Photochemical activity and Physical properties of the Preparation new Catalyst Sb$_2$O$_3$-Cr$_2$O$_3$/Al$_2$O$_3$

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Abstract- In this work we prepared a new catalyst from Antimony (III) oxide, Chromium (III) oxide which mixed in different weight ratio and supported on granulated alumina by suspension method .

The film structure of prepared catalyst was characterized by X-Ray Diffraction spectrum. The physical properties also studied such as: porosity, pores size and surface area. The photocatalytic activity of prepared catalyst was studied by photocatalytic degradation of Neutral red dye by using sun light .In this work the effects of parameters such as amount of catalyst, effect of pH and dye concentration on photocatalytic degradation of dye were studied.

Index Terms*- Photocatalyst, Sb$_2$O$_3$-Cr$_2$O$_3$/Al$_2$O$_3$, Neutral red, sunlight, granulated alumina.*

I. INTRODUCTION

Water is the wellspring of life. It is the most important liquid in the world to maintain plant and animal life.
The two thirds of the earth's surface covered by water and human body consists of 75% of it, hence it is one of the prime element responsible for life on earth. Water is fouled due to the disposal of wastes generated from the various developmental activities and cause pollution problems [1] .

There are two choices to overcome these problems, *i.e.,* normally prevention and control by adopting various strategies to minimize the waste generation and by different treatment methods. Liquid waste is of great concern because of their harmful effects. Industrial wastewater contains wide variety of materials of both organic and inorganic nature including toxic substances like dyes and pigments, which are usually discharged with or without treatment [2].

Since the beginning of industrialization, the variety and quantity of pollutants emitted into the environment have steadily increased. But the rates of development of waste production are not likely to diminish; efforts to control and dispose of wastes are also rising. Several studies have been carried out for biological, physical and chemical treatment of dye containing [3].

Among these, biodegradation, adsorption, chlorination and ozonization are the most commonly used conventional methods. Dyes are usually resistant to aerobic degradation. Hence bio treatment alone has been found to be ineffective for the treatment of dye [4]. On the other hand, physical methods such as flocculation, reverse osmosis and adsorption are not destructive and mainly create pollutant concentrated phases, and many of this process are not found economically [5].

The use of heterogeneous photocatalytic treatment is more attractive for the degradation of organic dyes contrary to physical process; it can facilitate the complete mineralization of organic compounds to carbon dioxide, water and mineral acids [6-8]. Moreover, photocatalysis does not require expensive oxidant and can be carried out at natural sunlight.

In the present work, the photocatalytic degradation of Neutral Red (N.R), a textile azo dye   (3-Amino-7-dimethylamino-2-methylphenazine hydrochloride) , [Figure 4[16]]
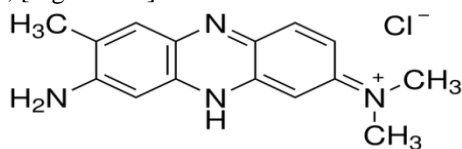

Figure 1. Structure of Neutral red

on synthesized Sb$_2$O$_3$-Cr$_2$O$_3$/Al$_2$O$_3$ under natural sunlight has been investigated by varying other parameters such as initial dye concentration, solution pH and catalyst amount [9]. Semiconductors activity have big band gab increase to UV-Visible light by using mixture as (Sb$_2$O$_3$-Cr$_2$O$_3$) where one oxides make injection photo electron from valance band to conduction band for another oxide as this figure 1[10].
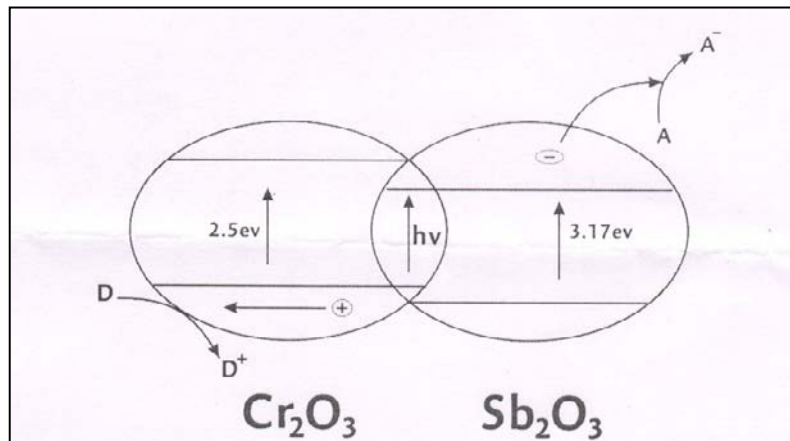
Figure 2:Charge separation in coupled semiconductors.

II. EXPERIMENTAL

2-1 Synthesis of Sb$_2$O$_3$-Cr$_2$O$_3$/Al$_2$O$_3$

The catalyst was prepared by mixed antimony oxide (III) and chromium oxide (III)   in different ratio (0:2,0.5:1.5,1:1,1.5:0.5,2:0) and supported on 10gm granulated alumina by dissolved in 20ml distilled water. The   thermal   vacuum evaporation technique was used at 323K to removal the solvent and then the catalyst dry in oven at 60°C .

2-2photooxidation of N.R dye

 1.Preparation the solution of N.R dye .

For most of the experiments, dye solutions of 100 ppm concentration were prepared by dissolving 0.1g of N.R dye in 1000 ml distilled water and from this solution prepared other dilute solutions (1-90) ppm by using dilution law .

As shown in the Fig. 3 below the absorption spectra of the NR dye solution shows the peaks in the visible region was observed at maximum wavelength 534 nm .

To determine calibration curve prepared series from NR solution in range from(1-20) ppm and measured absorbance at λ_{max} 534 nm and draw standard curve between absorption and concentration. A calibration curve of NR solution obtained at 534 nm wave length at different concentrations was prepared in order to correlate the concentration of dye at different reaction time by converting the absorbance of the sample to NR dye concentration Typical calibration value are plot in Fig.(4)
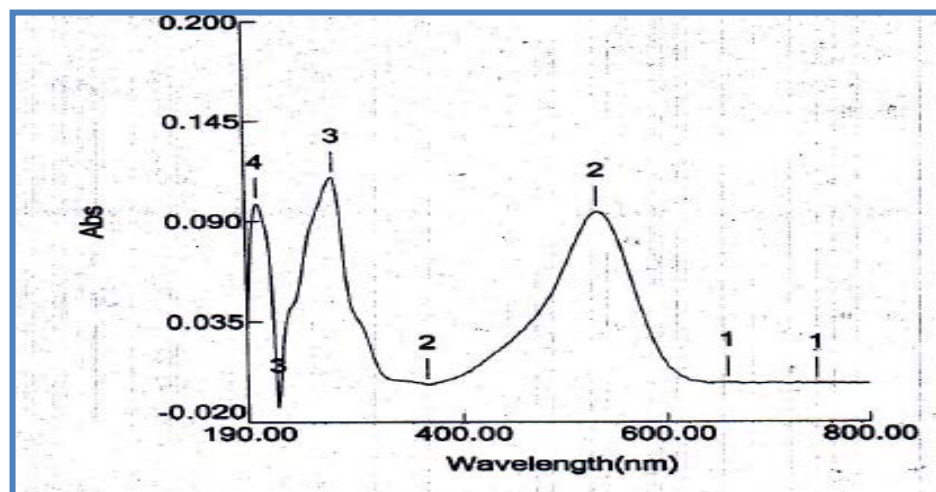
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Figure 3. Absorbance spectra of N.R dye solution(Concentration of dye =20ppm)
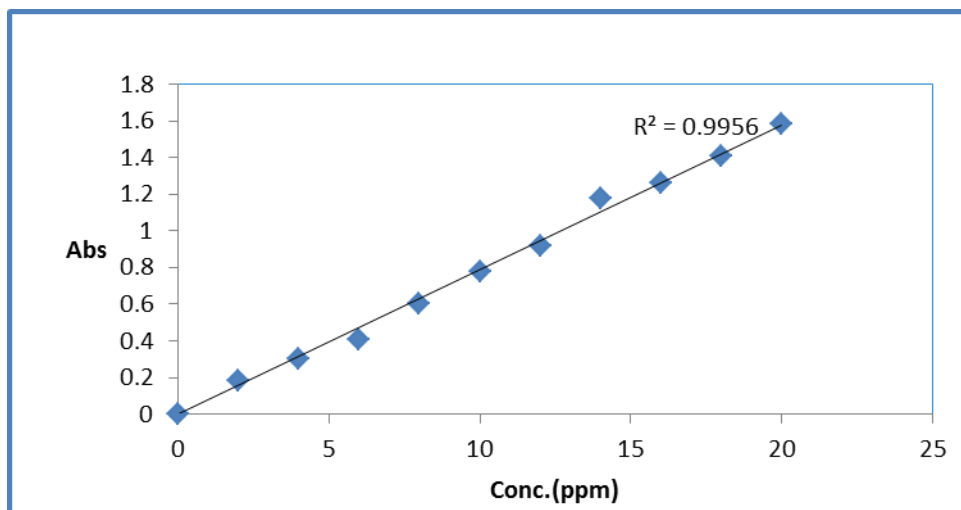
Figure 4 . Standard calibration curve of N.R dye solution

These experiments were made to determine the conditions optimum for photocatalytic degradation of N.R dye solution. They were carried out to evaluate the effect of catalyst and sunlight on photocatalytic degradation reaction.

In the a series of experimental has been done as blank experimental. In each one 200ml of N.R solution in concentration 20ppm at temperature 28C° was stirred to 60 min in the presence of the catalyst and the absence of the irradiation.

The effect of sunlight alone was study by using 200ml of N.R dye solutions in concentration 20ppm was irradiation with sunlight with the absence of catalyst, and continuous stirring for 60 min with cooling solution to keep the temperature $28C^0$ the time of irradiation (9: 00-10: 00) am on 1/5/2015.

2.Effect of catalyst amount on photoreaction

Catalyst was used in varying weights rang from (0.1- 1.25)gm during the photocatalytic treatment process for determination the optimization of the weight, was added to 200ml of 20ppm of dye solution in each trial at natural PH = 7.6 at temperature (28±2)C° to determine optimum for each concentrations. The degradation efficiency (%) has been calculated as :

 P.D.E = C_0-C_t/ C_0 x 100

where, Co and C_t are the initial and final concentration of dye for time t of irradiation. [17-18]

3. Effect of initial dye concentration on photoreaction

To study the effect of initial concentration of the dye solution on photocatalytic degradation, the initial concentration of N.R dye solution was selected in the range of (5,10,20,30,40) ppm with the best weight of the catalyst and follow the same steps to work as in the previous paragraph.
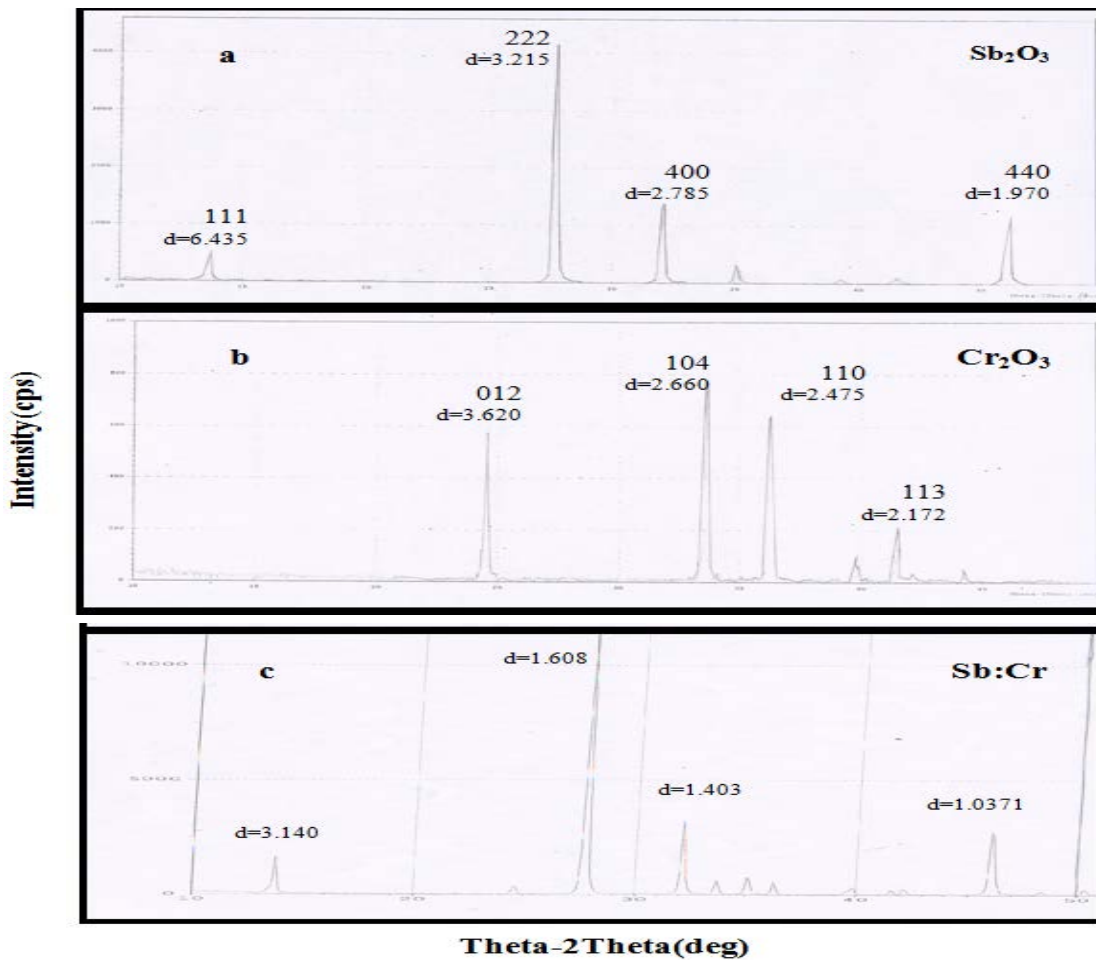
4.Effect of pH on photoreaction of N.R dye solution

After determination the best optimum weight of catalyst, the affect of pH on the rate of photocatalytic degradation was studied by keeping all the other experimental conditions constant at 20 ppm conc. of dye and 0.5g of catalyst at temperature (28±2)C° and changing the initial pH values of N.R dye solution from (2-14). The obtained of pH value result by add few amount of (0.05-1)N HCl or (0.05-1)N NaOH solution to adjust the acidity of the solution using a PH-Meter.

<div align="center">III.   RESULT AND DISCUSSION</div>

Determination of new catalyst Sb_2O_3-Cr_2O_3/Al_2O_3 1-3.
The X-ray diffraction of the synthesized Sb_2O_3-Cr_2O_3/Al_2O_3 was shown in Figure 5.

Figure 5: The spectrum of X-ray diffraction a- antimony (III) oxide
b- chromium (III) oxide c - a mixture of antimony oxide and chromium oxide.

The crystallization case for pure Sb_2O_3 pure Cr_2O_3 and the prepared new catalyst Sb_2O_3-Cr_2O_3 was study by using X-ray diffraction fig.(5) . As shown in tableS (1,2) the values of angles 2θ for pure Sb_2O_3 pure Cr_2O_3 and the prepared new catalyst Sb_2O_3-Cr_2O_3 . and space d and the relative intensity of the values of absorption of oxides, individually and in combination thereof at 50 % planned Consecutive.

Table 1: 2θ for standard Sb_2O_3 standard Cr_2O_3 and Sb_2O_3-Cr_2O_3 in the catalyst.

Metals oxide	2θ		
Pure Sb_2O_3	46.0269	32.1107	27.7240
Pure CrO_3	24.5708	36.2615	33.6558
Sb_2O_3-Cr_2O_3	13.82	46.32	27.79

Fig.2 also show the space d , which was calculated based on the Barak equation for X-ray diffraction values described as :
$$n \lambda = 2d \sin \theta$$

Where n = complications wavelength
   λ = wavelength of the x-ray
   d = the distance between the surfaces in the intra- crystalline structure
   θ = angle of diffraction

Table (2) the values of the distance between the surfaces in the intra- crystalline structure d for pure Sb_2O_3 pure Cr_2O_3 and the prepared new catalyst Sb_2O_3-Cr_2O_3 .

Table 2: d for pure Sb_2O_3 pure Cr_2O_3 and Sb_2O_3-Cr_2O_3 in the catalyst.

Metals oxide	d			
Pure Sb_2O_3	1.97034	2.78523	3.21515	
Pure CrO_3	3.62015	2.47536	2.66081	
Sb_2O_3-Cr_2O_3	3.1400	1.0371	1.4036	1.6085

Table (3) show the values of the relative intensity of the absorption peaks for pure Sb_2O_3 pure Cr_2O_3 and the prepared new catalyst Sb_2O_3-Cr_2O_3 .

Table 3: λ_{max} for pure Sb_2O_3 pure Cr_2O_3 and Sb_2O_3-Cr_2O_3 in the catalyst.

Metals oxide	λ_{max}			
Pure Sb_2O_3	601	608	1700	
Pure CrO_3	215	275	338	
Sb_2O_3-Cr_2O_3	1720	2760	3250	11750

The change in positions and values of 2θ , the Glades d and intensity of absorption relative of the new catalyst Sb_2O_3-Cr_2O_3 compared with each pure metals oxide. These deviations may due to the interaction between pure metals oxide which leads to change in crystals lattice of each oxide.
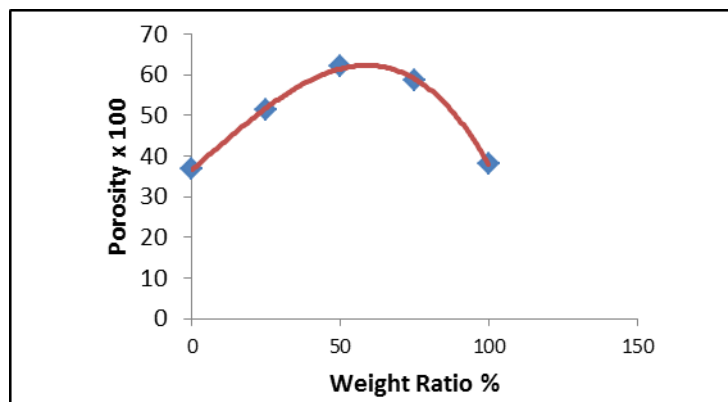
3-2 physical properties of Prepared Catalyst .
a.Surface Area.
The surface area of prepared catalyst was measured in weight ratio (50%) , the surface area was determine by adsorption of nitrogen gas on the surface of the catalyst and measuring the amount of nitrogen adsorbed at equilibrium conditions at the boiling point (-195 ° C) and at different nitrogen pressures that less than atmospheric pressure by using a special device and the result show that the surface area was 59.6 m^2/gm.
b. Porosity.
The porosity of prepared catalyst was measured in different ratio [0:2 (0%), 0.5:1.5 (25%), 1:1 (50%), 1.5:0.5 (75%), 2:0 (100%)] by Impregnation method [11-12], and the results were described in Figure 6.

Figure 6. Catalyst porosity in different weight ratio.

Figure 6. show the value of porous of the catalysts in percentages above we noted increase the value of the porosity with increase percentages weighted to reach to the highest value at the percentage of the weight (% 50 1:1) then the porosity decrease due to the removal of bonding water [13] , carbon dioxide and other volatile substances which leave after volatility slots and many pores, and the different between the crystalline system phases for the combination of oxides lead to a gathered randomly between two oxides, and leading to formation of holes [14].
c.Pore Size

The pore size of prepared catalyst was measured at different ratio [0:2 (0%), 0.5:1.5 (25%), 1:1 (50%), 1.5:0.5 (75%), 2:0 (100%)] by Impregnation method [11-12],and the results were described in Figure 7.
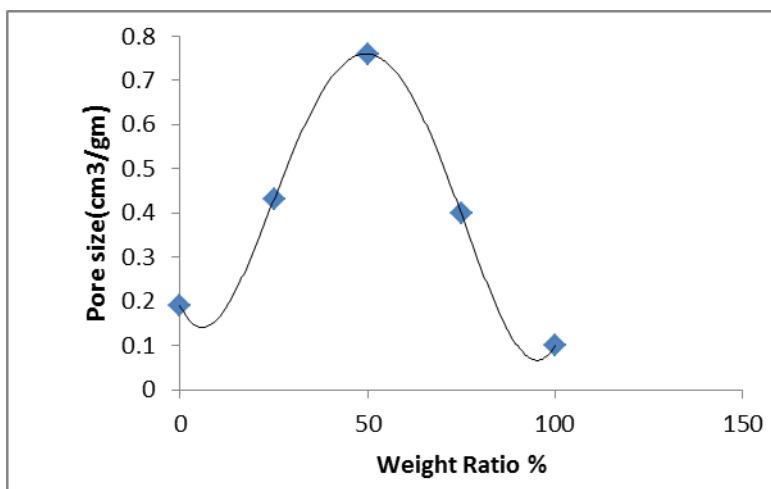

Figure 7. Catalyst pore size in different weight ratio.

Figure 7 show the value of pore size of the catalyst which increase with incrase of weight ratio to reach the highest value at the weight ratio (%50 1:1) then decrease. The reason for this is that the stacking , convergence and interaction between asymmetric atoms in produce catalyst was largest than of single oxide catalyst and this reduces the pore size between the atoms, but the stacking , convergence and integration between the various atoms is less and this leads to increase the pore size between the various atoms [15] and leads to combines random component between oxides atoms and produce larger pore size .

3-3 Photooxidation activity of new catalyst:

Several optimization experiments were carried out to determine the conditions necessary for photo oxidation of N.R dye solution by using the preparation catalyst with sunlight for one hr. irradiation for each experiment at 28C° .

The results of the present study clearly show that the photooxidation treatment of aqueous solution of N.R dye under sunlight by using the preparation catalyst molecule which in turn helps the formation of free radicals ˙OH Which is one of the oxidizing agents powerful [19] , leads to degradation of dye. Experiments were performed using a catalyst and in the absence of sunlight receptor proved proportion of the remaining dye NR at 40min where then is the adsorption of the first layer of dye molecules on the surface of the semiconductor and the lack of disintegration of the pigment, but by very few are almost negligible[20], and as shown in Figure (8)

1- **Solar reaction .**

Was conducted experiments the presence of sunlight and the absence of catalyst was observed disintegration of the dye by 75%, but a longer time if compared to the disintegration of the dye in the presence sunlight and catalyst where the sun light provides the visible rays that run on exciting the dye and adsorbed on the surface of the catalyst and the ultraviolet rays that transport electrons from the valence band to conduction band and this package is available factors dye adsorption and electrons ,positive holes on the surface of the catalyst (h^+, e) In this way the dye disintegrate and significantly shorter time[21]
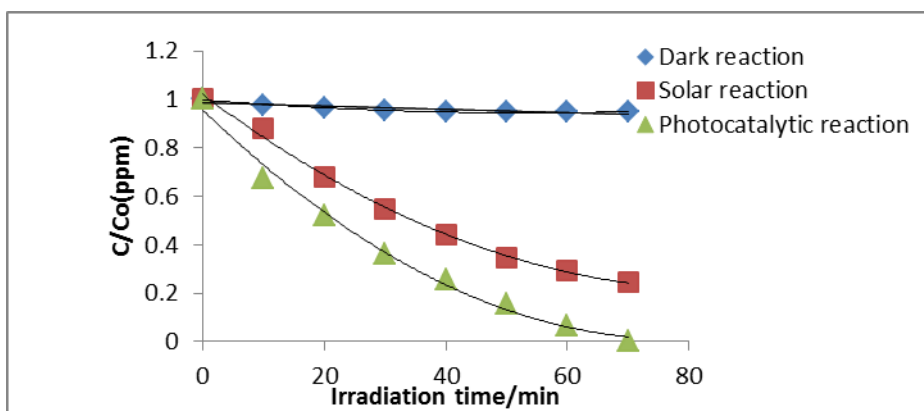

Figure 8. The dye oxidation in different condition.

2-The effective factors in photo catalytic oxidation reactions for N.R dye.
Effect of the amount of catalyst on photo oxidation reactions.

The rate of decolorization increased with the increasing catalyst amount from 0.1 g to 1.25g .The best catalyst weight 0.5gm which gave the ratio of the decolorization .

we noticed from Figures (9,10) that the best catalyst weight 0.5gm which gave the ratio of the disintegration 99.2% at (4min) by using sunlight note from the results that the rate of dissolution of the dye increases with the weight of the catalyst to reach the highest value and then decreases or prove. These results strongly agreed with our previous findings. and explain it on the basis that increasing the amount of catalyst leads to an increase sites active on the surface of the catalyst and increase the weight of the catalyst is higher than the maximum value lead to increased suspended particles in the aqueous solution of the dye and thereby prevent penetrating rays to reach all parts of the aqueous solution of the dye leading to a decrease in the proportion of disintegration[22-26]
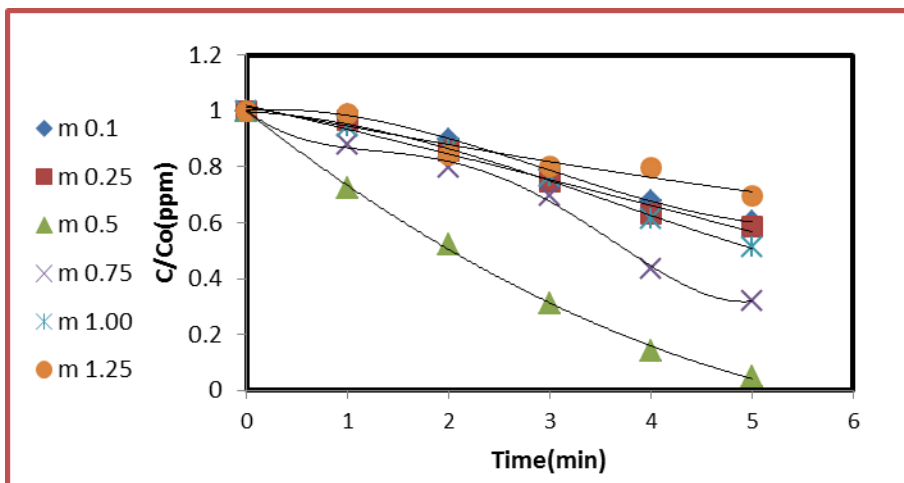

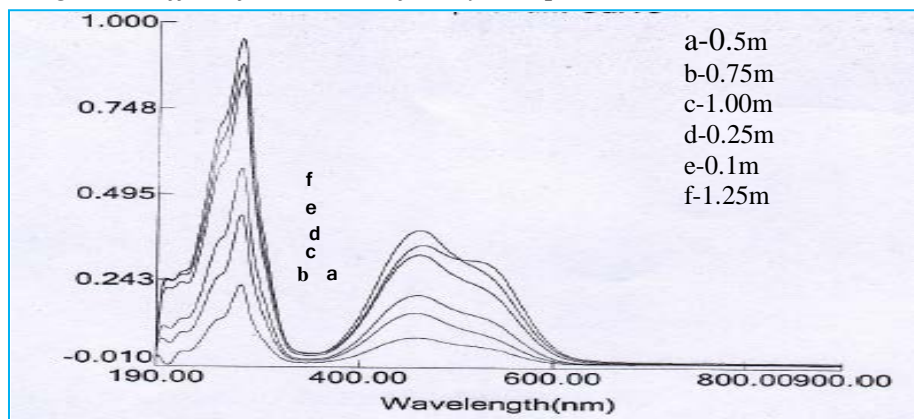*Figure 9 . Effect of the amount of catalyst on photo oxidation reactions.*

Figure 10 . Absorbance spectra of the N R dye in different amount of catalyst .

Effect of dye concentration

The effect of initial dye concentration on the photocatalytic degradation of N.R dye was studied at different concentrations of dye in the range of (5- 40) ppm. Fig.(11) shows the percent degradation at various initial dye concentrations. It was observed that the percent degradation gradually increased with the decreasing initial dye concentration.

Percentage of decolorization was found to be (99.2, 97.8, 92.5 and 82.9) at (5, 10, 20,and 30) ppm initial concentrations of dye, respectively.

This behavior may be due to the decrease in the concentration OH^- adsorbed on catalyst surface with the increasing dye concentration. The competitions between OH^- ions to adsorb on active sites of the catalyst will be in the favor of dye ions, when the concentration of dye was increased. As a result, •OH formation rate decreased and then the rate of decolorization also decreases. The inverse proportionality of rate of decolorization with dye concentration may be also due to increase of reduction of light intensity reach the catalyst surface and consequently, photon absorption on surface of catalyst is also reduced with the increasing dye concentration[27-29]
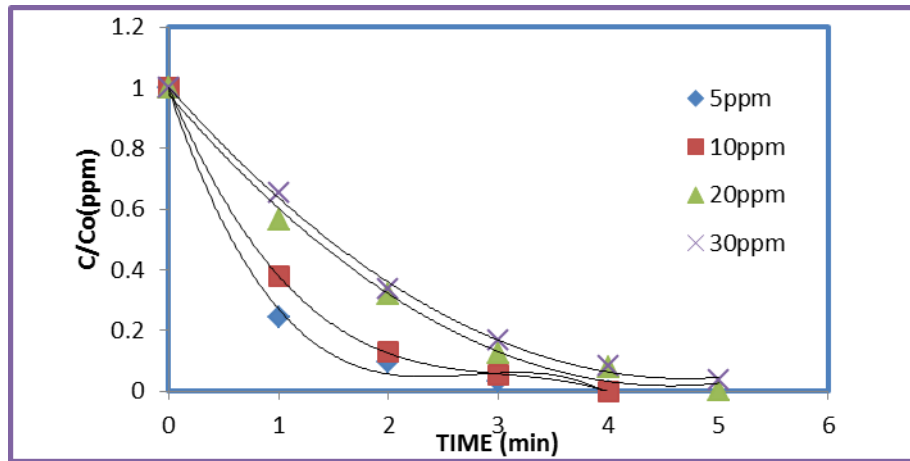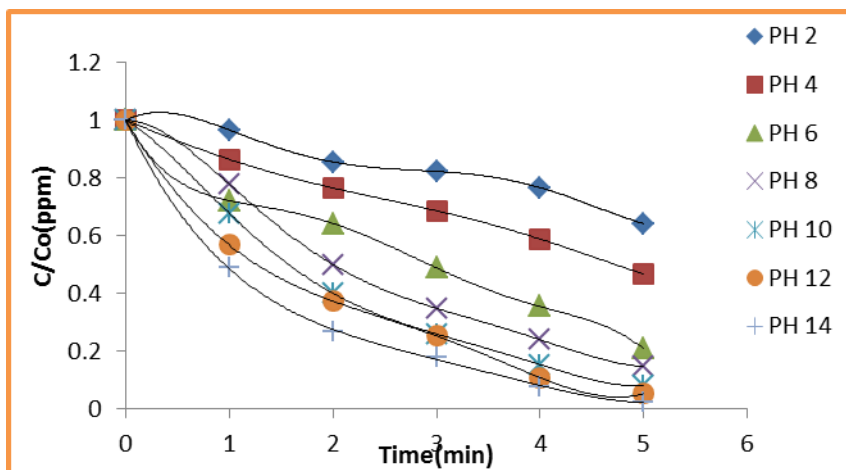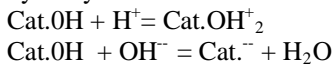
Figure 11. Effect of initial dye concentration .

Effect pH of the N.R dye solution.

Aqueous solution pH is an important variable in the evaluation of aqueous phase mediated degradation reactions. pH change effects the adsorption photo catalytic quantity of organic pollutants and the ways of adsorption on the surface of photo catalyst (coordination). As a result, the photo catalytic degradation efficiency will greatly be influenced by pH changes. Zero Point Charge (pHzpc), is a concept relating to adsorption phenomenon and defined as the pH at which the surface of an oxide is uncharged.

If positive and negative charges are both present in equal amounts, then this is the isoelectric point(iep). However, the zpc is the same as iep when there is no adsorption of other ions than the potential determining H+/OH– at the surface. In aqueous solution, at pH higher than pHzpc, the oxide surface is negatively charged and then the adsorption of cat ions is favored and as a consequence, oxidation of cationic electron donors and acceptors are favored. At pH lower than pHzpc, the adsorbent surface is positively charged and then the adsorption of anions is favored and as a consequence, the acidic water donates more protons than hydroxide groups[30-32].

We studied the effect of PH dye solution and the results are shown in figures (12,13) which showing increasing proportion of degradation the dye at PH> 7 , because depending that on the catalyst zero point charge PH zpc of Sb_2O_3 and Cr_2O_3 5.1,6.8 respectively [33], where the surface of the catalyst positively charged in PH <7 so that the cationic dye not adsorbate on the catalyst surface ,while in PH> 7 the surface of the catalyst negatively charged due to the adsorption of hydroxyl ions OH-, which help to form hydroxyl free radical which is responsible for the decolorization of dye.[34]

$Cat.0H + H^+= Cat.OH^+_2$
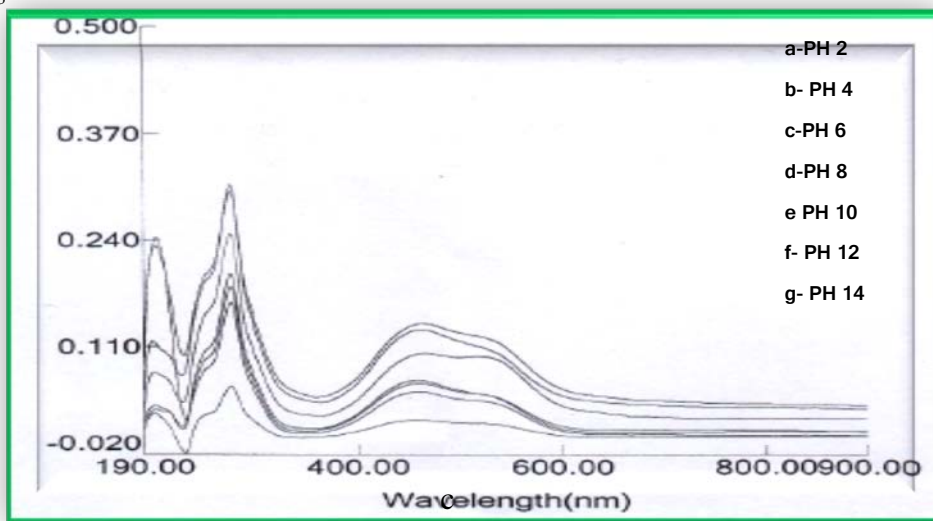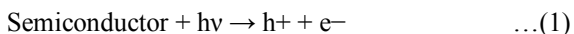
$Cat.0H + OH^- = Cat.^{..-} + H_2O$

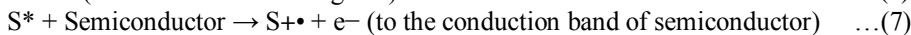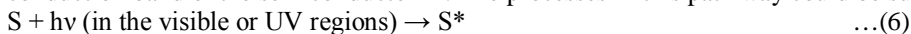*Figure 13 . Absorbance spectra of the N R dye in different pH of the N.R dye solution .*

Mechanism of photooxidation of dye:

Based on experimental observations, a tentative mechanism for the bleaching of dye(Dye) i.e. neutral red has been proposed. The illumination of suspended semiconductor in an aqueous solution of dye with solar light lead to the possibility of the existence of two pathways[35,36].

(i) In the first pathway, the part of light with energy equal to or more than the band gap of the illuminated semiconductor will cause a promotion of an electron to conduction band of the semiconductor and as a result, a positive hole will be created in the valence band .The formed photo holes and photoelectrons can move to the surface of the semiconductor in presence of light energy. The positive hole will react with adsorbed water molecules on the surface of semiconductor producing •OH radicals and the electron will react with adsorbed oxygen on the surface. Moreover, they can react with deliquescent oxygen and water in suspended liquid and produce per hydroxyl radicals ($HO^{\bullet}_2$) with high chemical activity[37]. The processes in this pathway could be summarized in the following equations:

$$Semiconductor + h\nu \rightarrow h+ + e- \quad \dots(1)$$
$$h+ + OH- \rightarrow \bullet OH \quad \dots(2)$$
$$h+ + H2O \rightarrow H+ + \bullet OH \quad \dots(3)$$
$$e- + O2 \rightarrow O2-\bullet \quad \dots(4)$$
$$O2-\bullet + H+ \rightarrow HO2\bullet \quad \dots(5)$$

(ii) In the second pathway, the other part of light with energy less than the band gap of the illuminated semiconductor will be absorbed by the adsorbed dye molecules. Dye molecules will be decolorized by a photosensitization process. The photo catalytic de colorization of dyes, which is described as a photo sensitization processes are characterized also by a free radical mechanism. In this process, the adsorbed dyes molecules(s) on the surface of the semiconductor could absorb a radiation in the visible range in addition to the radiation with a short wavelengths[38-40]. The excited colored dye (S*) (in the singlet or triplet state) will inject an electron to the conduction band of the semiconductor[41]. The processes in this path way could be summarized in the following equations:

$$S + h\nu \text{ (in the visible or UV regions)} \rightarrow S* \quad \dots(6)$$
$$S* + Semiconductor \rightarrow S+\bullet + e- \text{ (to the conduction band of semiconductor)} \quad \dots(7)$$
$$e- + O2 \rightarrow O2-\bullet \quad \dots(8)$$
$$O2-\bullet + H2O \rightarrow OH- + HO\bullet2 \quad \dots(9)$$
$$S+\bullet + OH- \rightarrow \bullet OH + S \quad \dots(10)$$

IV. CONCLUSION

Sb_2O_3-Cr_2O_3/Al_2O_3 catalyst was prepared by a suspension method from Antimony (III) oxide, Chromium (III) oxide which mixed in different weight ratio and supported on granulated alumina .

The film structure of prepared catalyst was characterized by X-Ray Diffraction spectrum. The physical properties also studied such as: porosity, pores size and surface area. The photocatalytic activity of prepared catalyst was studied by photocatalytic degradation of azo textile neutral red dye by using sun light .In this work the effects of parameters such as amount of catalyst, effect of pH and dye concentration on photocatalytic degradation of dye were studied.

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