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## **COD and Color Mineralization of Azure C dye Using UV/ClO<sub>2</sub> technique**

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### **Abstract**

This research is to investigation of the efficacy of UV in combination with chlorine dioxide (UV/ClO<sub>2</sub>) for decolorizing the azure C dye in aqueous solution. The (UV/ClO<sub>2</sub>) is the one of the most importance methods for water pollution treatment. The objective was to determine the best treatment for reducing of color. The parameter studied were the effect of initial dye concentration [A.C], temperature (T), pH, gas flow rate (f) and light intensity (I<sub>0</sub>). Complete degradation was achieved in the relatively short time of 60 minutes irradiation Fast decolourization was achieved at high pH and high temperature. The best results were obtained at pH=8 with efficiency more than 97.7%. The concentration of undegraded dye was detected by use spectrophotometer at  $\lambda_{\text{max}}=611.5\text{nm}$ . In present study the effect of all above effects on chemical oxygen demand COD. Were studied the decolourization reaction was found to follow a first order kinetics with respect to the dye concentration.

## دراسة COD وإزالة صبغة الأزور C باستخدام تقنية (UV / ClO<sub>2</sub>)

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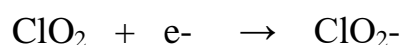
### الخلاصة

تحرى هذا البحث فعالية الأشعة فوق البنفسجية (UV) عند اتحادها مع غاز ثنائي أوكسيد الكلور (ClO<sub>2</sub>) في إزالة صبغة الأزور C من المحلول المائي، تعد تقنية (UV/ClO<sub>2</sub>) واحده من أهم الطرق المستخدمة لمعالجة تلوث المياه، وكان الهدف هو تحديد الظروف المثلى في اختزال اللون . تضمنت هذه الدراسة تأثير تركيز صبغة الأزور C ، درجة الحرارة ، الدالة الحامضية ، سرعة انسياب الغاز، شدة الضوء وزمن التشعيع . تحققت التجزئة التامة في فترة زمنية قصيرة نسبيا (٦٠ دقيقة) من التشعيع وأسرع إزالة للون عند دالة حامضية و درجة حرارة عاليتين. وأفضل نتيجة تم الحصول عليها عند دالة حامضية مساوية إلى (8) مع نسبة إزالة 97.7%. تم تحديد الطول الموجي لصبغة الأزور C باستعمال جهاز Spectrophotometer وكان الطول الموجي الأعظم يساوي 611.5nm . وفي هذا البحث تم دراسة جميع التأثيرات المشار إليها أعلاه على الحاجة الكيميائية للأوكسجين (COD). وقد تمت دراسة حركية التفاعل لإزالة الصبغة وأوضحت النتائج إن سرعة الإزالة تتبع حركيات المرتبة الأولى بالنسبة لتركيز الصبغة .

## 1- Introduction

Generally all human activities need clean water. Many parts of the world, increase the demand on water <sup>(1)</sup>. The World Bank estimates (17–20) % of industrial water pollution comes from textile dyeing and treatment <sup>(2)</sup>. This effect pay attention of the scientists to the socio – economic and political dimensions <sup>(3)</sup>. The fact of the pollution that accompanies the dyeing industry is primarily due to generate a high volume of waste water <sup>(4)</sup> nature of the dyes along with the strong presence of toxic metals, acid, alkali <sup>(5)</sup> and strong colour <sup>(6)</sup>. There are more than 100,000 commercially available dyes and pigments, and the total world organic colorant product is over  $7 \times 10^7$  tons/year <sup>(7,8)</sup>. 10 - 15 % of these dyes are released as effluent during the dyeing processes <sup>(9)</sup>. To protect the aquatic environment, many methods such as adsorption, advance chemical oxidation, nanofiltration and chemical coagulation followed by sedimentation were used to remove dyes from waste water <sup>(10)</sup>. Specifically, Advance Oxidation Processes (AOP's) involving hydroxyl radical <sup>(11)</sup>, are introduced to treat textile dyes effluents <sup>12</sup>. Hydroxyl radicals ( $\text{OH}\cdot$ ) has the highest oxidation potential equal to ( $E^0=2.8$  eV) after fluorine radical <sup>(13)</sup>. 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 <sup>(14)</sup>. Advance oxidation processes (AOP's) include UV/ $\text{H}_2\text{O}_2$ , UV/ $\text{O}_3$ ,  $\text{O}_3/\text{H}_2\text{O}_2$ , Fenton's reagent, and the wet-air oxidation processes <sup>(15)</sup>. The concept behind an (AOP's) is that exposure of a strong oxidizing agent exposed to UV light generates hydroxyl free radicals even stronger oxidants <sup>(16)</sup>. Advance oxidation processes are better treatment options than the conventional treatment methods commonly adopted in wastewater treatment plants <sup>(17)</sup>. Ultraviolet irradiation following chlorine dioxide (UV/ $\text{ClO}_2$ ) is the one of the most importance methods for water pollution treatment <sup>(15)</sup>. Chlorine dioxide is commonly used as a oxidant and primary disinfectant during treatment of drinking water <sup>(18,19)</sup>. It is also used for taste-and-odor control and color removal. In the USA, over 500 water treatment plants use  $\text{ClO}_2$  24/7 (hour/day) and as many 900 use it either part time or seasonally <sup>(20)</sup>. The most common methods for the generation of  $\text{ClO}_2$  for water treatment involve sodium chlorite ( $\text{NaClO}_2$ ) <sup>(20)</sup>. Chloride dioxide is less expensive

than ozone. Chlorine dioxide is a strong selective oxidizer and is used in the pulp bleaching process <sup>(22,23)</sup>. It has ability due to unique one-electron exchange mechanisms. Chlorine dioxide attaches the electron-rich centers of organic molecules <sup>(24,25)</sup>. Chlorine dioxide is its high solubility in water, particularly in chilled water. Chlorine dioxide does not hydrolyze in water but remains a highly soluble gas above 11<sup>0</sup>C over abroad range of pH (2.0-10.0) <sup>(26)</sup>. Aqueous solutions must be protect from light. The main reaction product of chlorite ion  $\text{ClO}_2^-$  in water is the chlorite ion  $\text{ClO}_2^-$  <sup>(27,28)</sup>.



The present study was carried out to investigate the removal of azure C dye Figure 1 from aqueous solution by using (UV/ $\text{ClO}_2$ ) technique and various reaction conditions involved initial dye concentration, temperature, initial pH, light intensity and gas flow rate.

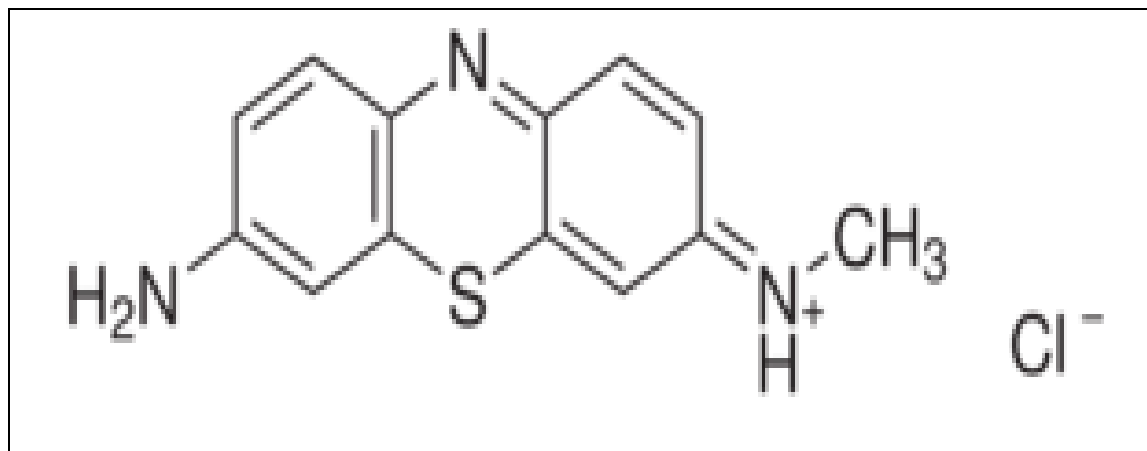


Figure (1): Structural formula of azure C dye

## 2- Materials and Methods

### 1. Materials

Sodium hydroxide (NaOH), Hydrochloric acid (HCl), sulphuric acid ( $\text{H}_2\text{SO}_4$ ) and sodium chlorite  $\text{NaClO}_2$  were supplied from B.D.H. Azure C dye (product of

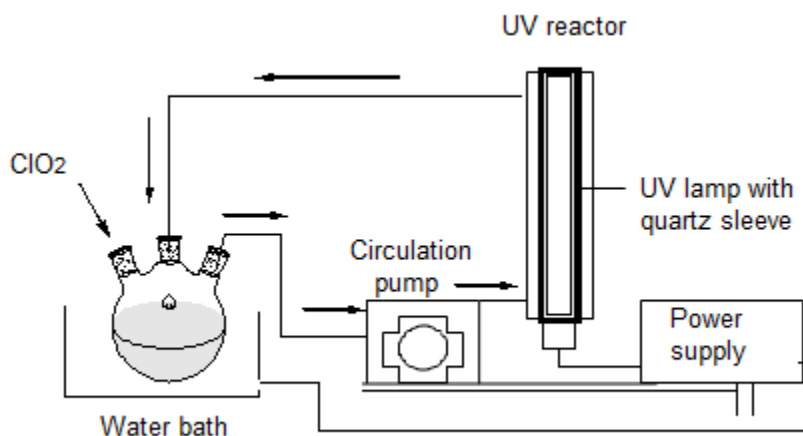
USA.MSDS) was purchased from Omega. All the other chemicals and solutions were prepared with double distilled water.

## 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 maximum absorbance of dye solutions at (611.5nm). 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). The instrument of chlorine dioxide preparation is locally collection .

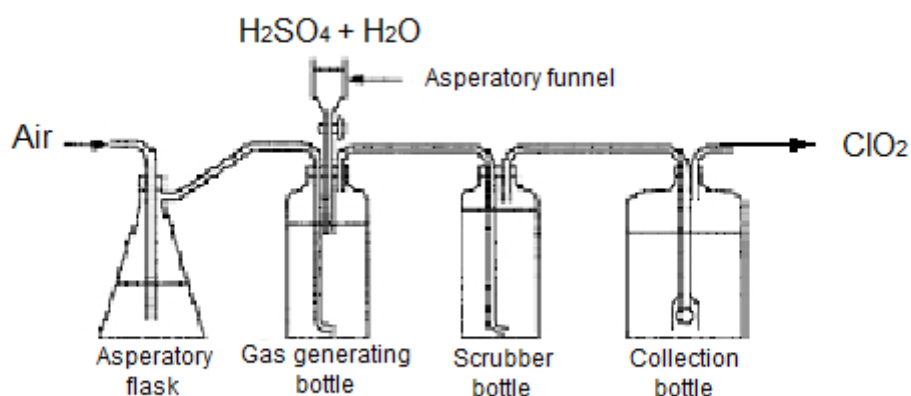
## 3. Photoreactor Setup

The Photoreactor shown in Figure 2 <sup>(29)</sup>, was fitted with a fixed low-pressure mercury lamp (12, 8, 6 and 4W with the light emitted at 254 nm). 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 tri orifice flask. A syringe was fitted with Photoreactor to withdrawn of dye solution samples at meaning time.



**Figure (2): Photoreactor setup****4- Preparation of Chlorine Dioxide**

A pure solution of chlorine dioxide is prepared by slowly adding dilute sulfuric acid to a sodium chlorite solution, removing any contaminants such as chlorine by means of a sodium chlorite scrubber, and passing the gas into distilled water by means of a steady stream of air. The experimental setup to generate the chlorine dioxide is described Figure 3<sup>(30)</sup>.

**Figure (3) : Chlorine dioxide generation and absorption system**

Connect aspirator flask, 500mL capacity, with rubber tubing to a source of purified compressed air. Let air bubble through a layer of 300mL distilled water in flask and then pass through a glass tube ending within 5 mm of the bottom of the 1-L gas-generating bottle . Conduct evolved gas via glass tubing through a scrubber bottle containing saturated  $NaClO_2$  solution and finally, via glass tubing, into a 2-L borosilicate glass collecting bottle where the gas is absorbed in 1500 mL distilled water. Provide an air outlet tube on collecting bottle for escape of air. Select for gas generation a bottle constructed of strong borosilicate glass and having a mouth wide enough to permit insertion of three separate glass tubes: the first leading almost to the bottom for admitting air, The second reaching below the liquid surface for gradual introduction of  $H_2SO_4$ , and the third near the top for exit of evolved gas and air. Fit to second to

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tube graduated cylindrical system separator funnel to contain  $\text{H}_2\text{SO}_4$ . Located this system in a fume hood with an adequate shield. Dissolve 10g  $\text{NaClO}_2$  in 750 mL distilled water and place in generated bottle. Carefully add 2mL conc.  $\text{H}_2\text{SO}_4$  to 18 mL distilled water and mix. Transfer to funnel. Connect flask to generating bottle, generating bottle to scrubber, and the later to collecting bottle. Pass a smooth current of air through the system, as evidenced by the bubbling rate in all bottles. Introduce 5 mL increments of  $\text{H}_2\text{SO}_4$  from funnel into generating bottle at 5min intervals. Continue air flow for 60 minute. The concentration of  $\text{ClO}_2$  thus prepared varies between 250 and 600 mg/L. corresponding to approximately 500 to 1200 mg free chlorine/L.

## 5. Experimental Procedures

Collect photoreactor with the chlorine dioxide generating instrumental, as figure 4. The operating irradiation time for all experiments were fixed at 60min, 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<sup>(31)</sup>. Chemical oxygen demand COD was measured according to the Standard Methods<sup>(32)</sup>. Control experiments were carried out under UV irradiation with  $\text{ClO}_2$  in the solutions. 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 shown in Figure 5. The absorbance of dye was measured at maximum absorption at=611.5nm.as Figure 6 shows.

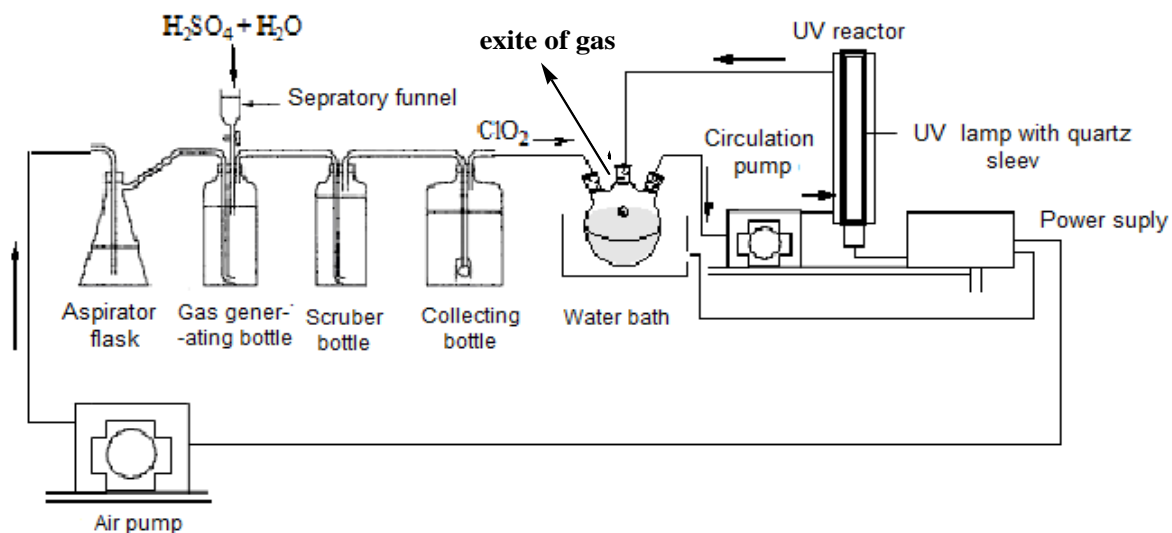


Figure (4): Instrumental for degradation of azure C

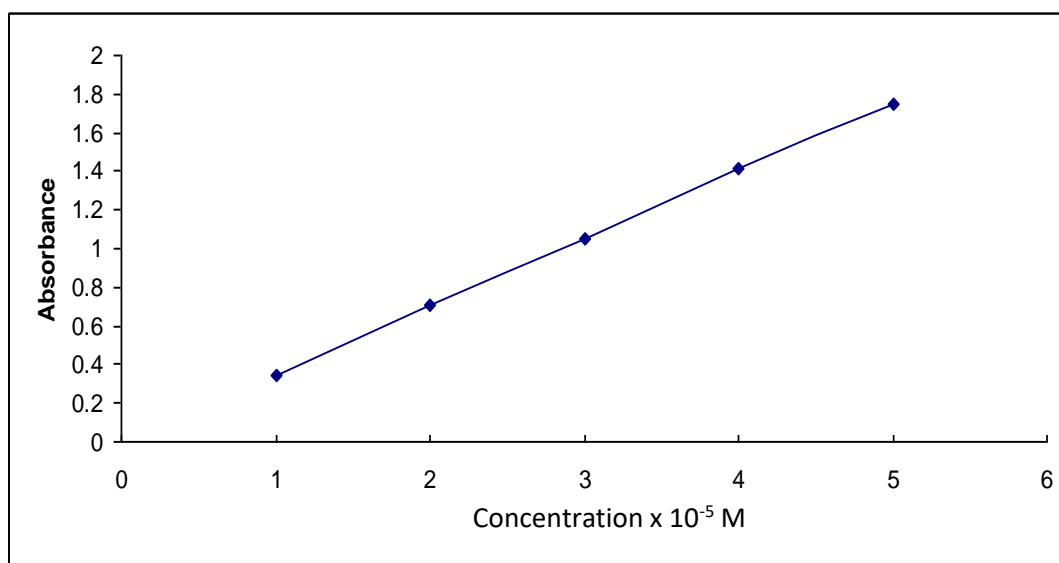
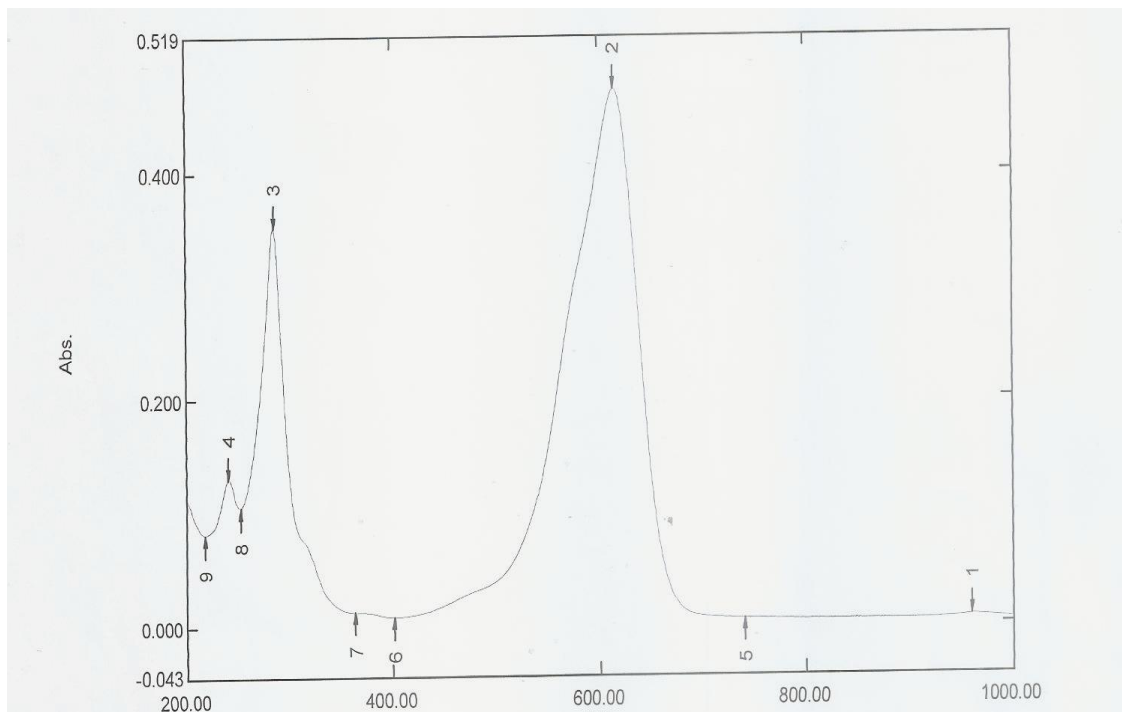


Figure (5): Calibration curve for azure C dye at pH=6, T=298K, I<sub>0</sub>=173.711mW/cm<sup>2</sup> and f= 160 L/h



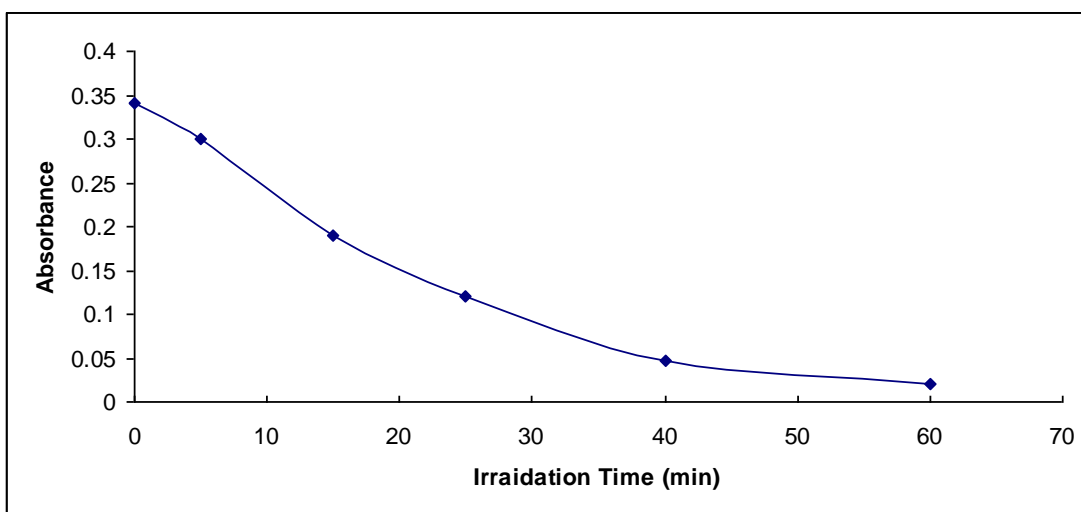


**Figure (6): UV-Visible spectrum of aqueous solution of Azure C dye [A.C]= $1 \times 10^{-5}$  M, pH=6, T=298K,  $I_0=173.711 \text{ mW/cm}^2$  and  $f=160 \text{ L/h}$ .**

## 6- RESULTS AND DISCUSSION

### 1. Effect of Initial Dye Concentration [A.C]

Various initial dye concentration in the range ( $1 \times 10^{-5}$ - $5 \times 10^{-5}$  M) were exposure to (UV/ $\text{ClO}_2$ ). It has found that increasing in the initial concentration of dye lead to decreasing the color removal and COD, because of decreasing penetration of photons entering into the solution and lowering the formation of hydroxyl free radicals<sup>(33)</sup>. In this study show effect concentration of dye on ratio color removal during period different time under range (0-60) min. in presence of (UV/ $\text{ClO}_2$ ) for azure C dye concentration ( $1 \times 10^{-5}$  M), determine absorption at this time. The results in Figure 7.



**Figure (7): Show the maximum absorption of dye as a function of irradiation time at  $\text{pH}=6, T=298\text{k}, I_0=173.711\text{mW}/\text{cm}^2$  and  $f=160\text{ L/h}$ .**

Also the overall spectrum of dye decreases as the irradiation time increasing show as Figure 8 , and the results proved that the highest percentage with COD at the concentration  $1 \times 10^{-5}\text{M}$  while , the lowest percentage with COD at the concentration  $5 \times 10^{-5}\text{M}$ . The results are shown in Figure 9 .

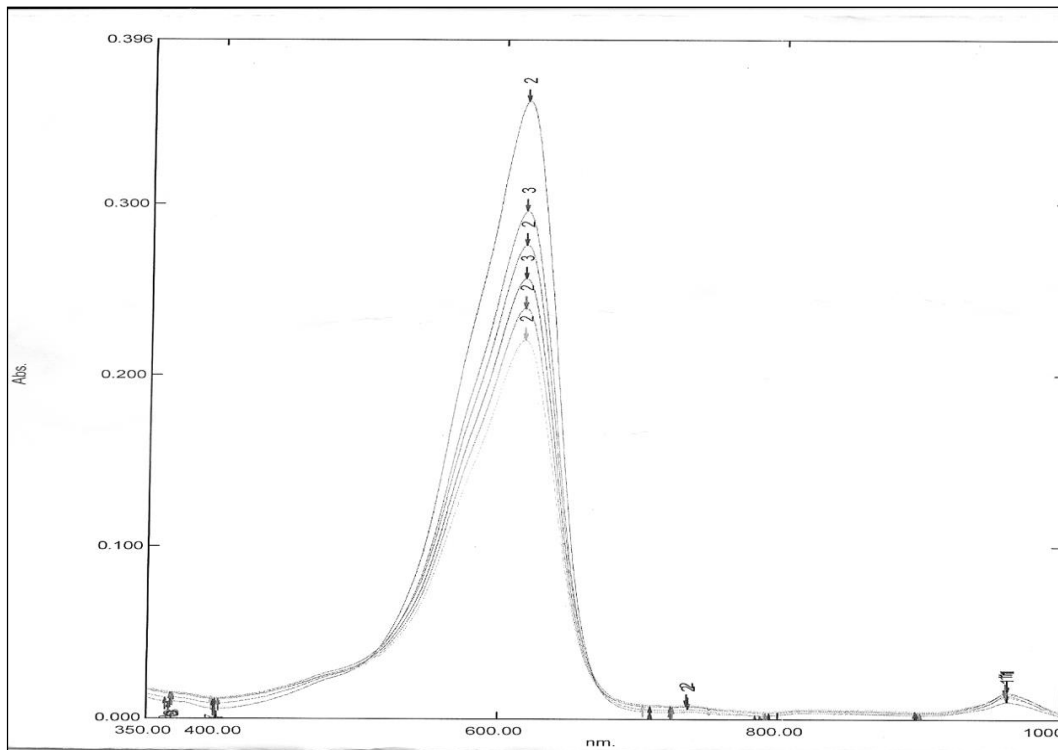
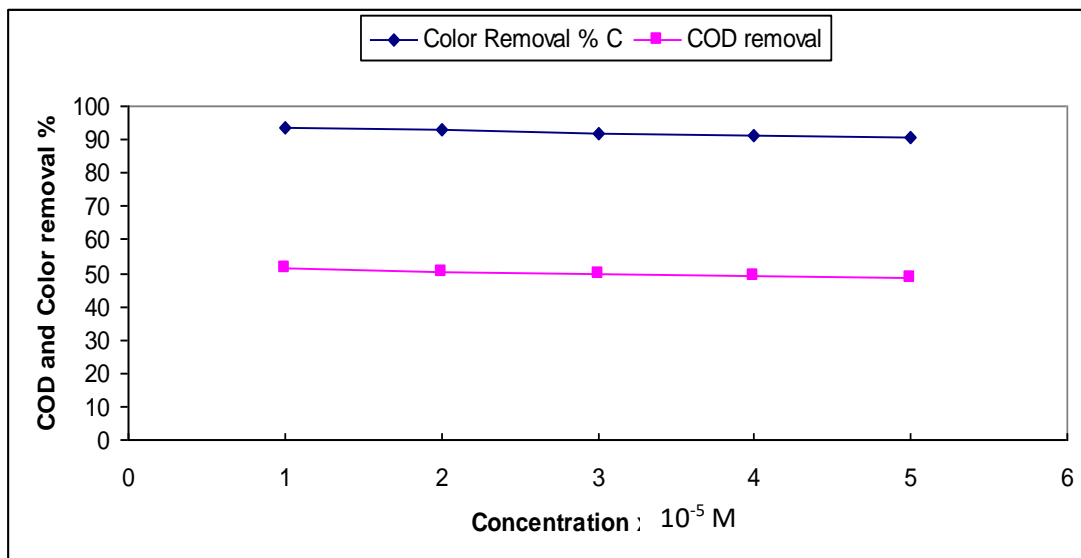
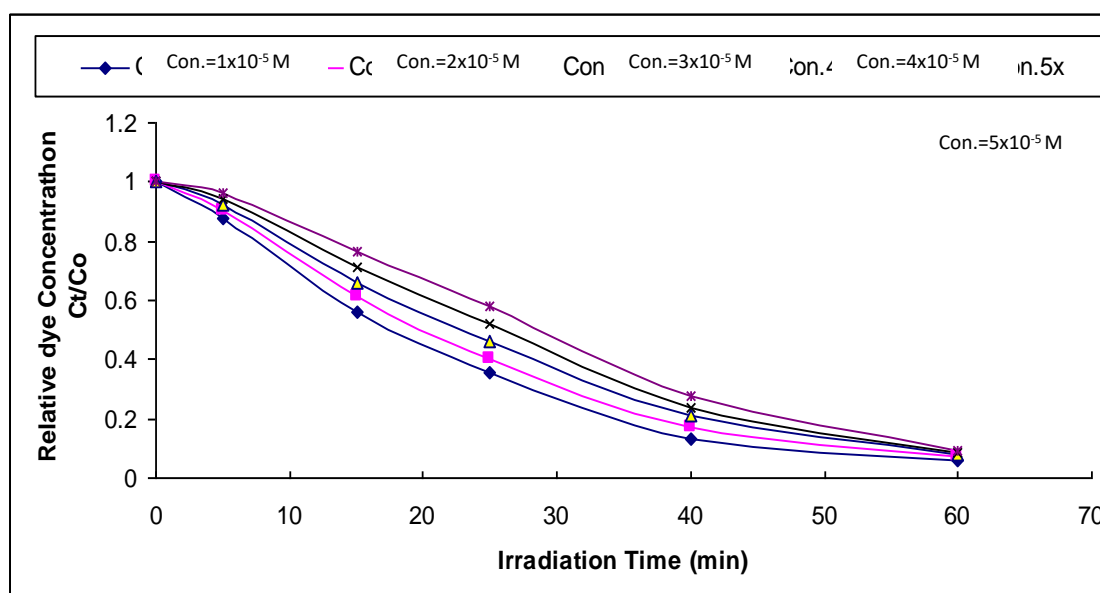


Figure (8): The UV-visible spectra of dye solution ( $1 \times 10^{-5} \text{ M}$ ) as a function of irradiation time at  $\text{pH}=6$ ,  $T=298 \text{ K}$ ,  $I_0=173.711 \text{ mW/cm}^2$  and  $f=160 \text{ L/h}$ .



**Figure (9): Effect of dye concentration on color removal and COD removal of azure C dye at pH=6, T=298bk, I<sub>0</sub>=173.711mW/cm<sup>2</sup> and f=160 L/h .**

Different initial dye concentration were used in the test and observed the fact on azure C color removal by using (UV/ClO<sub>2</sub>). The results are shown in Figure 10. It is clear the higher dye concentration increased the absorption of UV radiation, lead to decreasing the availability if (UV/ClO<sub>2</sub>) and lowering the formation of hydroxyl radicals in the solution.

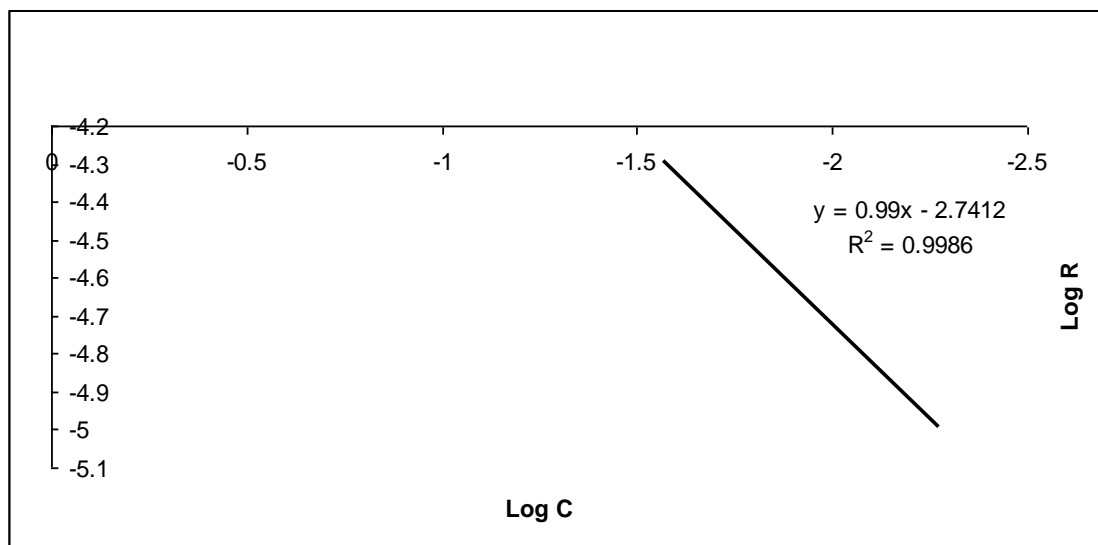


**Figure (10): Effect of different initial dye concentration on the color removal of azure B dye by using UV method at pH=6, T=298k, I=173.711mW/cm<sup>2</sup> and f=160 L/h.**

The obtained results proved that the Photo oxidation reactions of the Azure C dye are reactions of first order. Figure 11 shown the relationship between Log R and Log C to produced order reaction.

$$\text{Log R} = \log k + n \log C \quad (1)$$

Where C: concentration of dye, n: order reaction, R: reaction rate, K: reaction rate constant.



**Figure (11): Relationship between Log R and Log C of oxidation of Azure C dye**  
[A.C] =  $1 \times 10^{-5}$  M, pH=6, T=298k, I=173.711mW/cm<sup>2</sup> and f=160 L/h.

## 2. Effect of Temperature (T).

Temperature is an important kinetic factor on enhancing the color removal and COD percentage. The dye removal rate by using the (UV/ClO<sub>2</sub>) increased with increasing temperature of the system as shown in Figure 12. The experiments were carried out in a range between (298-318k) and the results are clarified in the Figure 13.

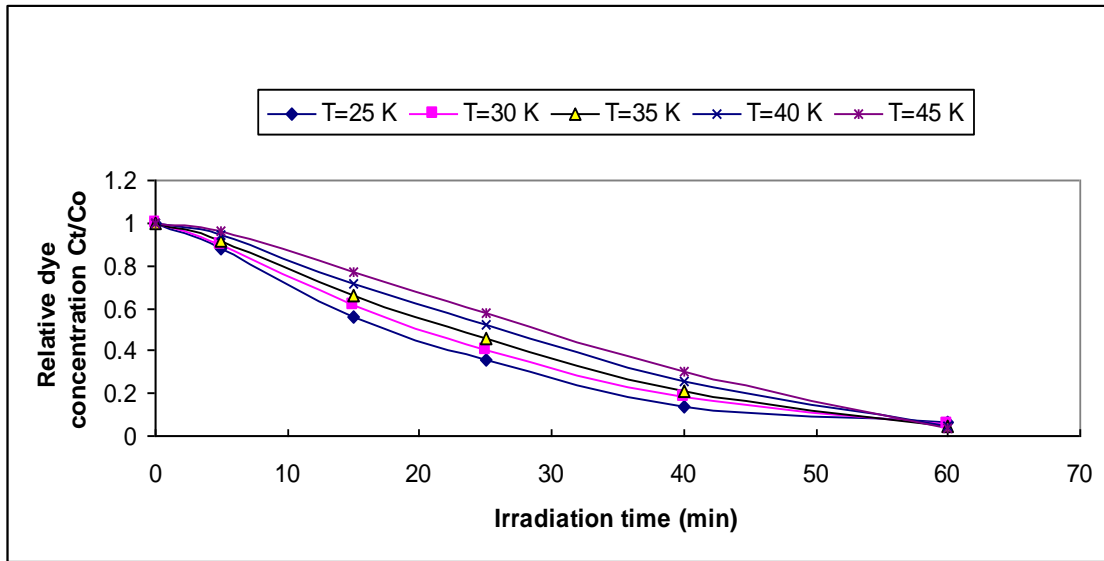


Figure (12): Effect of different temperature on the color removal of azure C using (UV/ClO<sub>2</sub>). [AC]=1x10<sup>-5</sup>M, pH=6, I<sub>0</sub>=173.711mW/ cm<sup>2</sup> and f=160 L/h.

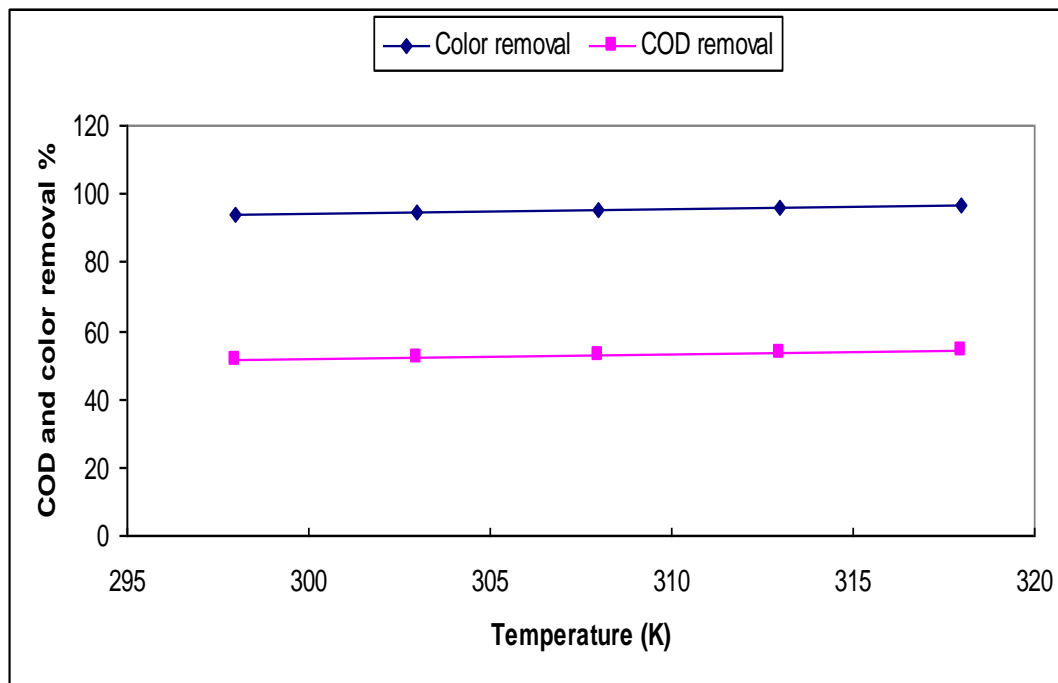


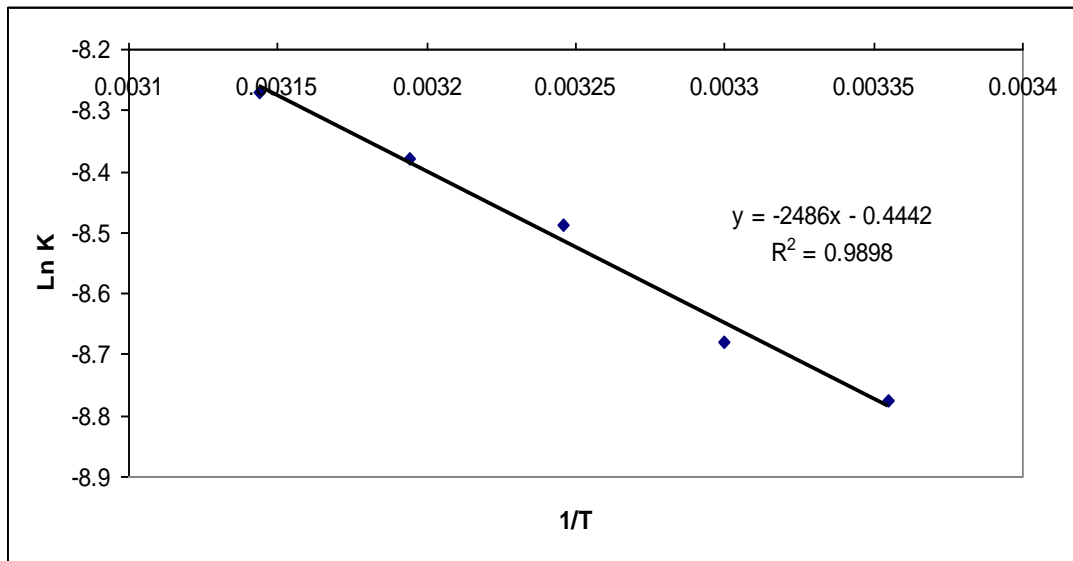
Figure (13): Effect of the temperature degree on color removal and COD of the [AB] =1X10<sup>-5</sup>M at pH=6, I<sub>0</sub>=173.711mW/cm<sup>2</sup> and f=160 L/h.

In this work we found out that with the increase of the temperature the value of COD removal increases because a decrease in the oxygen amount demanding for oxidation. As the oxygen the relationship between rate constants and temperature and draw decreases COD increases. The Arrhenius equation is the used to describe the relationship as show in Figure 14.

$$k = A e^{(-Ea/RT)} \dots\dots\dots(2)$$

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

Calculated the activation energy in plot was equal 20.668 KJ/mole.



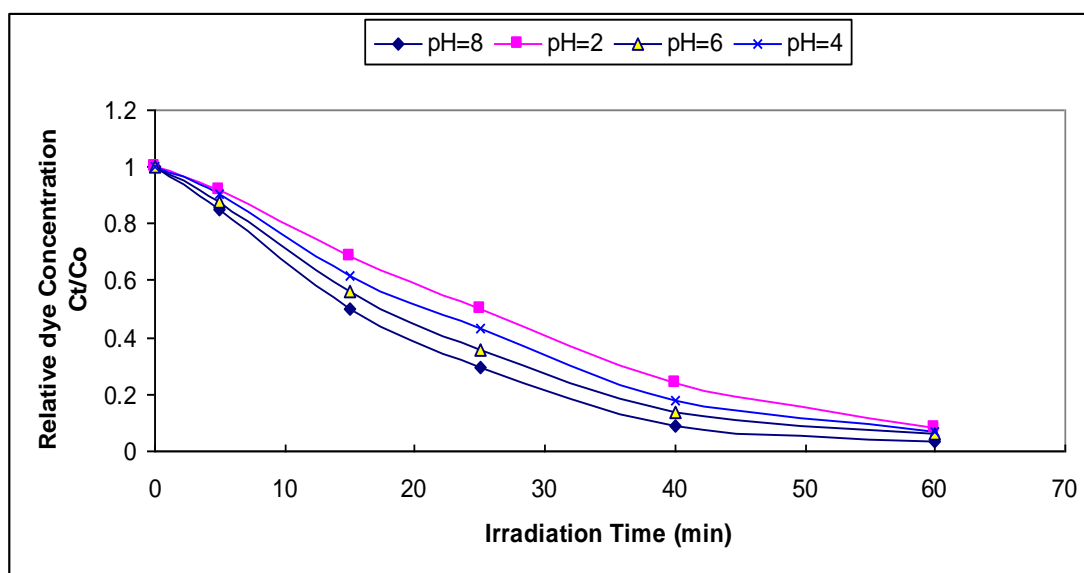
**Figure (14): Arrhenius plot of color removal of dye.[A.C]= 1x10<sup>-5</sup> M, pH=6 , I<sub>0</sub>=173.711 mW/cm<sup>2</sup> and f=160 L/h .**

**3- Effect of Initial pH**

The results showed a clear effect of initial pH in oxidation reaction of azure C dye. The high color removal was obtained under a basic media, because the chlorine dioxide function as a highly selective oxidant due to its unique one-electron abstraction mechanism, whereby it is reduced to chlorite ( $\text{ClO}_2^-$ ).

Chlorine dioxide is more reactive at higher pH<sup>(34)</sup>, and low color removal in acidic conditions due to chlorine dioxide is relatively inert and stable under acidic pH and it becomes unstable with increasing pH<sup>(35)</sup>.

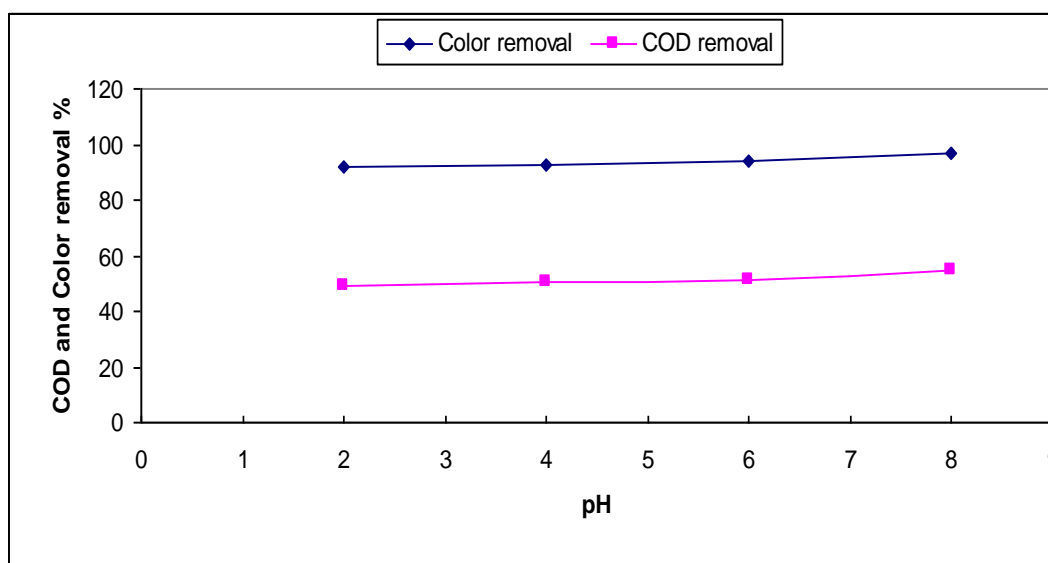
The higher ratio color removal of azure C dye was obtained under basic media at pH=8 under (UV/ $\text{ClO}_2$ ) and decreasing ratio in acidic media. The higher decolourization rate demand reduce pH value because changing in molecular structure. From Figure 15 it has been found that during 60 min. of (UV/ $\text{ClO}_2$ ), the relation dye concentration  $C_t/C_0$  was increased as the pH value increased.



**Figure (15): Effect of different pH value on color removal from azure C dye as function of irradiation time,  $[A.C]=1 \times 10^{-5} \text{M}$ ,  $T=298\text{k}$ ,  $I_0=173.711 \text{ mW/cm}^2$  and  $f=160 \text{ L/h}$ .**

The COD reached the maximum removal after irradiation of dye 60 minute at the initial pH value equal of 8. Figure 16 show effect of initial pH on COD and color removal.

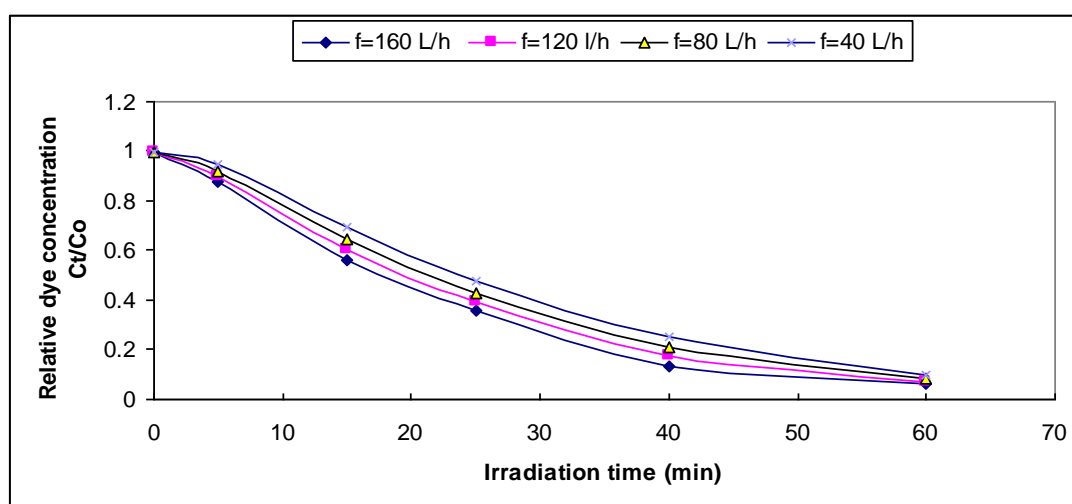




**Figure (16): Effect of the initial pH on the COD and color removal from azure C dye**  
 $[A.C]=1 \times 10^{-5} M$ ,  $T=298 K$ ,  $I_0=173.711 \text{ mW/cm}^2$  and  $f=160 \text{ L/h}$ .

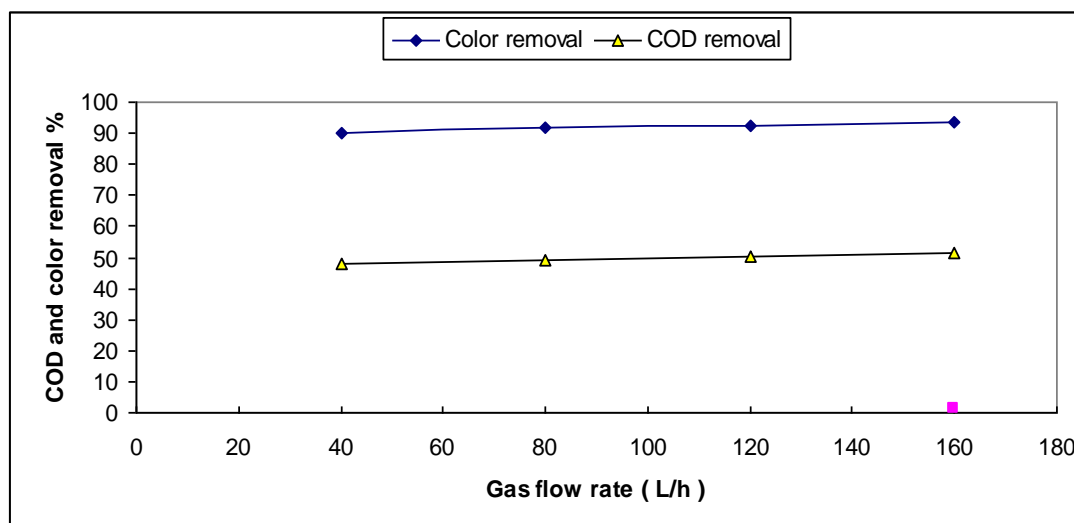
#### 4- Gas flow rate (f)

The effect of gas flow rate was evaluated at ( $f = 160, 120, 80$  and  $40 \text{ L/h}$ ). From Figure 17 the results proved the high color removal was 93.8% at high gas flow rate and low value was obtained in low gas flow rate 90.2%.



**Figure (17): Effect of gas flow rate value on color removal from azure C dye as function of irradiation time,  $[A.C]=1 \times 10^{-5} M$ ,  $T=298 K$ ,  $pH=6$  and  $I_0=173.711 \text{ mW/cm}^2$ .**

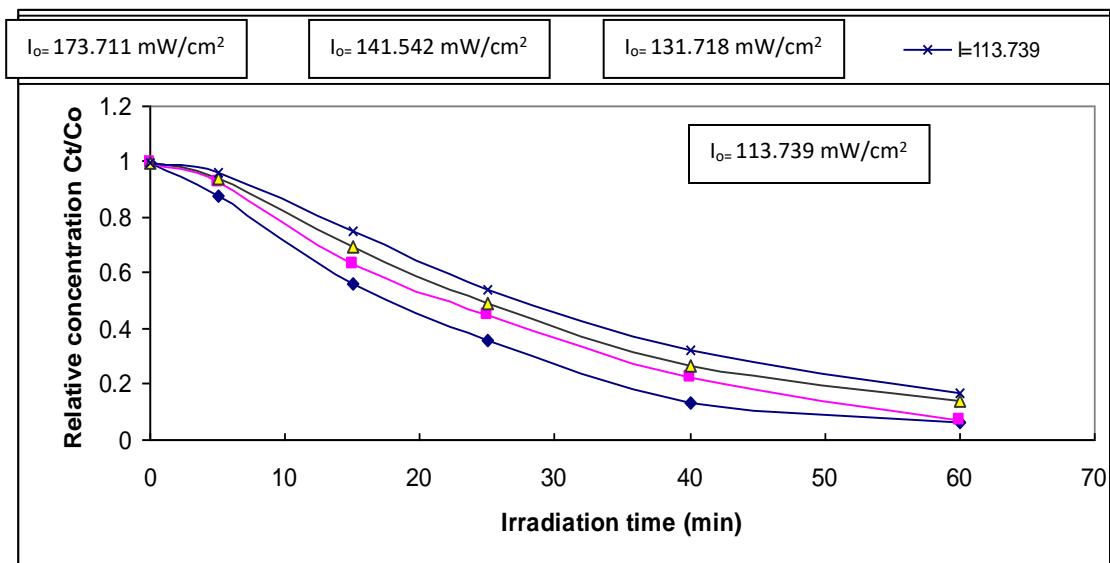
The effect of different gas flow rate on the color and COD removal of azure C dye by using the UV/ $ClO_2$  method was studied as show in Figure 18 .



**Figure (18): Effect of the gas flow rate on the COD and color removal from azure C dye  $[A.C]=1 \times 10^{-5} M$ ,  $T=298 K$ ,  $pH=6$  and  $I_0=173.711 \text{ mW/cm}^2$ .**

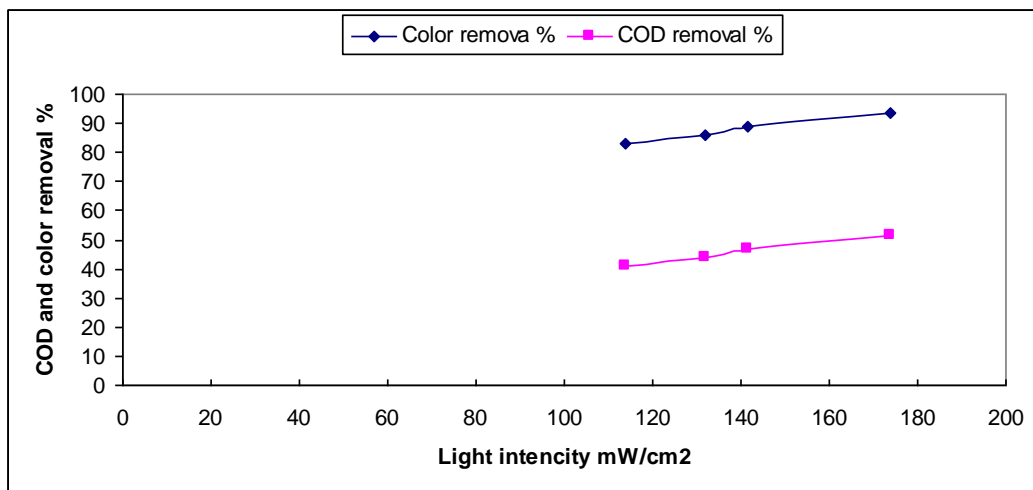
### 5- Light intensity ( $I_0$ )

The effect of light intensity was evaluated at ( $I_0 = 173.711, 141.512, 131.781$  and  $113.739 \text{ mW/cm}^2$ ). From Figure 19 the results proved the high color removal was 93.8% at high light intensity and low value was obtained in low light intensity 83.2% .



**Figure (19):** Effect of different light intensity value on color removal from azure C dye as function of irradiation time,  $[AC]=1 \times 10^{-5} \text{ M}$ ,  $T=298 \text{ K}$ ,  $\text{pH}=6$  and  $f=160 \text{ L/h}$ .

The effect of different light intensity on the color and COD removal of azure C dye by using the UV/ $\text{ClO}_2$  method was studied as show in Figure 20.



**Figure**

**(20):** Effect of the light intensity on the COD and color removal from azure C dye  $[A.C] = 1 \times 10^{-5} \text{ M}$ ,  $T=298 \text{ K}$ ,  $\text{pH}=6$  and  $f=160 \text{ L/h}$ .

## 6. Conclusion

1. The degradation was strongly influenced by various parameters, particularly the pH, temperature, dye concentration, gas flow rate, light intensity as well as irradiation time.
2. Faster degradation kinetics, higher color removal and COD removal efficiency was observed at higher temperature 318K.
3. The photooxidation of azure C by using the UV/ClO<sub>2</sub> is more effective in basic medium at pH = 8.

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