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Study of chemical oxygen demand (COD) and Removal of azure B dye from aqueous solution by using Advanced Oxidation Processes

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Abstract

Decolorization of azure B dye was studied using UV/H_2O_2, H_2O_2/Fe^{2+} and $UV/H_2O_2/Fe^{+2}$ processes. The effect of initial dye concentration, , pH, Fenton reagent ,temperature , initial hydrogen peroxide dosage, and irradiation time was studied .It has been found that the removal rate increased as the initial concentration of H_2O_2 and ferrous ion increase to optimum value. The degradation is fast with $UV/H_2O_2/Fe^{2+}$ system. The best results were obtained from photo Fenton's reagent with efficiency more than 99% at pH = 6 , $[H_2O_2]$ = 500mg/L , $[Fe^{2+}]$= 150mg/L . The concentration of undegraded dye was detected by using parameters at λ_{max}=646.5nm.In present study the effect of all above effects on chemical oxygen demand(COD), was studied. The results showed that the removal rate were increased as the initial concentration of H_2O_2 and ferrous ion increased. The decolourization reaction was found to follow , first order kinetics with respect to the dye concentration.

1- Introduction

The environmental risks by effluents of textile wastewater industry are the major source of water and ground water pollution. Advanced Oxidation Processes (AOP_S) refer to a set of chemical treatment techniques procedures designed to remove organic and inorganic materials in water and waste water by oxidation (1). The advantages of AOP_S are their high treatment efficiencies ,fast reaction rates, hence relatively small reactors, high flexibility removal and the possibility of using them in to water recycling processes(2-5). AOP_S processes involve the generation of hydroxyl radicals ($^{\cdot}OH$) , the oxidation potential of this radical is 2.80v making it the most powerful oxidizing agent after fluorine (3.03v)(6). The hydroxyl radical can oxidize a broad variety of organic substances such as dyes, pesticide, insecticide, etc.(7-12). The oxidation of organic compounds in water with AOP_S usually produces oxygenated organic products and low molecular weight acids that more biodegradable (13,14).Over the last few years the tendency has been carried out chemical oxidation in the presence of catalyst that serves as a generator of hydroxyl radicals . Most dyes can be easily treated if the conventional treatment methods are incorporated with the advanced oxidation processes which can break the complex structure of the dye and make it more a minable to bio-degradation (15). The study of effect mixing H_2O_2 include irradiation of dye solution in presence of H_2O_2 which cause the generate of $^{\cdot}OH$ radicals. Ultraviolet radiation is used to cleave the (O-O) bond in hydrogen peroxide and generate the hydroxyl radical(16).The reactions describing UV/H_2O_2 processes are presented below(17) :

$H_2O_2 + h\upsilon \rightarrow 2\,HO\bullet$ (1)

$H_2O_2 + HO\bullet \rightarrow HO_2\bullet + H_2O$ (2)

$HO\bullet + HO_2\bullet \rightarrow H_2O + O_2$ (3)

Fenton's reagent , a mixture of ferrous iron (catalyst) and hydrogen peroxide (oxidizing agent) ,has been known as a powerful oxidant for organic contaminants. The mechanism of the Fenton process is reported below(18-19) .

$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^- + HO^{\cdot}$ (4)

$Fe^{2+} + HO^{\cdot} \rightarrow Fe^{3+} + OH^-$ (5)

$HO^{\cdot} + RH \rightarrow H_2O + R^{\cdot}$ (6)

$R\bullet + Fe^{3+} \rightarrow R+ + Fe^{2+}$ (7)

Photo Fenton reaction gives faster rates and higher degrees of mineralization comparing to conventional Fenton process[20] . Photo Fenton prefer Fenton process because this reaction can be driven by low energy photons and reduce the operational cost of the treatment[21]. The COD, which is a measure of the concentration of all compounds that can be oxidized by the $Cr_2O_7^{2-}$ anion in acidic media, gives a measure of degradation of pollutants and produced intermediates during the dye degradation[22]. These risks include highly colored compounds, high level of COD , wide range of pH natural, and resistance to the natural degradation. The kinetics of COD and color removal were consistent with those of H_2O_2 and Fe^{2+} dosage, respectively. Higher dosage of $Fe^{2+}$was found to be significant on the initial removal rate of COD whereas the color was favorably removed by increasing UV power[23].The present study was carried out to investigate the removal of azure B dye Figure(1) using processes and various reaction conditions UV/H_2O_2 and $UV/H_2O_2/Fe^{+2}$ involved initial dye concentration, temperature ,initial H_2O_2 concentration, initial ferrous ion concentration, initial pH ,and irradiation time.

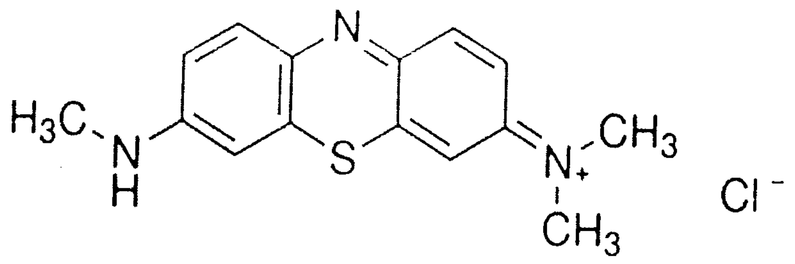


Figure (1): structural formula of azure B dye.

2- Materials and methods

2-1-Cemicals and Reagents

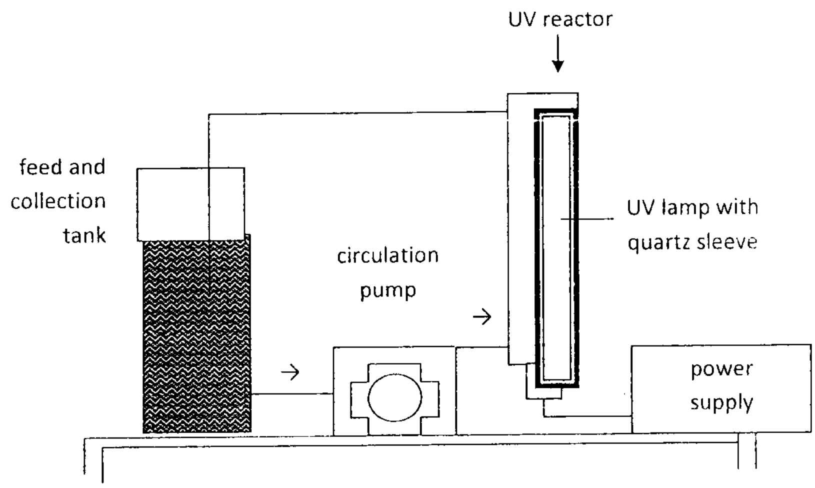
All chemicals were used without further purification. Hydrogen peroxide (H_2O_2 30% w/v), ferrous chloride ($FeCl_2$), sodium hydroxide ($NaOH$) and hydrochloric acid(HCl) and sulphuric acid H_2SO_4 were supplied from BDH. Azure A dye(product of USA.MSDS) was purchased from Omega. . All the other chemicals and solutions were prepared with double distilled water.

2-2-Instruments

UV-Visible 1650 spectrophotometer (Shimadzu ,Japan)was used to recording the absorption spectra of aqueous solutions of dye. UV-Visible 7804C spectrophotometer (Sunny, China)was used to measured of absorbance of dye solutions at (646.5nm).The pH was measured by using microprocessor pH meter 211, (Hanna ,Romania) instruments. The Temperature was adjusted by used regulator water bath WB(Optima). Chemical oxygen demand (COD) was measured using.(Lovibond, Vario LR) and a Thermoreactor TR 300 (Merck, Germany).

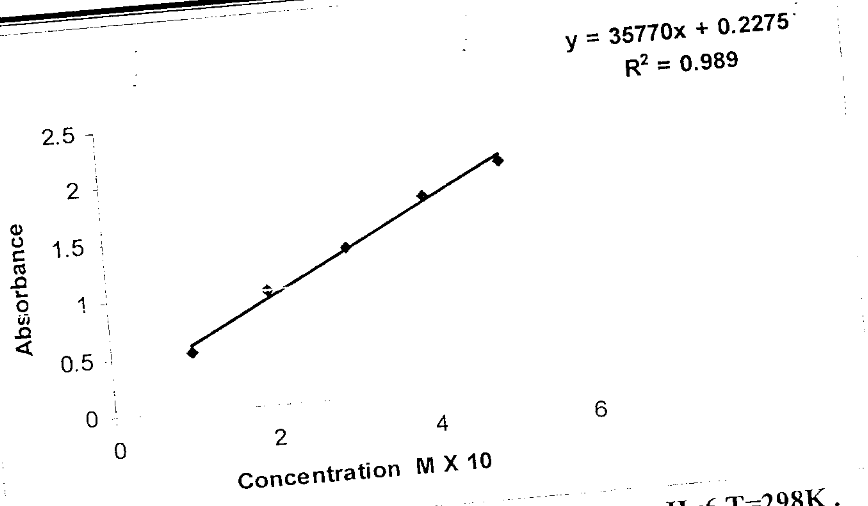
2-3- Photoreactor setup

The Photoreactor shown in Figure (2), was fitted with a fixed low-pressure mercury lamp (6W with the light emitted at 253.7nm). The UV lamp was putted in the side of reactor and the quartz sleeve was enclosed. The Photoreactor was fitted with a regulator water bath to maintain the temperature and a 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.



2-4-Experimental procedures

The operating irradiation time for all experiments were fixed at 75 minutes ,due to the primary experiments indicated that the most of dye molecules are degraded and the dye solution become colorless at the time near to this period. The pH was adjusted to the desired value using 0.1N of sodium hydroxide and hydrochloric acid(24). Chemical oxygen demand (COD) was measured according to the Standard Methods(25). Control experiments were carried out under UV irradiation without H_2O_2 in the solutions and with H_2O_2 but no irradiation. In all experiment, the lamp was warming on for 10 min prior to initiation of reaction. Determination of dye concentration was carried out by using the calibration curve as shown in Figure(3).The absorbance of dye was measured at maximum absorption at=646.5nm.as shown in Figure(4).



Figure(3):Calibration curve for azure B dye at pH=6,T=298K .

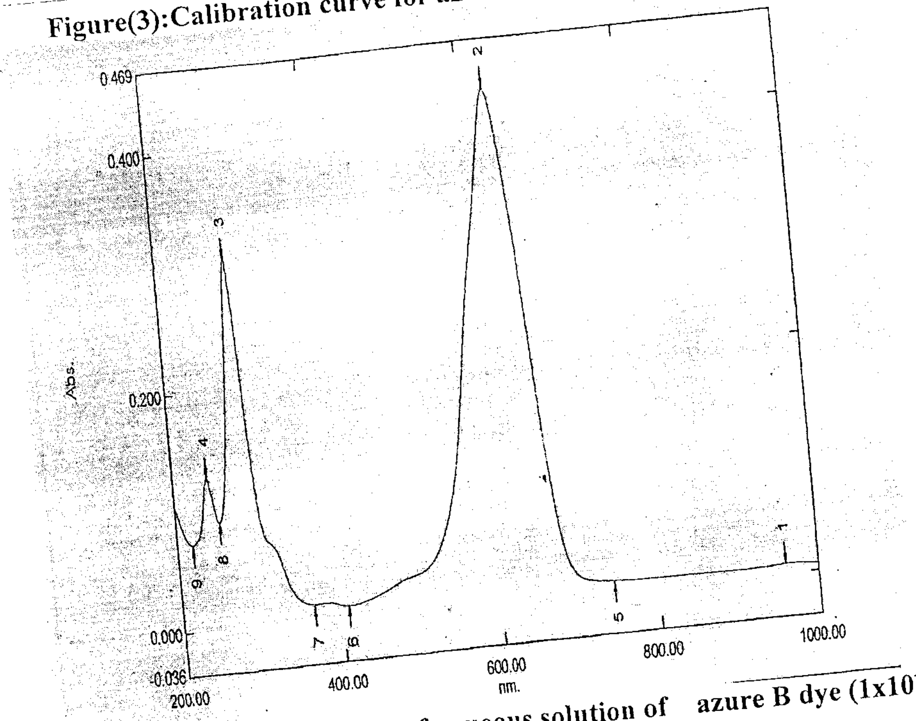


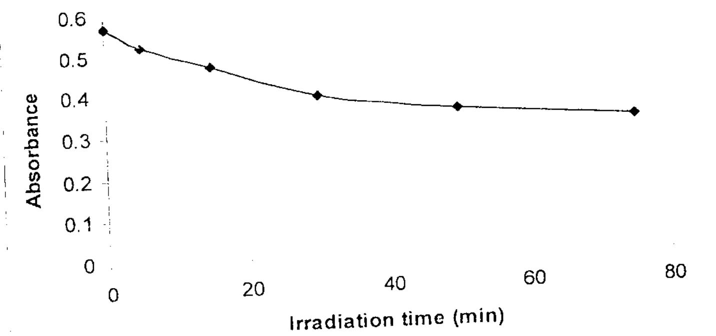
Figure (4): UV-Visible spectrum of aqueous solution of azure B dye ($1x10^{-5}$ M), pH=6, T=298K .

3- Results and Discussion:-

1- UV/ H_2O_2 Method.

1-1- Effect of initial dye concentration .

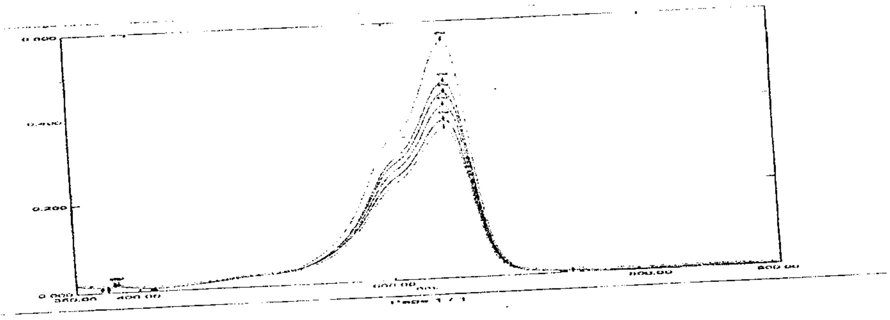
Various initial dye concentrations in the range ($1x10^{-5}$-$5x10^{-5}$M) were exposured to UV irradiation. It has found that increasing in the initial concentration of dye leads to decreasing the COD and color removal , because of decreasing penetration of photons entering into the solution and lowering the formation of hydroxyl free radicals [26]. The results are show in Figure(5).



Figure(5): The absorption of dye as a function of irradiation time at ($1x 10^{-5}$ M), pH=6 ,T=298K .

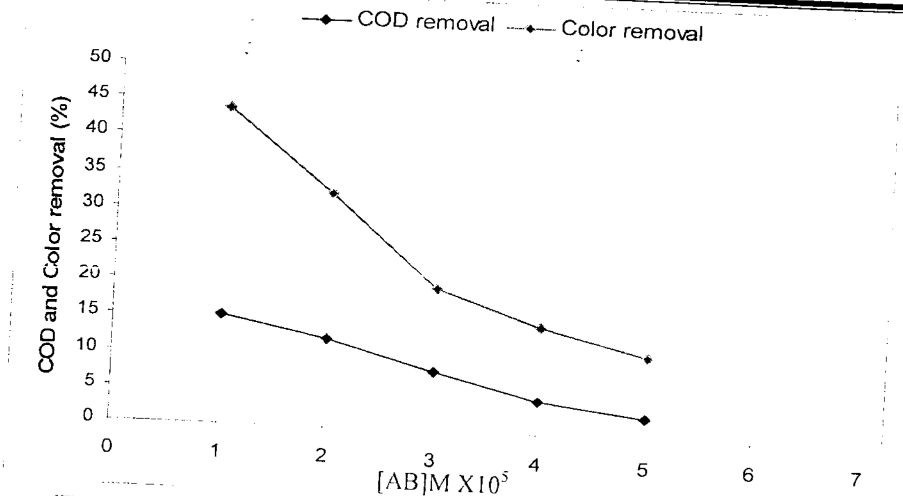
Also , all spectrum of dye decreases as the irradiation time increasing as shown in Figure(6) .

The percent of color removal decreases from 43% to 12% with increasing the



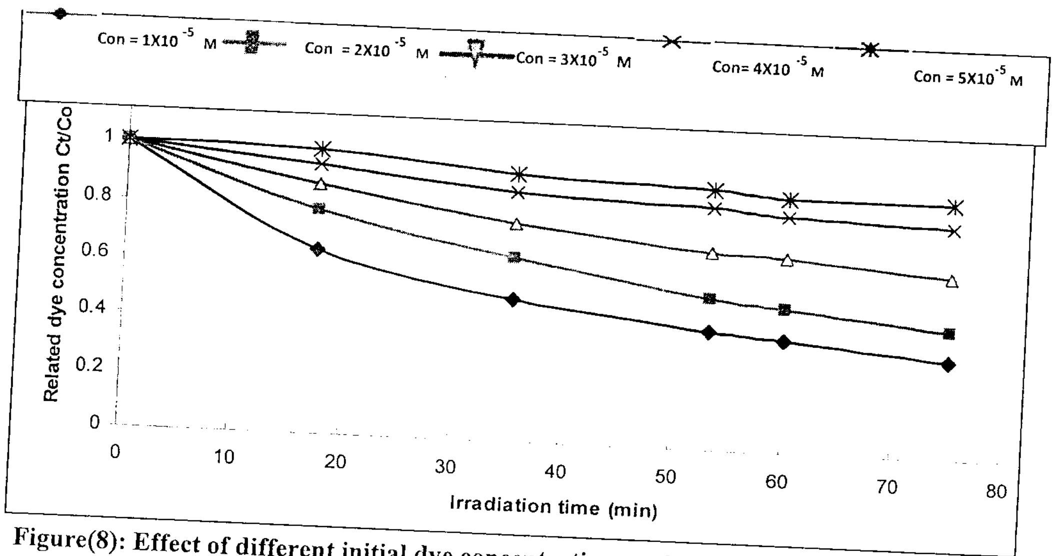
Figure(6):The UV-visible spectra of dye solution($1x10^{-5}$M) as a function of irradiation time at pH=6, T=298K.

concentration of dye in the above range ,and the results proved that the highest percentage of COD removal was obtained at the concentration $1X10^{-5}$M while , the lowest percentage was obtained at the concentration $5x10^{-5}$M. The results are shown in Figure(7).



Figure(7):Effect of dye concentration on COD and color removal of azure B dye at pH=6 ,T=298K .

Different initial dye concentrations were used in the test and observed the effect on azure B color removal by using UV method. The results are shown in Figure(8). 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[27] .



Figure(8): Effect of different initial dye concentration on the color removal of azure B dye at pH=6,T=298K .

The obtained results proved that the photooxidation reactions of the azure B dye are reactions of first order with respect to dye concentration , the coincident of the rapidity of reaction can be related with the absorption of the dye and can be calculated by using the law is called empirical method[28]. Figure(9) shows relationship between Log R and Log C to determining order reaction. Also, the order of reaction was calculated by

1- Effect of H_2O_2 by using the low

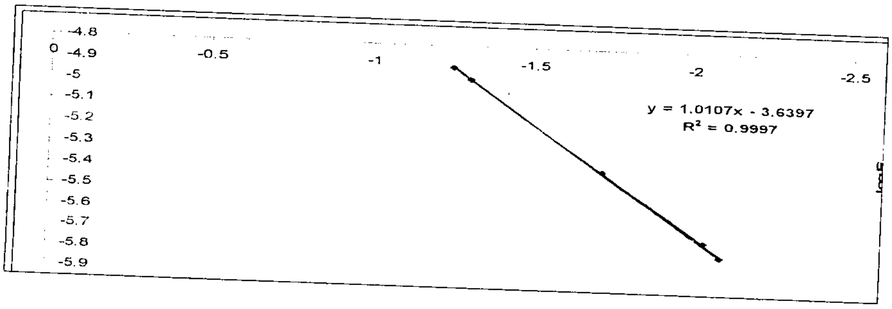
$Rate_1 / Rate_2 = k\,[Dye]_1^x\,[H_2O_2]_1^Y / k\,[Dye]_2^x\,[H_2O_2]_2^Y$ (8)

lead to this order x equal (0.8) from azure B dye .

$LogR = logk + nlogC$ (9)

Where:

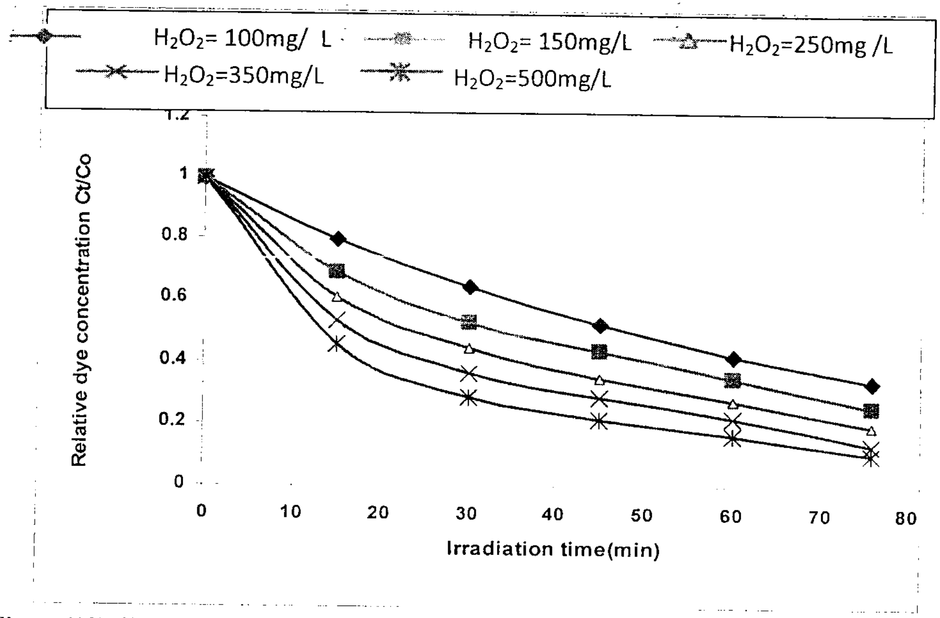
C: Concent ration of dye, n: order of reaction, R: reaction rate, k: reaction rate constant.



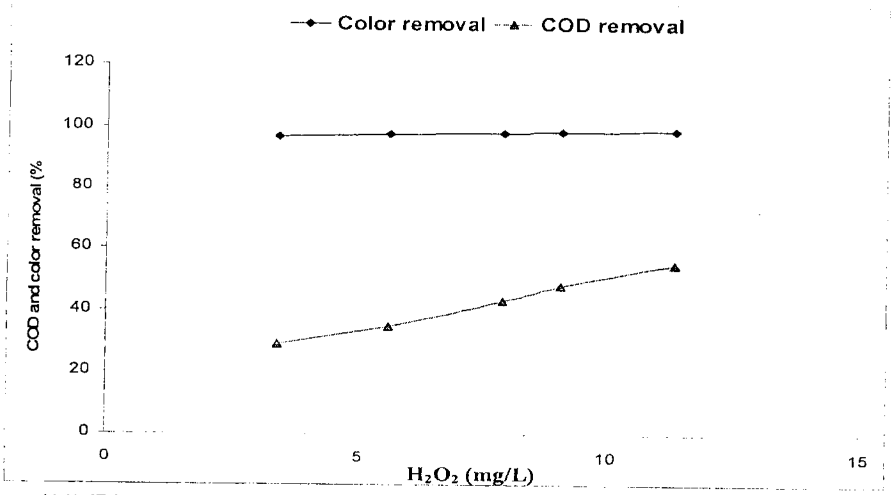
Figure(9): Relationship between Log R and Log C of oxidation of azure B dye =1x10^{-5} M , pH=6, T=298K .

1-2- Effect of initial Hydrogen peroxide concentration.

In this study the effect of different concentrations of H_2O_2 (100-500mg/L) on the decolourization rate was studied . Figure(10) shows the decolourization of azure B as a function of UV irradiation time for various initial H_2O_2 dosages. Color removal is decreased over time in the presence of UV / H_2O_2 light. The results show that the higher color removal in presence light was 96.4% for 500mg/L of H_2O_2 and the ratio decreased from 96.4% to 94.2% when H_2O_2 concentration was reduced from 500 to 100mg/L . Also , the effect of H_2O_2 concentration on COD removal was investigated . It is found that the value of COD removal increases with increasing of the hydrogen peroxide concentration [29] .The results are shown in Figure (11).



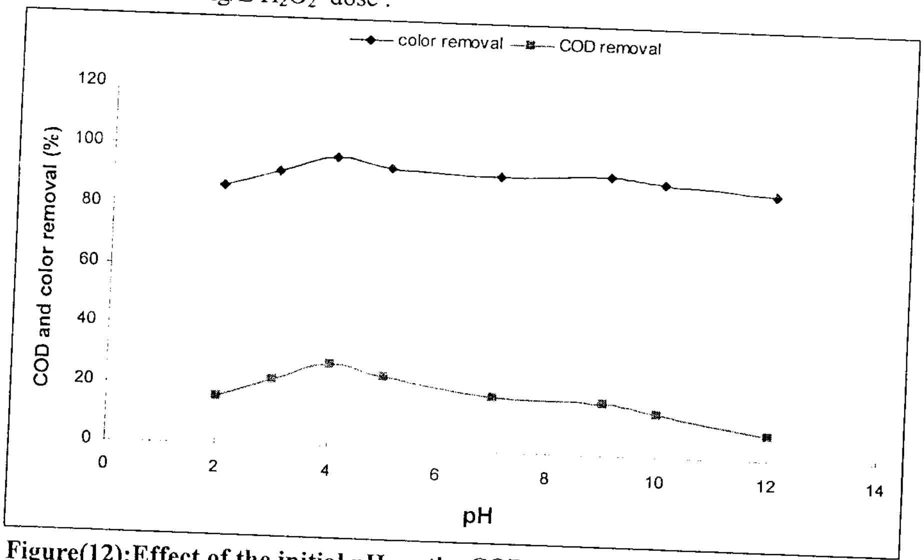
Figure(10): Effect of different initial H_2O_2 concentration on the of azure B dye $=1x10^{-5}$ M, pH= 6, T=298 K .



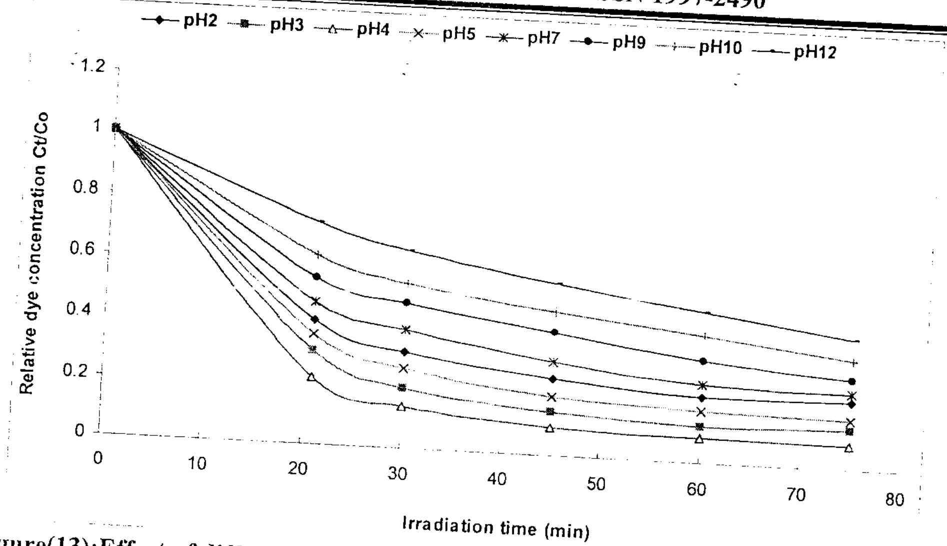
Figure(11):Effect of different initial H_2O_2concentration on COD and color removal of azure B dye $=1x10^{-5}$M, pH=6 at T=298K .

1-3- Effect of initial pH

The effect of pH was evaluated at pH media (2,3,4,5,7,9,10 and 12) . From Figure(12), the results deduced that the high color removal was 97.7% under acidic medium at pH= 4 and low value was obtained in basic media was 87.3%. The increase of the rate of removing the color in low pH value due to more ˙OH radical generation changes in the structure of the molecule because of having a free hydrogen atom which makes the dye molecule exposed for attack by the hydroxyl radical in acidic conditions [30] . Hydrogen peroxide undergoes decomposition in alkaline medium .The removal was low because the generation of HO˙ slowed down and decomposition of oxygen and water rather than producing hydroxyl free radicals under UV irradiation[31]. Figure (13) shows the gradient of relative dye concentration Ct/Co under increasing of irradiation time. The experiments are conducted at $1x10^{-5}$ M dye concentration in the presence of 500mg/L H_2O_2 dose .



Figure(12):Effect of the initial pH on the COD and color removal from azure B dye=$1x10^{-5}$M,[H_2O_2]=(500mg/L), T=298K.



Figure(13):Effect of different pH values on color removal of azure B dye as function of irradiation time, $1X10^{-5}$M , $[H_2O_2]$=(500mg/L), T=298K .

2- Fenton's system

2-1- Effect of initial Ferrous ion concentration

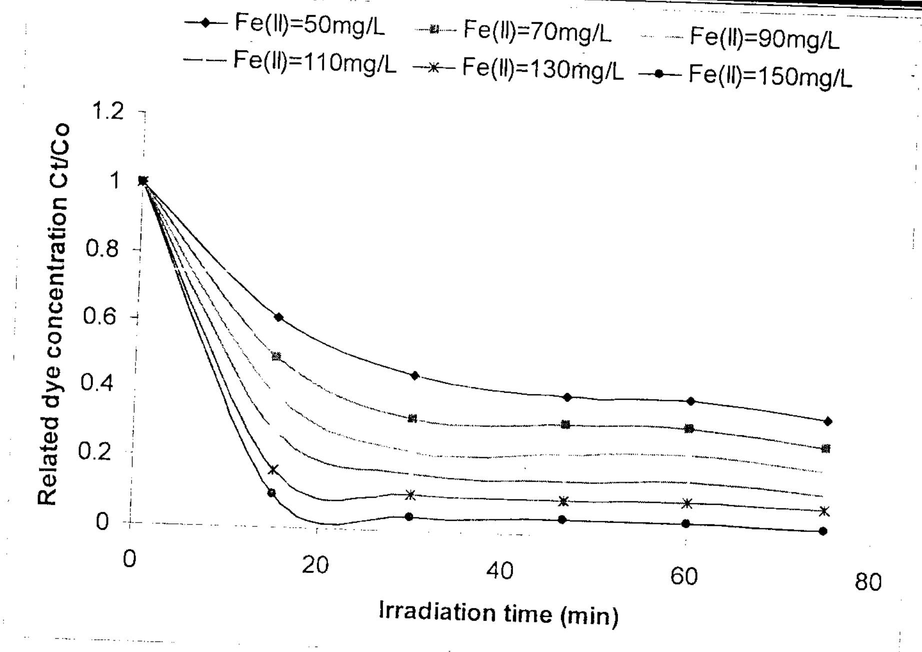
The effect of Fenton reagent on dye azure B removal was studied concentration under the same condition by using various concentration of ferrous ion in the range 50- 150m g/L in the presence of fixed concentration of H_2O_2 (500mg/L). Figure(14) shows the decolourization of azure B as a function of UV irradiation time for various concentration of ferrous ion. In this study, the effect of Fe^{+2} dosage in the Photo-Fenton oxidation process on the COD and color removal as shown in Figure(15), higher dosages of Fe^{+2} lead to a high level of COD removal because a large amount of Fe^{+2} can promote the formation of ·OH .

Also, the order of reaction was calculated by:

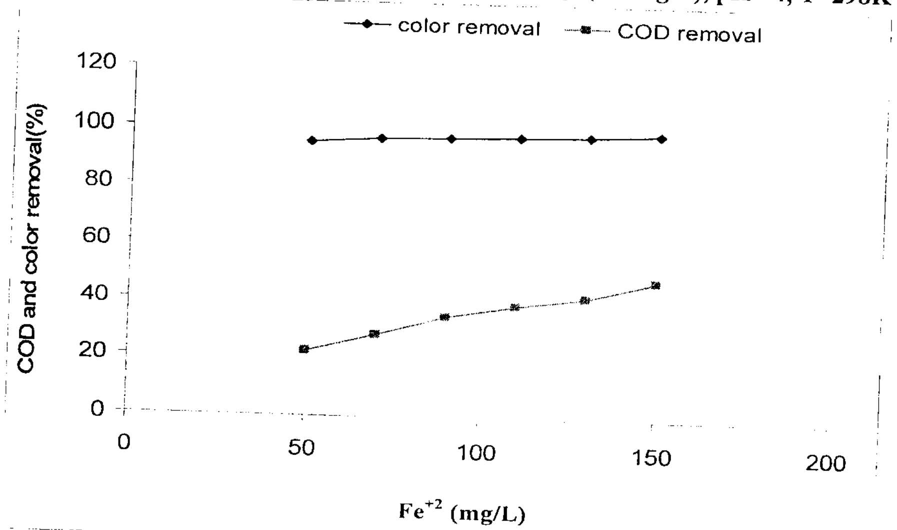
2- Effect of Fe^{+2} /H_2O_2 by using the low

$$Rate_1/ Rate_2 = k\,[Dye]_1^x\,[Fe^{+2}]_1^z\,[H_2O_2]_1^Y / k\,[Dye]_2^x\,[Fe^{+2}]_2^z\,[H_2O_{+2}]_2^Y \qquad (10)$$

lead to this order x equal (1.1) from azure B dye .



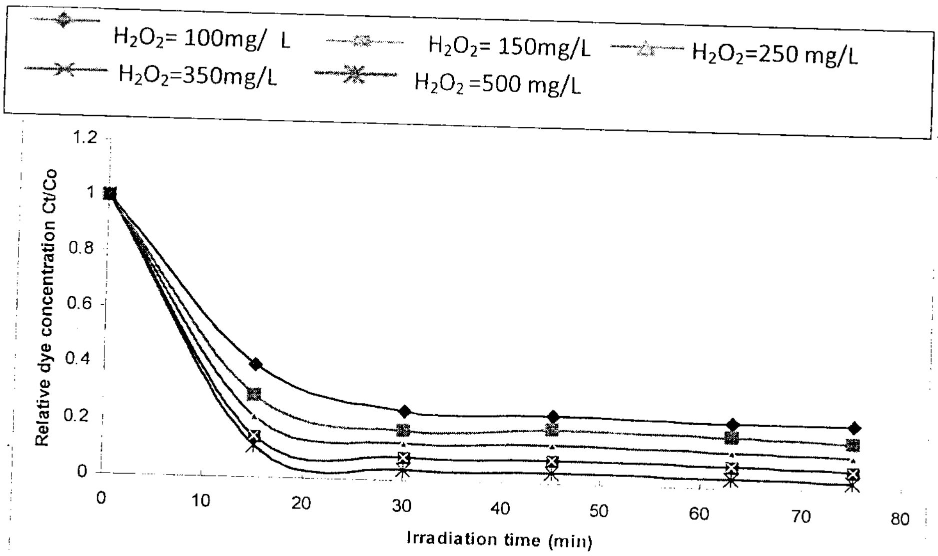
Figure(14): Effect of different Fe^{+2}concentration on the color removal of azure B dye = $1X10^{-5}$M as a function of irradiation time.,$[H_2O_2]$=(500mg/L), pH= 4, T=298K .



Figure(15): Effect of different Fe^{+2}concentration on the COD and color removal of azure B dye = 1X 10^{-5}M ,$[H_2O_2]$=(500mg/L), pH= 4 , T= 298K .

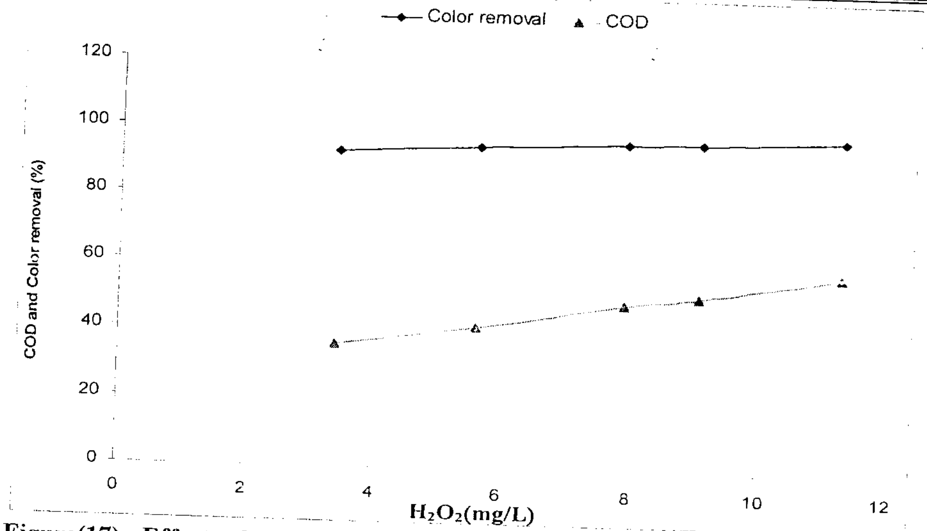
2-2-Effect of initial H_2O_2 concentration

Concentration of hydrogen peroxide has an important role in degradation of azure B dye in Fenton systems . As it observed through experiments that carried out using different concentrations of hydrogen peroxide ranging 100 – 500 mg/L at constant concentration of ferrous ion (150mg/L) , this proves effectiveness of UV irradiation in color removal. The removal percentage of color increased from 97% to 99.1% and the COD increased from 33% to 56% by increasing the H_2O_2 dosages. Figure (16) shows the color removal of azure B as a function of UV irradiation time for various initial H_2O_2 dosages. Figure (17) shows the effect of H_2O_2 on COD and color removal .



Figure(16): Effect of different initial H_2O_2 concentration on the color removal from azure B =$1X10^{-5}$ M, $[Fe^{+2}]$=(150mg/L), ,$[H_2O_2]$=(500mg/L), pH= 4, T=298 K .

The increasing of concentration of hydrogen peroxide effected in the reaction rate , this is obvious through the results ratio of color removal dye azure B of increasing from 97 % to 99.1% under UV light . Therefore as same of hydrogen peroxide increased the ratio of degradation of pollutants increases due to increase the quantity of generated hydroxide radicals and this conforms with many studies(32-34).

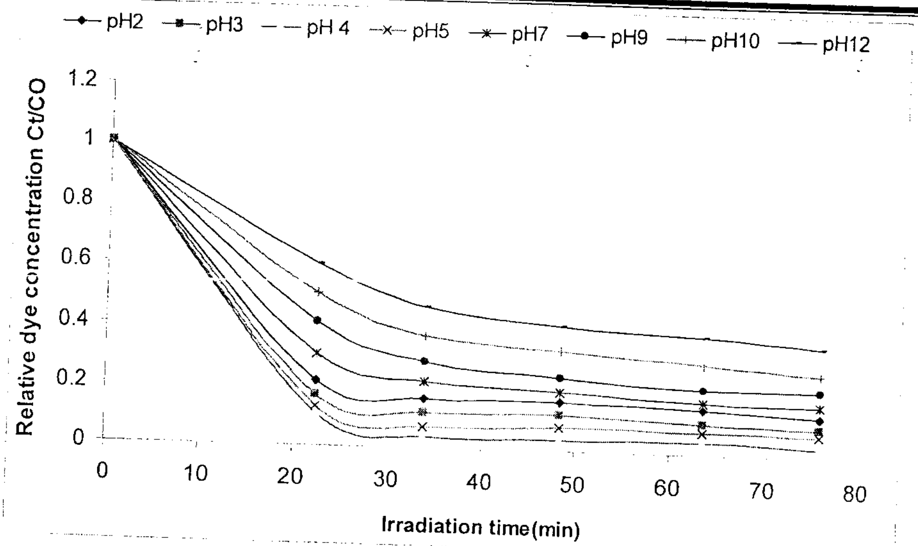


Figure(17): Effect of H_2O_2 on the COD and color removal from azure B dye $=1X10^{-5}M$,$[Fe^{+2}]$=(150mg/L), pH= 4, T=298 K .

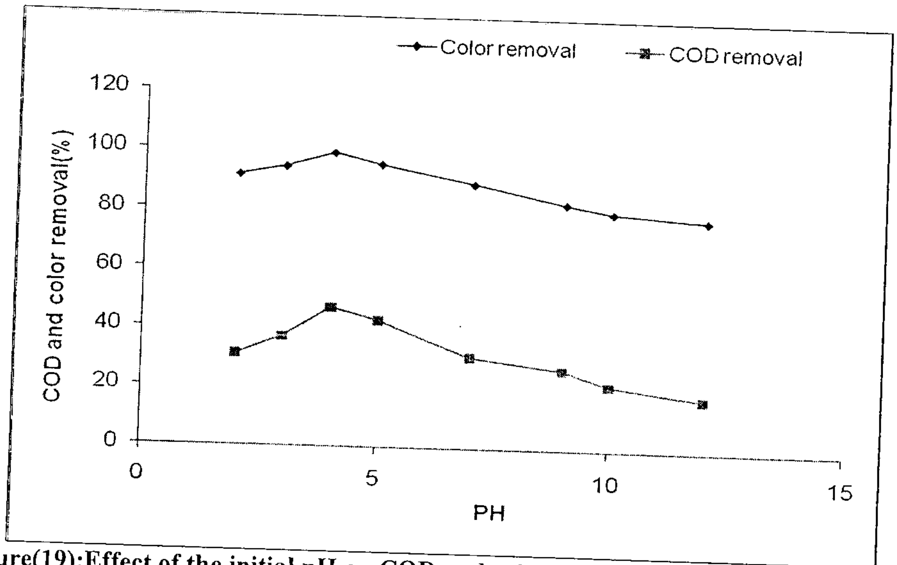
2-3- Effect of initial pH

The results show a clear effect of initial pH in oxidation reaction of azure B dye . The high color removal was obtained under acidic media (due to more ·OH radicals generation) and low color removal in basic conditions (due to $Fe(OH)_3$ formation)[32]. The higher ratio color removal of azure B dye was obtained under acidic media at pH=4 under UV light and decreasing ratio in basic media. The higher decolourization rate demand reduces pH value because of changing in molecular structure. From Figure(18) it has been found that during 75 min of UV irradiation, the relative dye concentration Ct/Co was decreased as the pH value increased .

The COD reached the maximum removal after irradiation of dye for 75 minutes of dye and was at the initial pH value equal of 4 . In basic media the removal was low because the generation of ·OH slowed down due to the decomposition of H_2O_2 to H_2O and O_2. This pH was consistent with same previous works[35-36]. Figure (19) show effect of initial pH on COD and color removal.



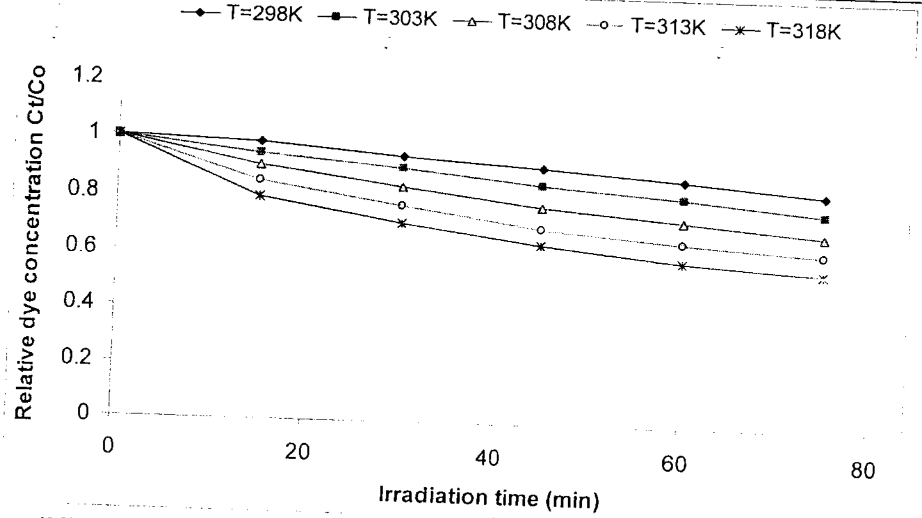
Figure(18):Effect of different pH value on color removal from azure B dye as function of irradiation time, $1X10^{-5}M$, $[Fe^{+2}]$=(150mg/L), pH= 4, T=298 K .



Figure(19):Effect of the initial pH on COD and color removal from azure B dye=$1x10^{-5}M$,$[H_2O_2]$=(500mg/L),$[Fe^{+2}]$=(150mg/L),T=298 K .

3- Effect of Temperature

Temperature is an important kinetic factor on enhancing the color and COD removal percentage . The dye removal rate by using the UV method increased with increasing temperature of the system as shown in Figure (20). The experiments were carried out in a range between 298 to 318k and the results are clarified in the Figure (21). In this work, it is that the value of COD removal increases with the increase of the temperature because a decrease in the oxygen amount demanding for oxidation.

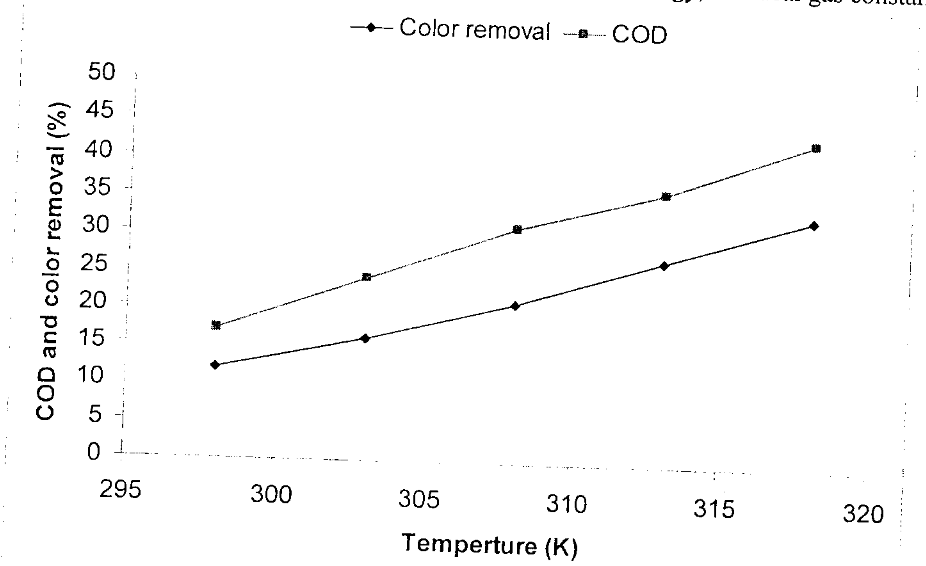


Figure(20): Effect of different temperature on the color removal of azure B dye =$1x10^{-5}$M using UV method at pH=6.

The Arrhenius equation is used to describe the relationship between rate constants and temperature and draw this the relationship as shown in Figure (22)

$$k = A\,e^{(-Ea/RT)} \qquad \text{...........(11)}$$

where: k : rate constant, A : frequency factor, Ea : activation energy, R : ideal gas constant.



Figure(21): Effect of the temperature degree on color removal and COD of azure B dye =$1X10^{-5}$ M at pH=6.

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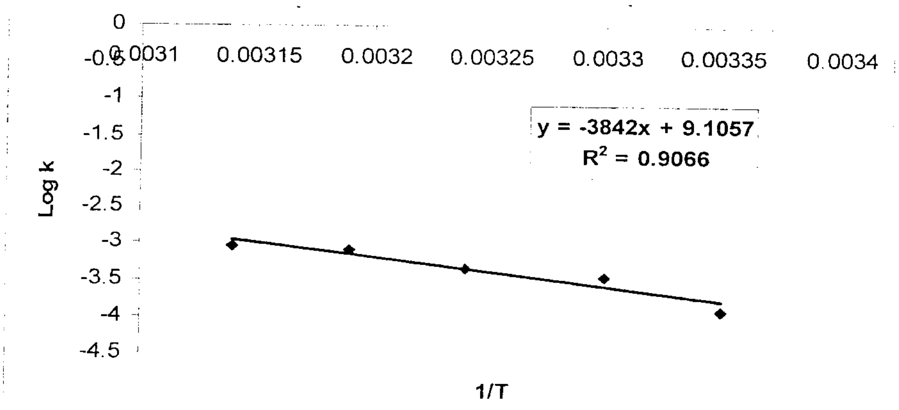

Figure(22): Arrhenius plot of color removal of dye.[AB]= $1x10^{-5}$ M at pH=6.

The activation energy calculated from plot was equal to 74.16 KJ/mole with the presence of light .

4- Conclusions

1. The degradation was strongly influenced by various parameters, particularly the initial H_2O_2 dosage, dye concentration, pH, temperature as well as irradiation time.
2. The photooxidation of azure B dye by using the UV/H_2O_2 is high effective in an acidic medium with the initial dosage of H_2O_2 ranging between 100-500 mg /L.
3. Higher efficiencies of color removal and COD removal in presence of Fenton's reagent were achieved at pH media (3,4) ,and high efficiencies were observed by increasing the concentration of Fe^{+2} .
4. The speed of degradation rate and efficiency of COD removal are high in temperature 308K.

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دراسة المتطلب الكيميائي للأوكسجين وإزالة صبغة الازور B من المحاليل المائية باستخدام طرائق الأكسدة المتقدمة بوجود الأشعة فوق البنفسجية

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الخلاصة

تمت دراسة إزالة صبغة الازور B باستعمال عمليات الأكسدة المتقدمة التي تتضمن UV, UV/H_2O_2, $/H_2O_2/Fe^{+2}$. تضمنت هذه الدراسة تأثير تركيز الصبغة الابتدائي ، pH المحلول ، كاشف فنتون ، ودرجة الحرارة ،جرعة بيرو كسيد الهيدروجين المستخدمة وزمن التشعيع على سرعة إزالة اللون . وجد إن سرعة الإزالة تزداد بزيادة تركيز بيرو كسيد الهيدروجين وايون الحديدوز للوصول إلى القيمة المثلى. أن التجزئة الضوئية باستعمال $UV/H_2O_2/Fe^{+2}$ أسرع والنتائج أفضل حيث نحصل على كفاءة أكثر من 99% عند pH= 4 عندما يكون $[H_2O_2$]=500mg/L, $[Fe^{+2}$= 150mg/L]، . تم تحديد الطول الموجي لصبغة الازور B باستعمال جهاز Spectrophotometer وكان الطول الموجي الأعظم يساوي 646.5nm. في هذا البحث تم دراسة جميع التأثيرات المشار إليه أعلاه على الحاجة الكيميائية للأوكسجين(COD). أوضحت نتائج التجارب الحركية إن تفاعل التحطم الضوئي بتأثير الأشعة فوق البنفسجية هو من الدرجة الأولى بوجود الصبغة فقط .