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Synthesis, Characterization of Nb2O5\Cds Nanocomposites and Study of High Photo Catalytic Activity of Transition Metal Ion

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Abstract : This work includes the study of preparing the new Nb_2O_5/CdS coupled photocatalyst was prepare by wet commixing method at different ratios of (0.75:0.25, 0.6:0.4,0.5:0.5, 0.85:0.15,0:1 ,1:0) and calcinations at different temperature 200 ^{0}C,500 ^{0}C and800^{0}C for4 hours .The prepared powder was characterized by X-ray diffraction, and Fourier Transform Technique (FT-IR).The photocatalytic activity was estimated under mercury high pressure lamp for degradation $Co(NO_3)_2$ solution after find the wavelength at λ_{max}510nm . The result showed that (0.85:0.15) percentage at 800 ^{0}C has high activity than other ratio at different temperature. After this study some measure such as best of mass for the catalyst, initial of concentration for $Co(NO_3)_2$, effect of temperature, effect of PH.
Keywords: Co(NO3)2, couple Nb2o5/CdS, Degradation, photocatalytic.

Introduction:

Niobium pentoxide (Nb_2O_5) is considered one of the most committed transition metal oxides (TMO) for pseudocapacitive energy storage. Nb_2O_5 is to a great degreestudied in lithium-ion batteries (LIB)[1], electrochemical hydrogenation catalysts[2].gas sensing[3], electrochromic devices[4] and solar cells[5].

Nb_2O_5 exhibits a different of crystalline allotropes,with orthorhombic (T-Nb2O5), pseudo-hexagonal (TT-Nb_2O_5), tetragonal (M-Nb_2O_5) and monoclinic(H-Nb_2O_5)[6,7]. In the mainfoundin the form of stiochiometric oxides such as NbO, Nb_2O_3, NbO_2 and Nb_2O_5[8].

Cadmium sulphide is the inorganic compound with the formula CdS. Cadmium sulphide is a yellow solid[9]. CdS is a group II–VI semiconductor, and such as, CdS nanoparticles have made large importance due to their singlesize-dependent chemical and physical properties[10].CdS in bulk has band gap energy of 2.42eV at 300K with absorption maxima at 515nm[11,12].

Cobalt(II) nitrate hexahydrate, $Co(NO_3)_2$. 6 H_2O is a red-brown crystalline compound with a monoclinic unit cell .The nitrates are very soluble in water, alcohols, and acetone.

It is formedby concentration a nitric acid solution of cobalt oxide or carbonate. Cobalt nitrate is anmajor source of high-purity cobalt for utilizein the electronics and related industries, and the compound has also found uses in the chemical and ceramics industries[13].

Photocatalyst is the semiconductor that is able to increase the efficiency of removal pollutants of organic and inorganic in water and wastewater[14,15].

In this paper a new couple of Nb_2o_5/CdS prepared and studied as a good photocatalytic using for degradation of $Co(NO_3)_2$.

Materials and methods:

Chemical materials used in this work is NiobiumpentoxideNb2O5, Cadmiumsulphide CdS and $Co(NO_3)_2$.

Preparation and characterization of couple Nb2O5/CdS

Prepared couple of Nb_2O_5/ Cds by the wet commix method, that involved using Nb_2O_5withCdS mixed powders as initial materials andadding 10ml of distilled water, which mixed by Magnetic stirrer hot plat three hours after that drying in the oven at 100^0c for one hour and calcination this mixture in Furnace at 800^0c for 4 hours. Thecouple of semiconductors Nb_2O_5/ CdS prepare was characterized by x-ray diffraction (XRD), FTIR spectroscopy (FTIR) .

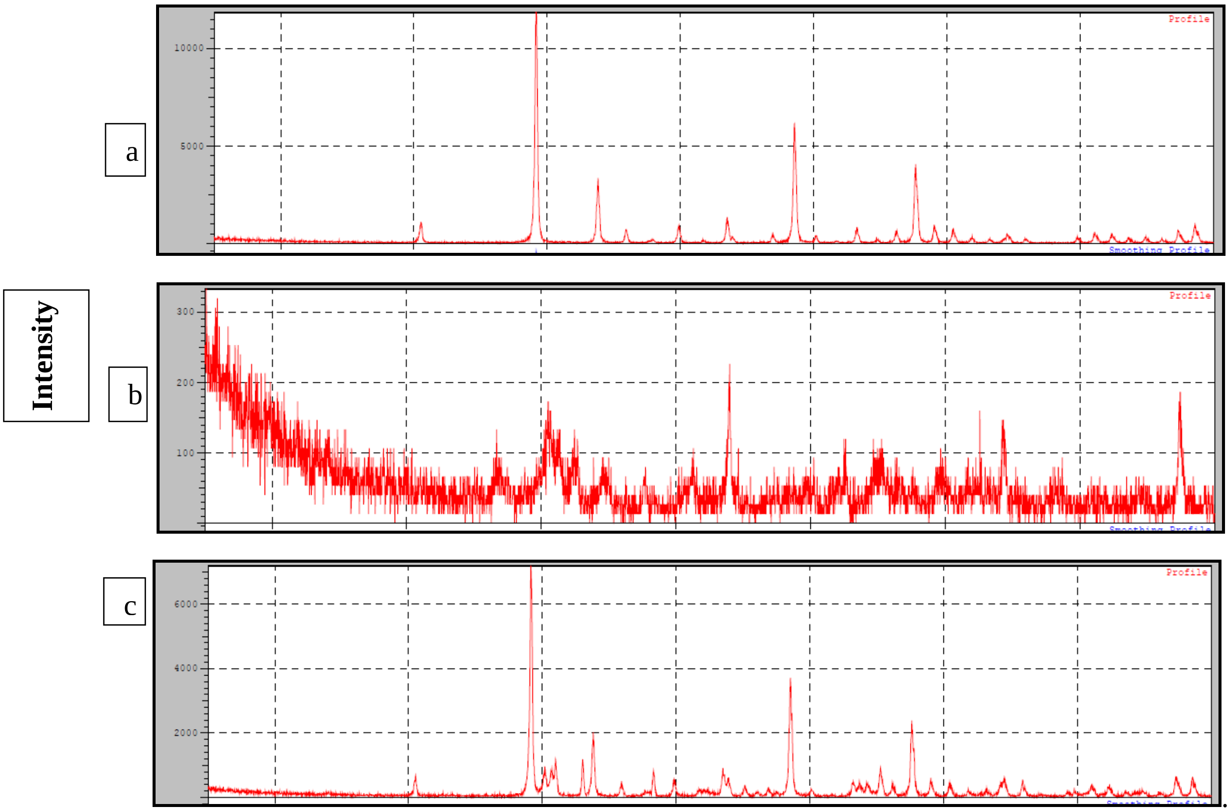
Result and Discussion

X-ray diffraction patterns

The Nb_2O_5 and CdS are characterized by x-ray diffraction(XRD), and compared with couple Nb_2O_5 /CdS. Figure 1.a. Find different peaks apparent in the shape of the spectrum represent 2θ at (29.1993 , 48.5727, 57.6540, 33.8265, 43.5250, 78.6291, 20.5662, 39.8886) back for initial material niobium pent oxide (Nb_2O_5).

Figure 1.b. Find different peaks apparent in shape of spectrum represent 2ɵ at (77.3992, 43.9213, 30.5576, 31.1819, 64.3029, 32.5703, 34.7182, 41.2730) back for initial material CdS.

While in figure1.c.for mixed (Nb_2O_5/CdS) find of different peaks ,at 2 Theta (29.1805,48.5498, 57.6326, 33.8154, 33.0343, 31.0088, 55.2794, 38.3226, 65.9035) notes appear peaks in spectrum at 2 Theta (33.0343, 31.0088, 55.2794 , 38.3226, 65.9035) not found in two initial material (Nb_2O_5 ,CdS)



2 Theta

Figure 1: X-ray diffraction spectrum of a. Niobium pentoxide Nb2o5 b. CdSc.CoupleNb2o5/CdS.

Table 1 : Particle Size of Niobium Oxide, CdS and Couple Nb_2O_5/CdS.

Catalyst	2Theta (deg.)	FWHM (deg)	Average Particle Size/nm
Nb_2O_5	29.1993 48.5727 57.6540 33.8265 43.5250 78.6291 20.5662 39.8886	0.22060 0.19910 0.20860 0.22800 0.21620 0.22400 0.22000 0.20660	37.25 43.86 43.58 36.47 39.60 46.04 36.86 41.00
CdS	77.3992 43.9213 30.5576 31.1819 64.3029 32.5703 34.7182 41.2730	0.44000 0.54000 0.61000 0.52000 0.44000 0.30000 0.32000 0.28000	23.17 15.87 13.50 15.87 21.35 27.60 26.05 30.39
Nb_2O_5\CdS	29.1805 48.5498 57.6326 33.8154 33.0343 31.0088 55.2794 38.3226 65.9035	0.20730 0.19960 0.20690 0.20280 0.16050 0.19520 0.19780 0.17160 0.18200	39.71 43.72 43.86 41.12 51.91 42.38 45.44 49.14 52.10

Calculation of the average crystallite size using the Debye–Scherer equation

results are shown in table 1.

$D= K \lambda/ß \cos\theta$

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.

θ = is Bragg angle

By using Scherrer equation that were calculated crystal size for (Nb2O5, CdS) and prepared couple of Nb2O5/CdS with ratio 0.85:0.15 from table 1 find theaverage particle size for couple Nb2O5\CdS is (45.48nm),CdS is (21.72nm), and Nb2O5 is (40.58nm),they are smaller than (100 nm) in Nano scale.

Fourier Transition for Infrared spectrum (FT-IR)

Study of the double prepared catalyst was achieved by using Fourier Transform Infrared (FTIR). All spectra were recorded at the wavenumber ranged from 400-4000 cm-1.

figure 2.a is characteristic of the sample niobium pentoxide Nb2o5 that show the peaks at (435.91, 466.77, 565.14, 893.04, 1132.21, 1321.24, 1436.97, 1481.33, 1523.76, 1558.48, 1595.13, 1664.57, 1716.65, 3741.90)(3741.90) cm-1 return to absorption water band in the sample , and bending band at 1716.65 cm-1 while the spectral appears the vibration modes assigned to Nb-O in the spectral range (910-850) cm-1 for Nb-O[16].

Figure 2.b. Appear the peaks at (499.56, 597.93, 1109.07, 1390.68, 1425.40, 1587.42, 1602.85, 1764.87, 2243.21, 2436.09, 2908.65, 3427.51, 3485.37,).the peaks (3485.37and 3427.51) cm-1 are returning to stretching and bending vibration for two water band these peaks indicate the hydroscopic character of the sample (cds) Figure2.c. Appear the peaks at (422.41, 470.63, 565.14, 893.04, 999.13, 1058.92, 1126.43, 1226.73, 1516.05, 1548.84, 1600.92, 1653.00, 1707.00, 1774.51, 2362.80).the peak at 1600.92 cm-1 also can see in spectrum of CdS but in the couple.

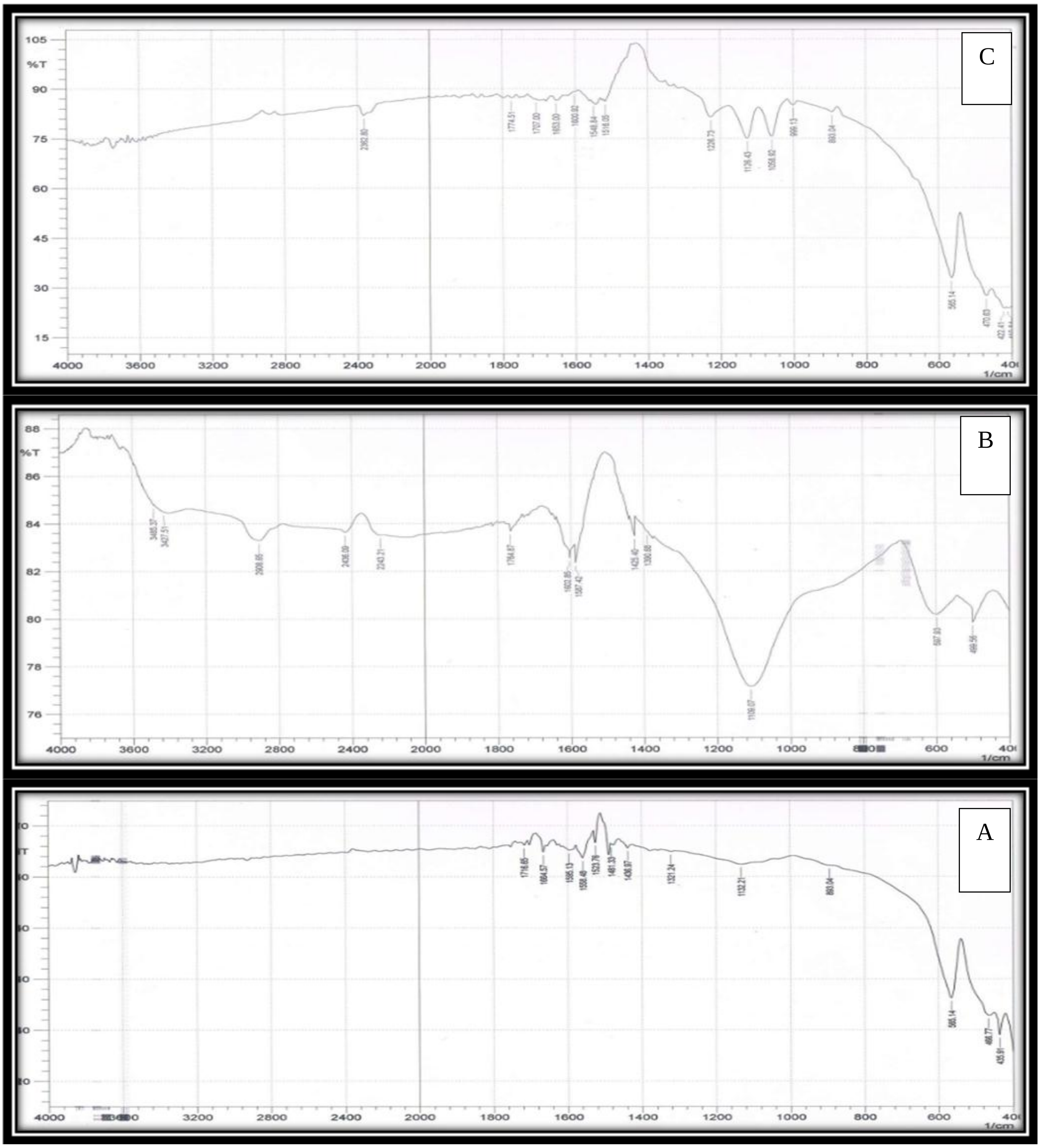


Figure 2: FTIR Spectrum for A. Niobium pentoxide (Nb2o5) B. CdS and C. Couple of Nb_2O_5\CdS.

Peaks CdS	**peaks Nb2O5**	**Peaks Nb2O5\CdS**
499.56	435.91	422.41
597.93	466.77	470.63
1109.07	565.14	565.14
1390.68	893.04	893.04
1425.40	1132.21	999.13
1587.42	1321.24	1058.92
1602.85	1436.97	1126.43
1764.87	1481.33	1226.73
2243.21	1523.76	1516.05
2436.09	1558.48	1548.84
2908.65	1595.13	1600.92
3427.51	1664.57	1653.00
3485.37	1716.65	1707.00
	3741.90	1774.51
		2362.80

Photocatalytic experiments

Effect of the mass couple Nb2o5/ CdS on the photodegradation of Co(NO3)2

Different masses use of catalyst and The effect on the photodegradation efficiency of $Co(NO_3)_2$ was studied by taking different masses of catalyst ranged from (0.05, 0.1,0.15, and 0.2) and 2000 ppm of $Co(NO_3)_2$ solution under UV light at 23 ^{0}C for 60 min . The obtained results are shown in Figure (3), these results showed that there was increased in the activity of Co(NO3)2 elimination as the masses of the used materials was increased. This probably emerges from the increase in the number of active sites available on the surface of catalyst for the reaction as the amount of the catalyst was increased. For high amount of the catalyst more than 0.1g the photodegradation efficiency was decreased due to an agglomeration that is causing the particle size is increased and decreased in specific surface area which leads to decrease in the number of surface active sites[17,18]. Also high amounts of catalyst lead to increase of light scattering . This tends to decrease the passing of irradiation through the sample. Therefore, the most effective photodegradation of Co $(NO_3)_2$ was observed with 0.1 g of catalyst weight[19,20].

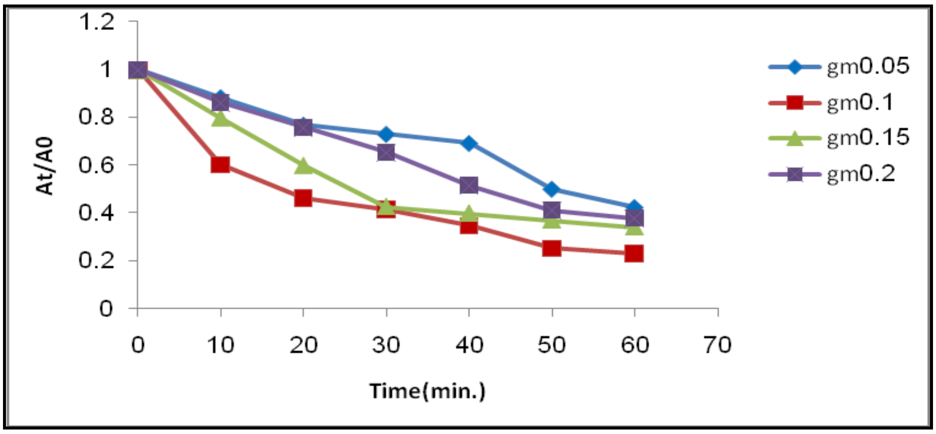


Figure 3:.The Effectof The Weight of Couple Nb_2O_5/Cdson Photodegradationof $Co(NO_3)_2$

Effect of concentration of$Co(NO_3)_2$

Used Different concentrations of $Co(NO_3)_2$ (500, 1000, 2000, 2100 ppm) with 0.10 g of catalyst. In addition, at 296.15 K.

The results show decreased ofthephotocatalytic degradation process when increased initial concentration of $Co(NO_3)_2$ because molecules of $Co(NO_3)_2$ are photosensitive and when concentration increased more photons would be absorbed that lead to low light transmittance and decreased depth of light passage[21,22].

From these results, the optimum initial concentration of $Co(NO_3)_2$ is (500ppm) because it results higher photocatalytic degradation than other concentrations of $Co(NO_3)_2$.

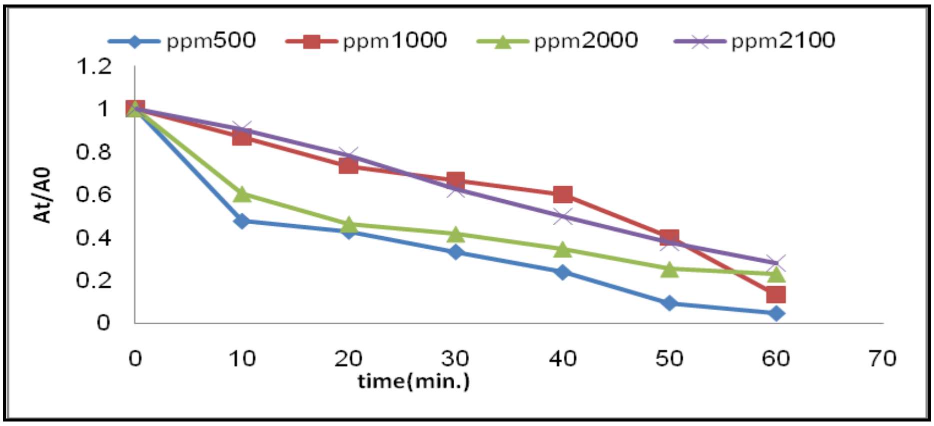


Figure4: Effect of the concentration of $Co(NO_3)_2$

Effect of temperature

Study the effect of temperature on Photocatalytic degradation rate of $Co(NO_3)_2$ using catalyst at different temperature ranging from (15-30C^o)[20]. Figure5, shows the effect of temperature on the photo catalytic degradation rate of $Co(NO_3)_2$ at a fixed initial concentration500 ppm and 0.1gmNb_2O_5\CdS catalyst . Andshow that the photo catalytic degradation rate of $Co(NO_3)_2$increases with temperature increase because increase temperature cause to increase generate free radicals and this lead to decrease in recombination process.

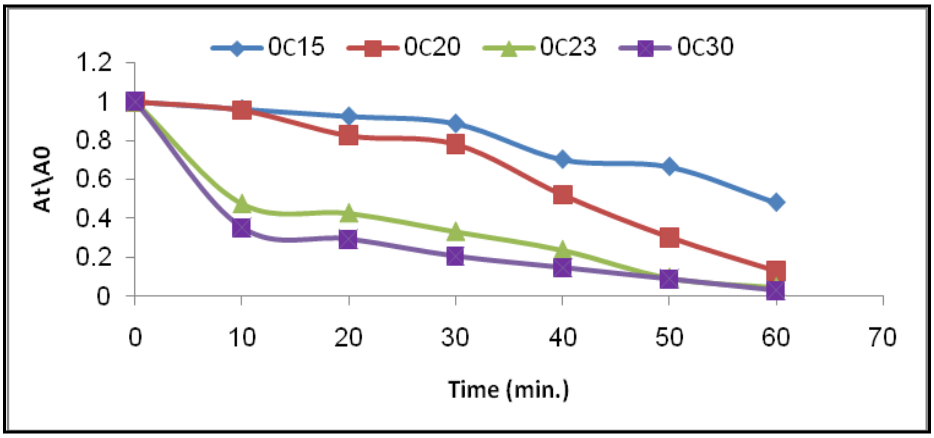


Figure.5.The change of (At / A0) with irradiation time at different temperature

Effect of pH

The photocatalytic degradationrate of $Co(NO_3)_2$ is highly influenced by the value of pH of the reaction mixture. The photodegradation efficiencies of $Co(NO_3)_2$ with different pH values for reaction mixture are shown in Figure (6). The degradation efficiency of the $Co(NO_3)_2$ was decreased with the increase in pH and the highest $Co(NO_3)_2$ degradation efficiency at pH= 4. Then the increasing of pH value of reaction mixture to 8 leads to decrease in photodegradation efficiency. The reduction in the efficiency of $Co(NO_3)_2$ removal at high pHs values can be attributed to the repulsion forces that are initiated between the negatively charged surface and the anionic groups that are present in $Co(NO_3)_2$ molecules . In addition to that, decrease in the photocatalytic activity of $Co(NO_3)_2$ removal can be due to increase in the rate of recombination between (e– /h+) pairs[24] .

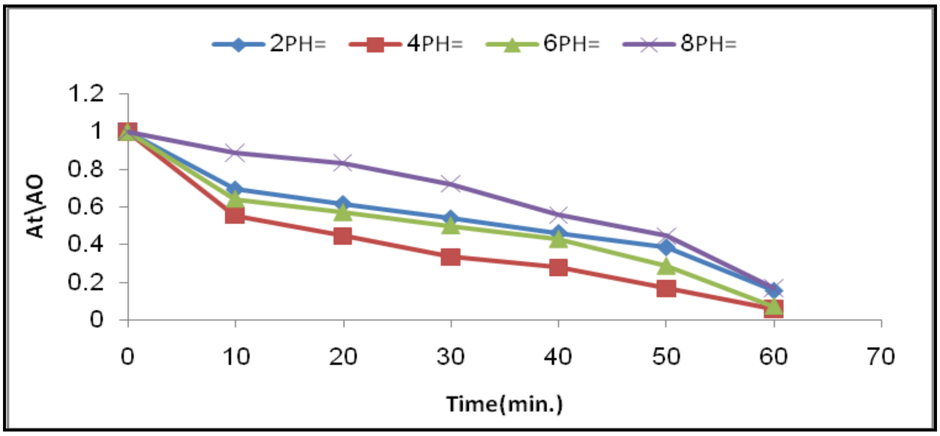


Figure 6: Shows effect of pH

Conclusion

Results showed that (0.85:0.15) percentage is more active than other percentage. The ability for photodegradation of $Co(NO_3)_2$74.46 % in the optimumcondition amount of couple 0.1 g, concentration of salt 500 ppm, temperature 30^{O}C ,PH=4 ,andradiation time 60 min.

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