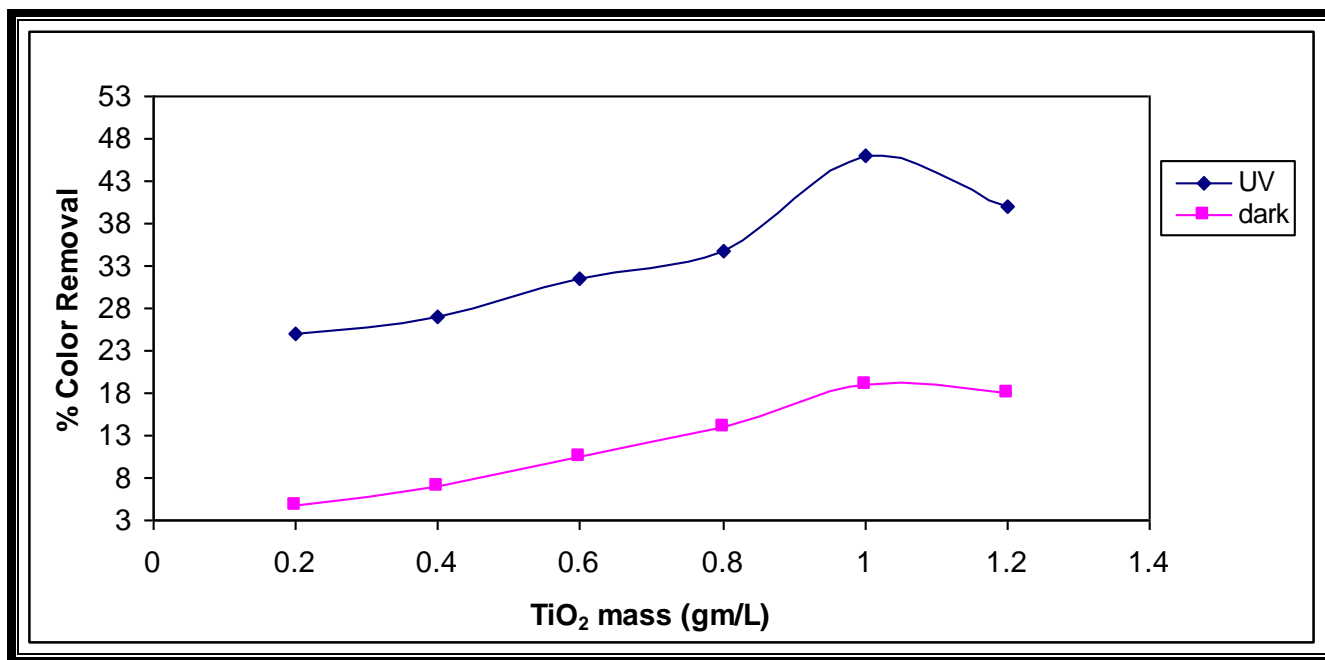
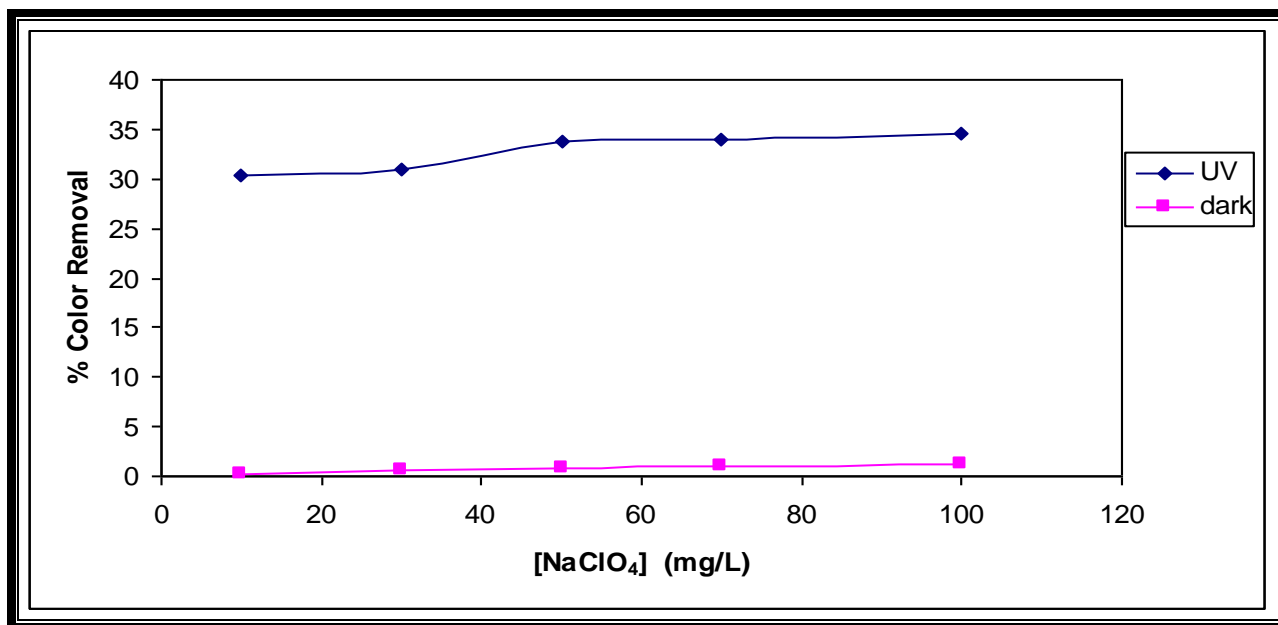
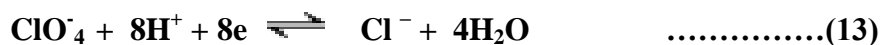


Fig (13): Effect of different TiO₂ mass on the degradation of dye.[TB]=3x10⁻⁵ M, pH=6 at T=293K.

7. Effect of initial sodium perchlorate concentration

The effect of varying initial concentration of NaClO₄ in the range e (10 - 100 mg/L) was investigated on the degradation of TB dye when dye concentration is 3x10⁻⁵ M , pH=6 at

T=293K . Fig (14) shows the results . Higher color removal efficiency was found 34.6% for 100mg/L of NaClO₄ under UV light and 1.16% for 100mg/L in dark . Results show the degradation efficiencies increase with increase of NaClO₄ concentration . As the oxidation state of the chlorine is +7 , the species is a strong oxidizing agent (eq. 13) . In this respect, perchlorate is slightly weaker than dichromate or permanganate. How - ever, perchlorate reduction is extremely no labile (slow) and can usually be observed only in concentrated strong acid Complexes of perchlorate are rare and usually note - worthy when they are encountered . Perchlorate does not sorbs well to most surfaces and most perchlorate salts are quite soluble (30) .



Fig(14): Effect of different NaClO₄ concentration on the degradation of dye.[TB]= 3x10⁻⁵M ,pH=6 at T=293K.

4. Conclusions

1. The degradation was strongly influenced by various parameters, particularly the initial H₂O₂ dosage, dye concentration, pH, temperature as well as irradiation time.
2. The photooxidation of TB by using the UV/H₂O₂ is more effective in an acidic medium with the initial dosage of H₂O₂ ranging between 100-500 mg /L.
3. Higher efficiencies of color removal in presence of Fenton's reagent was achieved at pH media (3,5) ,and high efficiencies were observed by increasing the concentration of Fe⁺² .

4. The photodegradation of the TB dye catalyzed by UV/TiO₂ was carried out with good results, Titanium dioxide could be used powerfully in photocatalytic degradation of textile industrial wastewater, where the extent of degradation was achieved about 46 % by using 1gm/L of TiO₂ at pH=6,T=293K after 75 min. of irradiations.
5. The photodegradation of the TB dye using NaClO₄ concentration is more effective in high concentration of NaClO₄ in presence UV light.
6. Faster degradation kinetics and higher color removal efficiency was observed at higher temperature 308K.

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ايجاد الظروف المثلى لتحطيم صبغة أزرق تربيان باستخدام طرائق الأكسدة المتقدمة والتحفيز الضوئي
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الخلاصة

تستخدم عمليات الأكسدة المتقدمة (AOP 'S) لتجزئة أو إزالة لون الأصباغ الموجودة في مياه التصريف الصناعية التي تطلق من مصانع الانسجة بانواعها المختلفة. إن مصدر الأشعة فوق البنفسجية المستخدم هو مصباح زنبقي واطى الضغط (قدرته ٦ واط ويبعث عند طول موجي 253.7 نانومتر). هذه الدراسة تضمنت دراسة تأثير تركيز الصبغة الابتدائي ، جرعة بيروكسيد الهيدروجين المستخدمة ، pH المحلول ، كاشف فنتون ، درجة الحرارة ، بيركلورات الصوديوم والتحفيز الضوئي باستخدام TiO_2 لمعرفة الظروف الأمثل لعمليات المعالجة في (الظلام والضوء) . التجزئة التامة تحققت في فترة زمنية قصيرة نسبيا لمدة ٧٥ دقيقة من التشعيع . وقد تحققت أسرع إزالة للون عند pH منخفضة وعند درجة حرارة عالية . وقد وجد إن سرعة الإزالة تزداد بزيادة تركيز بيروكسيد الهيدروجين H_2O_2 المستخدم . أن التجزئة الضوئية باستخدام نظام فنتون $UV/ H_2O_2 / Fe^{2+}$ هي الأسرع حدوثاً . أفضل نتائج تم الحصول عليها باستخدام نظام فنتون الضوئي بكفاءة إزالة عالية أكبر من ٩٨% عند pH=6 ، بوجود $[H_2O_2] = 100$ ملغم/لتر ، $[Fe^{2+}] = 100$ ملغم/لتر . تم استخدام المطياف لغرض قياس تركيز الصبغة غير المتحطمة وذلك عند امتصاص محلولها المائي الأعظم ($\lambda_{max} = 590$ نانومتر) . أوضحت نتائج التجارب الحركية إن تفاعل التحطم الضوئي بتأثير الأشعة فوق البنفسجية هو من الدرجة الأولى . أوضحت نتائج التجزئة الضوئية المحفزة أن سرعة تفاعل التجزئة يعتمد على وزن TiO_2 وأفضل نسبة إزالة ٤٦% باستخدام ١ غم/لتر من TiO_2 .