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Preparing and Characterizing Study of the Photocatalytic Activity of New Couple Semiconductor Nb_2o_5/Sb_2O_3.

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ABSTRACT

This work includes the study of preparing the new Nb_2o_5/ Sb_2o_3 coupled oxide photocatalyst was prepared by solid method at different ratios of (0.25:0.75 ,0.5:0.5 and 0.75:0.25) and calcination at different temperature 400 C^0,500 C^0 and 600 $C^0$for two hours .The prepared powder was characterized by x-ray diffraction , UV-Vis spectra , atomic flame microscopy (AFM) and Fourier Transform Technique (FT-IR).The photocatalytic activity was evaluated under mercury high pressure lamp for decolonization of azo dye orange G solution after determine the wavelength at λ_{max} 475 .the result showed that (0.5:0.5) percentage at 600 C^0 has high activity than other ratio at different temperature . After that study some parameter such as initial of concentration dye ,best of mass for the catalyst and effect of pH.

Keywords: couple Nb_2o_5/Sb_2o_3 , orange G , Degradation, photocatalytic

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INTRODUCTION

Niobium oxide catalyst has received attention in the recent years due to broad industrial application such as solar cell , optoelectronic technology and catalytic activity[1], it used in photo reaction as pure powder or couple with other semiconductor for degradation of organic compound such as dyes[2].

Dye type orange G from azo dyes are common used in many filed industries such as textile, leather, paint, food, cosmetics and pharmaceuticals[3], also used in biological filed by give the various color between different of the tissues to allow examination under light of microscopy.

Water treatment a very important step in new world, there are different methods using for water treatment, such as adsorption processes [4-12], photocatalytic degradation [13-19].

In this paper a new couple of Nb_2o_5/Sb_2o_3 prepared and examined as a good photocatalytic using for degradation of orange G dye.

EXPERIMENTAL

Materials

Chemical materials used in this work is antimony trioxide Sb_2O_3 purity 98%, supplied by Fluka AG.3-Normal, Niobium pent oxide Nb2o5 has pure 99% %, supplied by Fluka AG.3-Normal ,and orange G dye was supplied by sigma-Aldrich has purity more than 90%. Structure and physical characteristics for orange G dye are given in table (1) and figure (1).

Table 1: functional group and physical characteristics for orange G dye

Dye	Function group	IR band(cm^{-1})	physical characteristics
Orange G	-OH -N=N -SO3H Aromatic ring Naphthalene ring	3250.13 1051.6 1198 970 684.80	Acidic dye pH=6.8 $\lambda_{max=475}$ melting point = 141

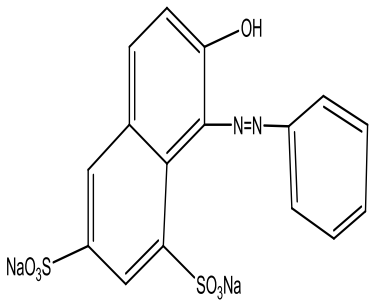

Figure 1: Structure of Orange G dye molecule

Preparation and characterization of couple Sb2O3/Nb_2O_5

Couple of Nb_2o_5/ sb_2o_3 was prepare by solid method as described that involved using 98% pure sb2o3 mixed with 99.9% pure Nb_2o_5 powders as initial materials which mixed by mortar for 15 minutes after that a calcination this mixture in oven at 600c for two hours. After that returned the mixed by mortar. The couple of semiconductors Nb_2o_5/ Sb_2O_3 prepare was characterized by x-ray diffraction (XRD- 600 - SHIMADZU), FTIR spectroscopy (FTIR- 8400S, SHIMADZU) and atomic flam microscopy (AFM).

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RESULT AND DISCUSSION

X-ray diffraction patterns

The naked sb_2o_3 and Nb_2o_5 are characterized by x-ray diffraction(XRD), and compared with couple sb_2o_3/ Nb_2o_5. Figure 2 A. find three different strong peaks apparent in shape of spectrum represent 2θ at (22.6472 , 28.3790 , 23.8089) back for initial material niobium pent oxide (Nb_2o_5).

Figure 2. B. find three different strong peaks apparent in shape of spectrum represent 2ɵ at (27.6940 , 45.9881 , 32.0745) back for initial material antimony trioxide (Sb_2O_3), While in figure2.c for mixed oxide (Nb_2o_5/Sb_2o_3) consist of three different strong peaks ,at 2 Theta (29.0311 , 30.3607 , 25.8261) notes appear two strong peaks in spectrum at 2 Theta (29.0311 , 30.3607) not found in two initial material (Sb_2o_3-Nb_2o_5) . and three strong peak for couple oxide at 2 Theta (25.8261) was the same in spectrum of initial material niobium pent oxide (Nb_2o_5) but in more intensity .

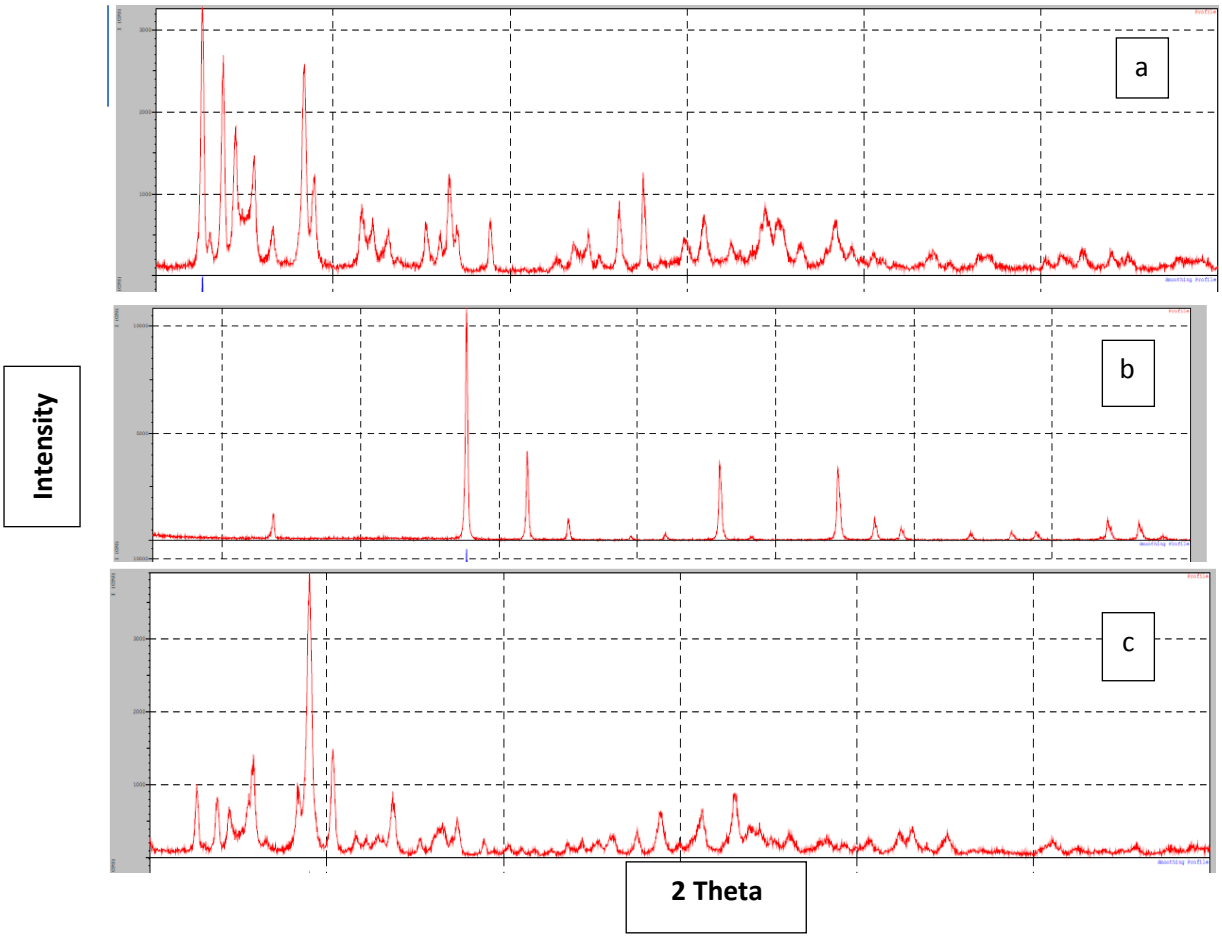

Figure 2: X-ray diffraction spectrum of a. Niobium pentoxide Nb2o5 b. Antimony trioxide sb_2o_3 c. Couple Nb_2o_5/ sb_2o_3.

From application of Scherer equation find the average piratical size (nm), results are shown in table 2.

$$D = K \lambda / ß \cos ɵ \qquad (1)$$

D: represent the average particle size.
K= is dimension shape factor 0.9
λ = is X-ray wavelength .
ß = is the liner broadening at half the maximum intensity.
ɵ = is Bragg angle

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Table 2: The average practical size for couples Nb_2O_5/ Sb_2O_3 and initial materials

Catalyst	2 Theta (deg)	Average particle size(nm)
Nb_2O_5	22.6472	**D = 41.22**
	28.3790	**D = 31.68**
	23.8089	**D = 40.18**
Sb_2O_3	27.6940	**D = 51.35**
	45.9881	**D = 54.14**
	32.0745	**D = 54.70**
Couple of Sb_2O_3/Nb_2O_5	29.0311	**D =25.40**
	30.3607	**D = 32.04**
	25.8261	**D = 23.85**

Fourier Transition for Infrared spectrum (FT-IR)

The measure of Fourier Transition for Infrared spectrum (FT-IR) for nanocrystal carried out in the IR region between the wavelength (400-4000) 1/cm.

The spectrum 0f figure 3.a is characteristic of the sample niobium pentoxide Nb_2O_5 that show the peaks at 3674.52 , 1734.06 , 1558.54 , 1049.31 , 806.27 , 621.10 503.44 437.86 (3674.52) cm^{-1} return to absorption water band in the sample , the peak observe at 1734.06 cm^{-1} is return for Nb=o overtone.

Figure 3. B. appear are the peaks at 3518.28 , 3290.67 , 2308.87, 1651.12 , 1321.28 1093.67 , 950.94 , 715.61,673.18, 617.24 .the peaks (3518.28 and 3290.67) cm^{-1} are return to stretching and bending vibration for two water band these peaks indicate the hydroscopic character of the sample (Sb_2O_3).

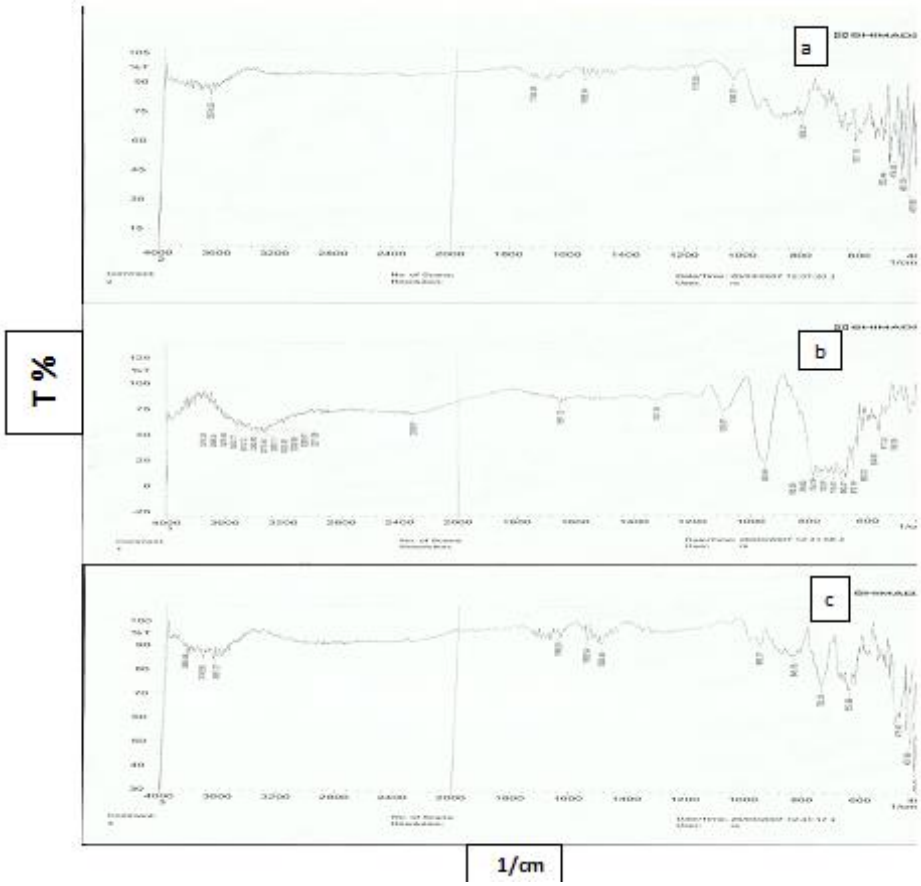

Figure 3: FTIR Spectrum for a. niobium pentoxide (Nb_2O_5) b. Antimony trioxide ($Sb_2O_{3)}$ and c. couple of sb_2O_3/Nb_2O_5

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Also notes the appear peak at 715.16 and 637.64 are attributed to the Sb-O–Sb vibration. Also see the peak at 617.24 is due to Sb-O stretching[20].

Spectrum of Figure c. appear the peaks at 3745.88,3865.48 , 1656.91 , 1558.54 , 1506.46 , 966.37, 846.78 , 750.33 , 653.89 ,437.86.the peaks at (3745.88,3865.48) indicate the hydroscopic character of the couple , the peak at 1656.91 cm-1 also can see in spectrum of sb_2o_3 but in the couple appear more intensity , the peak at1558.54 cm^{-1} returnee to the initial material Nb_2o_5 , the new peaks observe at 1506.46, 750.33 and 653.89 cm^{-1} the peak at 437.86 cm-1 also we can see in spectrum of Nb_2o_5.

Atomic Force Microscopy

AFM analysis provide for taking information about the surface of the sample .the images illustrate the surface morphology and explain the distribution of particle on the surface and dimension for it, results are shown in figure 4.

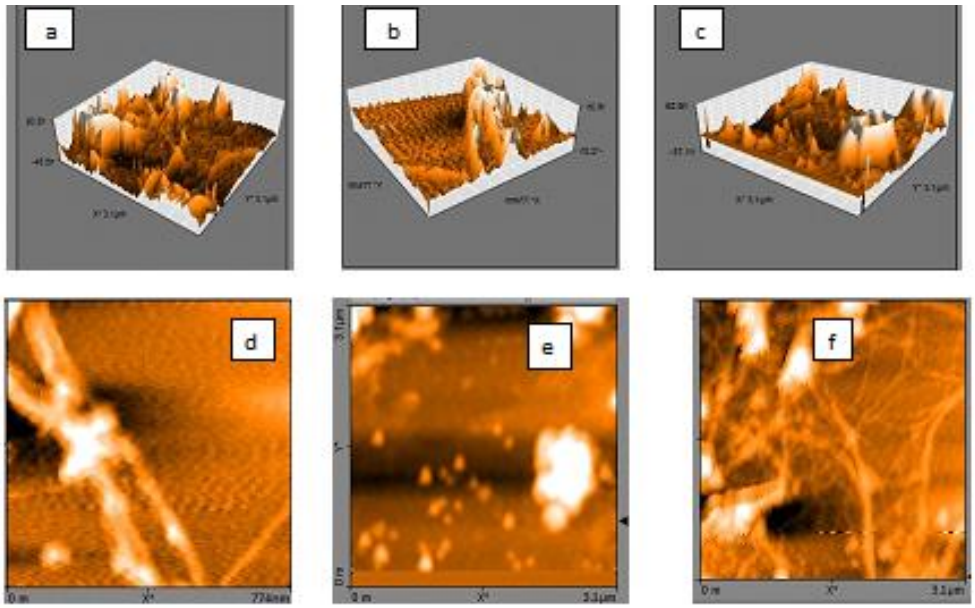

Figure 4: a. AFM 3D of couple Nb_2o_5/Sb_2o_3 , b. AFM 3D of Sb_2o_3 , c. AFM 3D of Nb_2o_5 , d. AFM 2D of Sb_2o_3 , e . AFM 2D of Nb_2o_5 and f. AFM 2D of couple Nb_2o_5/Sb_2o_3

Photocatalytic experiments

Effect of the mass couple Nb_2o_5/ sb_2o_3 on the photodegradation of orange G dye

Figure 4 .Show the change in rate constant of different masses of couple Nb_2o_5/Sb_2o_3 .

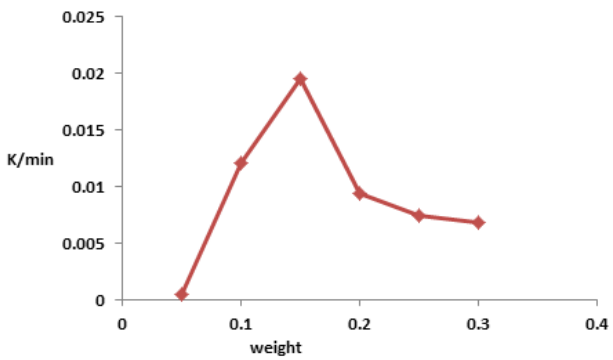

Figure 4: the effect of the weight of couple on rate constant

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This factor studied by utilized different mass of couple under optimum condition include the used concentration constant of orange G dye 10PPM , temperature at 298.15 K and normal pH for dye 6.8.

The rate constant of photodegradation process increase with weight increase the maximum degradation (86.71%) of the orange G at couple amount 0.15 g after that weight the percentage of degradation was decrease.

The rate of photocatalytic increase with increase the amount of couple it may due to the increase the active site on the surface of couple Nb_2o_5/sb_2o_3 , but increase the amount of couple causes the decrease in the efficiency of photodegradation this inhibition may due to successive layer of molecules couple that prevent light from passing through other layer[21, 22]

Effect of concentration of orange G dye

This experiments were carried out with different concentration of orange G dye under optimum condition 0.15 g of couple Nb_2O_5/ Sb_2O_3 , light of high pressure mercury lamp has intensity equal to 2.75 mW/cm^2 , normal pH 6.8 for solution and temperature at 298.15 K.

In range between 10- 25 ppm . the best concentration of degradation dye at optimum condition is 10 ppm.

Table 4 and Figure 5. show the change of rate constant of different concentration dye

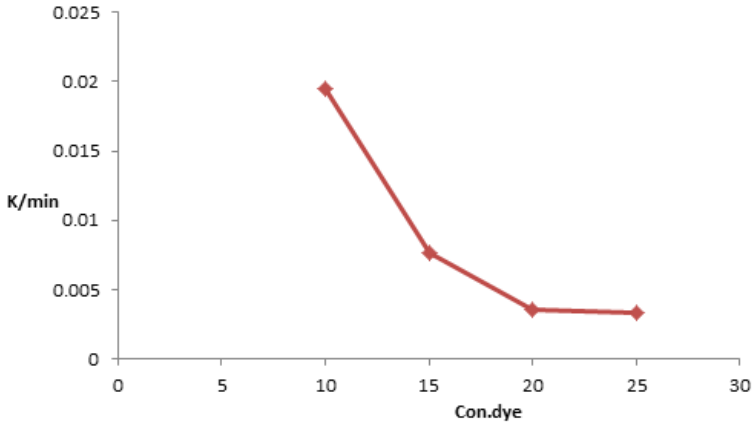

Figure 5: Show the effect of concentration dye on rate constant

Effect of pH dye

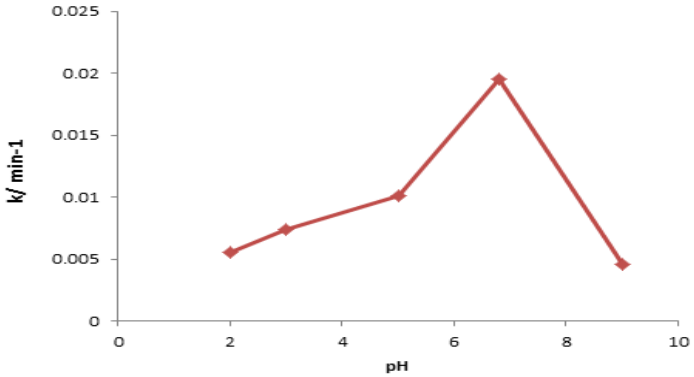

Figure 6: Shows effect of pH on rate constant

Found many factures which influence the effect of pH dye such as ionization state of the surface, nature of the dye, particle size and ability of molecular adsorbed adsorption on the catalyst surface. The degradation reaction of orange G dye carried out at the pH range between 2-9 at optimum condition 10 ppm dye concentration, 0.15 g catalyst amount and temperature equal to 298.15 K. the acid-base of the solution was adjusted by using HCl and NaOH prepare solutions . From measure the absorbance for different pH.it can observe the rate constant of different pH value from table 5.

From figure 6 notes the rate constant increased when the pH increase from 2 to 6.8 after that the rate constant of dye initial down on this behaved similar with literature[23]. The reason for this result the medium of acidic pH solution causes to more H+ ions are available for the adsorption to mask the surface of the catalyst therefore prevent the photoexcitation of catalyst particles , thereby reducing the generation of free radicals.

CONCLUSION

From the results we found at the 0.5:0.5 percentage is more active than other percentage. Formation the new couple Nb_2o_5/Sb_2o_3 of semiconductors registration that by x-ray diffraction patterns through appears new peaks at 2 theta 29.0311, and 30.3607 and in spectrum of FT- IR show the formation of new couple of semiconductors. This couple appears the ability for photodegradation of orange G dye 86.74% in the optimum condition amount of couple 0.15 g, concentration dye 10 ppm, temperature 298.15 K and normal pH 6.8 of dye solution. This photoreaction cared out with radiation time 120 min.

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