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TREATMENT OF TEXTILE DYE WASTEWATER USING ADVANCED OXIDATION PROCESS (AOP)

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ABSTRACT

Photocatalytic processes is used for improving conventional water treatment processes. The successful and efficient application of photocatalysis depends on quality of photocatalyst, nature of pollutants, and source of light, which should be in close contact with each other. Photodegradation percentage was followed spectrophotometrically by the measurements of absorbance. These advanced treatment methods are capable of removing many pollutant chemicals as well as pathogenic microorganisms. The objective of this work is to study the performance of small scale UV/TiO_2 & CO-TiO_2 photocatalytic process in treating direct dye & acetate red dye. Preparation of TiO_2 & CO-TiO_2 catalyst is done by using sol gel method. The TiO_2 & CO-TiO_2 photocatalyst is used for the effluent treatment of textile wastewater (Direct dye black, acetate red) in the presence of ultraviolet (UV) irradiation. Heterogeneous UV-TiO_2 photocatalysis is capable to remove organic pollutants from textilewastewater.

INTRODUCTION

Textile industries are one of the major environmental pollution problem creators in the world, as it releases unwanted dye effluents. Textile wastewater contains dyes mixed with different contaminants at a variety of ranges. Due to the large volume of effluents and the great variety of chemical composition, textile wastewater poses serious threats to downstream ecosystems and human health if discharged improperly. The highly colored component can impede passage of sunlight, inhibit the photosynthesis of aquatic plants, and affect the reoxygenation and self- purification of water bodies, thereby acting as a source of eutrophication. Advanced oxidation processes (AOPs) were first proposed in the 1980s for drinking water treatment and later were widely studied for treatment of different wastewaters *Yang deng & Renzunzhao et al (2015)*. The non-biodegradability of textile wastewater is mainly due to the presence of synthetic dyes Resistance to bacterial degradation has led to the development of new technologies in which photocatalysts appear to be the best method for this type of application *Nadia Chekir et al (2015)*. The degradation of methylene blue (MB) was studied in an aqueous suspension of TiO2 and a maximum degradation of 86% was achieved for concentration of 20 mg/l using 100 mg TiO_2 catalyst irradiated for 190 min in presence of UV light of wavelength 254 nm (Tallapaka et al., 2020).

Woottikrai, et al., 2023 created a mixed phase TiO_2 layer on glass beads for waste water treatment from textile dyeing. 93.7% dye removal achieved in 24hrs and optimal conditions for photodegradation were identified. The photocatalytic degradation of single (AO7, RG19) azo dye and binary (AO7 and RG19 mixture) azo dye aqueous solutions was photocatalyzed by commercial titanium dioxide (TiO2, P25) under solar light irradiation. Dilek Gumus & Feryal Akbal 2011 explored how titanium dioxide (TiO2) can break down a common dye (Remazol Red 133) using sunlight. They tested different factors like pH and TiO2 concentration. Higher TiO2 levels improved dye breakdown. In real textile wastewater, the process worked best at low pH, achieving high decolorization and degradation.

In this work TiO_2 & CO- TiO_2 photocatalyst is used for the effluent treatment of textile wastewater in the presence of ultraviolet (UV) irradiation. Heterogeneous UV-TiO_2 photocatalysis is capable to remove organic pollutants from textile wastewater. Direct dye black and acetate red of different concentrations were removed using TiO_2 & CO- TiO_2 photocatalyst. 86% of 40ppm Direct dye degradation is achieved in 180min and acetate red is 52%.

MATERIALS AND METHODS

MATERIALS:

Direct Dye (Black) & Acetate red dye purchased from Atul industries, Titanium (IV) Iso propoxide 98% purchased from Sigma Aldrich, 2-propanol 96% purchased from Sigma Aldrich, Distilled water, Ethanol 99% purchased from Sigma Aldrich, Cobalt chloride purchased from Molychem.

METHODS:

CATALYST PREPARATION

Sol-gel method was used for Preparation of photocatalysts

i) Titanium dioxide (TiO_2)

ii) Cobalt-doped TiO_2

Following photocatalyst is synthesized by sol-gel method

Preparations were taken from the work of Ajitkumar et al., 2022

Preparation of Titanium dioxide (TiO_2):

Sample A (30 ml titanium (IV) isopropoxide, as the raw material, mixed with 120 ml 2-propanol in a dry atmosphere). This mixture was drop wise added to another mixture consisting of sample B (30 ml water and 30 ml 2-propanol). After this the mixture was stirred at 450rpm for 2hours then a yellowish transparent gel was formed. The obtained gel was dried at 105^oC for 4hours until it turned into a powder form. The resulting powder was calcined at 500^oC for 6 hours in a furnace.

Synthesis of Cobalt doped TiO2 Nanoparticles:

Sample A (20 ml of titanium (IV) isopropoxide , as the raw material, mixed with 100ml of ethanol and stirred for 2hrs). Sample B (20m) Ethanol,0.3grm of cobalt chloride,2ml HNO3 and 2ml distilled waters), this mixture was drop wine added to sample A solution and stirred for 12hrs, gel was formed then dried as 80°C for 12hrs turned into a powder form. The resulting powder was calcined at 300C for 4 hrs.

Characterization of Prepared Catalysts:

X-Ray diffraction analysis technique:

Prepared TiO_2 catalyst powder shows major peeks at 2θ = 25.3^o, 26.1^o, 37.0^o and 48.0^0 these values attribute to anatase phase and minor peaks at 2θ=27.5^o, 31.5^o and 52.5^0 it shows rutile phase, anatase to rutile ratio 83:17 (Figure 1).

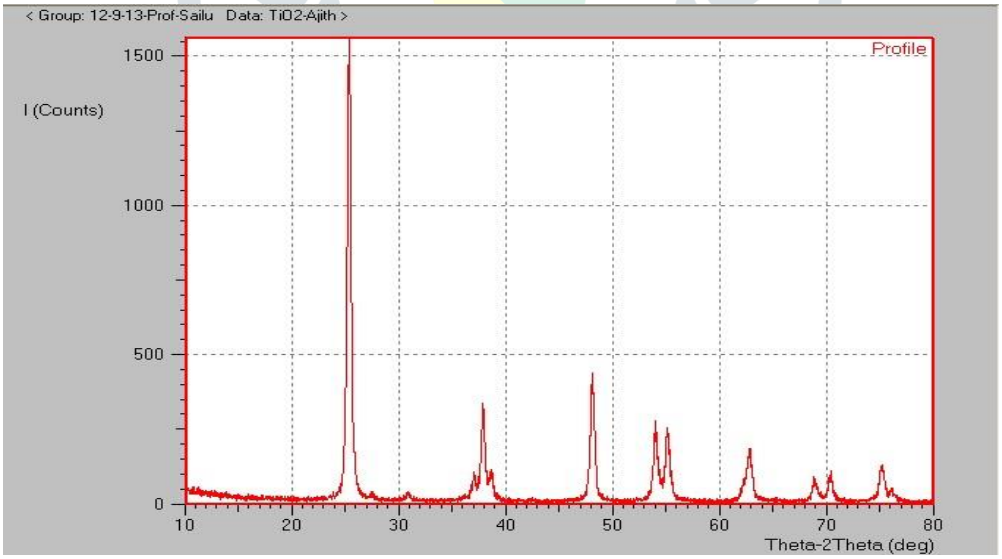


Figure 1 : XRD graph of TiO_2

Scanning electron microscope

Obtained catalyst has been characterized using SEM and the morphology is given below:

From Figure 2 it is observed that Prepared TiO_2 catalyst surface is nonporous and size of the particles range between (156-382 nm). this technique is confirmed prepared TiO_2 catalyst is Nano catalyst.

Figure 2: SEM image of (TiO_2) Nano catalyst

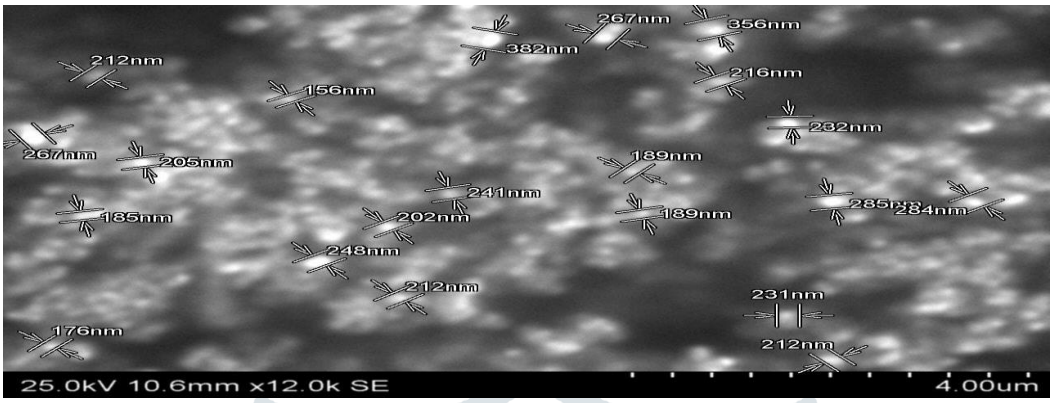
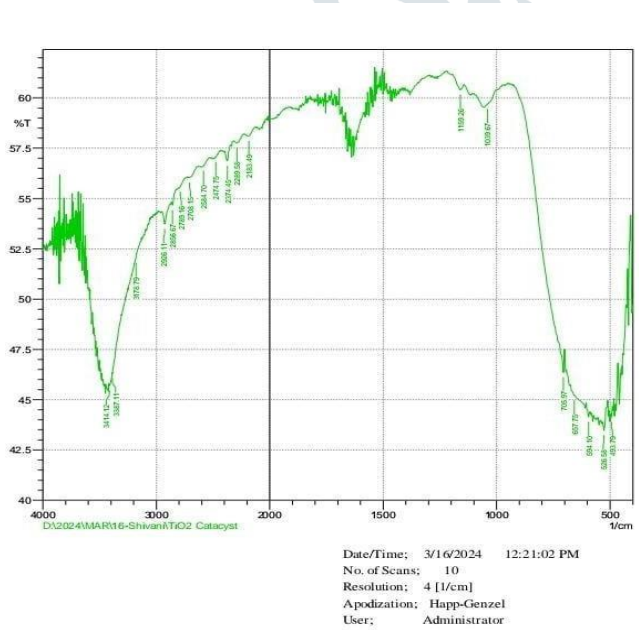


Figure 3: SEM image of Cobalt-doped TiO_2

SEM Analyzer is used to characterize the surface morphology of Cobalt-doped TiO_2 catalyst (Figure 3). In SEM analysis results, the image confirms that the sample is nonporous and agglomerated with the crystal phases.

FTIR: FTIR is employed to study the chemical composition and surface functional groups of catalysts. It detects molecular vibrations associated with chemical bonds present in the catalyst. FTIR spectra reveal characteristic peaks corresponding to functional groups like -OH, -NH, C=O, and C-H. Analysis of FTIR spectra provides insights into catalyst surface chemistry, adsorption sites, and interactions with reactant molecules.



SHIMADZU

	Peak	Intensity	Corr. Inte	Base (H)	Base (L)	Area	Corr. Are
1	493.79	44.135	0.282	495.72	489.94	2.044	0.008
2	526.58	43.469	0.734	534.3	516.94	6.212	0.064
3	594.1	44.147	0.402	605.67	588.31	6.123	0.039
4	657.75	45.202	0.071	675.11	655.82	6.615	0.015
5	705.97	46.35	1.388	925.86	702.11	60.418	0.793
6	1039.67	59.641	0.072	1043.52	1003.02	8.991	0.016
7	1159.26	60.4	0.419	1195.91	1143.83	11.293	0.081
8	2183.49	58.105	0.029	2185.42	2127.55	13.546	0.009
9	2289.58	57.757	0.081	2297.3	2233.64	15.09	0.031
10	2374.45	56.862	0.598	2393.74	2343.59	12.158	0.107
11	2474.75	57.044	0.047	2478.61	2443.89	8.427	0.008
12	2584.7	56.606	0.022	2586.63	2549.98	9.014	0.006
13	2708.15	56.031	0.099	2717.79	2642.57	18.805	0.052
14	2789.16	55.57	0.021	2791.09	2750.58	10.266	0.002
15	2856.67	54.662	0.285	2864.39	2825.81	9.995	0.027
16	2926.11	53.722	0.732	2947.33	2875.96	18.954	0.162
17	3178.79	52.012	0.041	3180.72	3140.22	11.332	0.001
18	3387.11	46.298	0.102	3389.04	3306.1	26.561	0.012
19	3414.12	45.69	0.181	3417.98	3390.97	9.135	0.047

Figure 4: FTIR spectra of Cobalt-doped TiO_2 catalyst

Estimation of % degradation:

Following the experiments, a residual concentration analysis was conducted to assess the percentage degradation of textile dye effluents utilizing a UV-Vis spectrophotometer, based on the absorbance peaks recorded by the spectrophotometer. The percentage degradation was determined as follows:

$$\%\text{Degradation} = [(C_i - C_f) / C_i] \times 100$$

Where C_i - initial concentration and C_f - final concentration.

Preparation of dye solution

The stock solution is prepared using volumetric method. The concentration of stock solution is part per million (1mg/lit) distilled water.

Figure 5: Direct Black dye and Acetate Red dye solutions

Experimental procedure:

Batch reactor system

The experimental setup of a Batch reactor which consists of a borosilicate 100ml cylindrical beaker, it was kept on a magnetic stirrer at 300rpm at 25^0C under UV-light short wave (254nm).

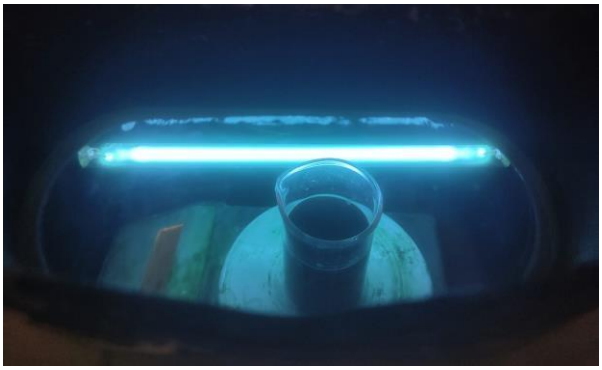


Figure 6: Batch reactor setup

Figure 7: Degradation of Direct Black dye with Tio_2 catalyst Dosage 25mg, 50mg,75 mg

RESULTS AND DISCUSSION:

The dye effluent is treated in the reactor for three hours and samples were drawn every half an hour. The concentration of dye determined using spectrophotometrically to find out concentration at respective times. The degradation percentage was calculated and analyzed in relation to the passage of time.

Table 1: Effect of TiO_2 catalyst for Degradation of direct dye (black)

Time (min)	Concentration			% Degradation		
	25 mg TiO_2	50 mg TiO_2	75 mg TiO_2	25 mg TiO_2	50 mg TiO_2	75 mg TiO_2
0	40	40	40	0	0	0
30	33.03	30.38	23.93	17.42	24.3	40.175
60	29.32	25.41	18.74	26.7	36.47	53.15
90	27.71	19.70	14.64	30.72	50.75	63.39
120	26.30	18.93	9.320	34.25	52.75	76.7
150	25.66	10.85	7.20	35.85	72.87	82
180	25.21	8.80	5.538	36.97	78	86.11

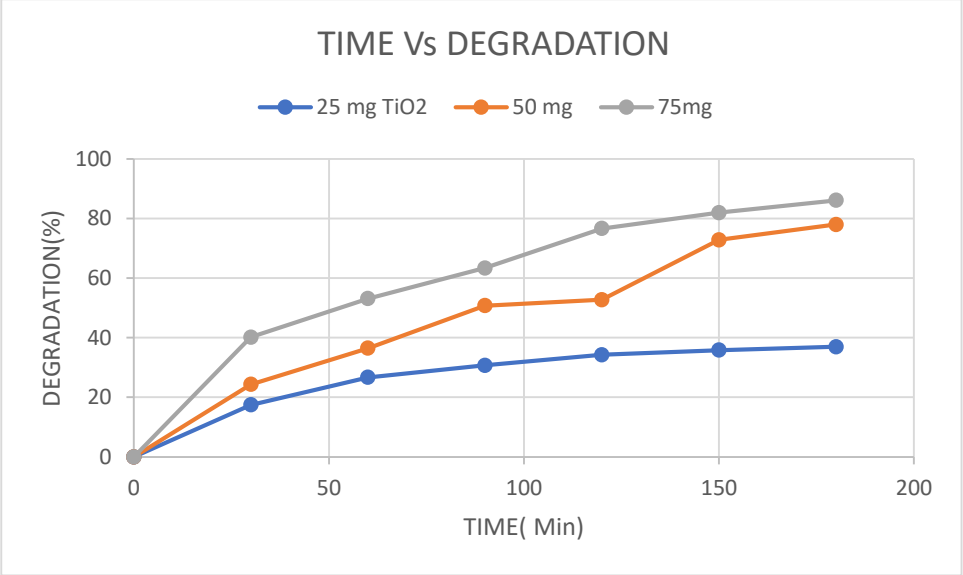


FIGURE 8: % Degradation of Direct Dye of 40 ppm with TiO_2 catalyst

From Figure 8 and Table 1 it can be observed that the photocatalytic activity of the TiO_2 catalyst at a dosage of 40 ppm concentrated Direct Black Dye is demonstrated. The percentage of degradation varies with time, indicating that as time progresses, the percentage of degradation also increases. The degradation percentage of the Direct Black Dye rises with an increase in catalyst dosage over time, ultimately reaching 86.1% degradation after a duration of 3 hours.

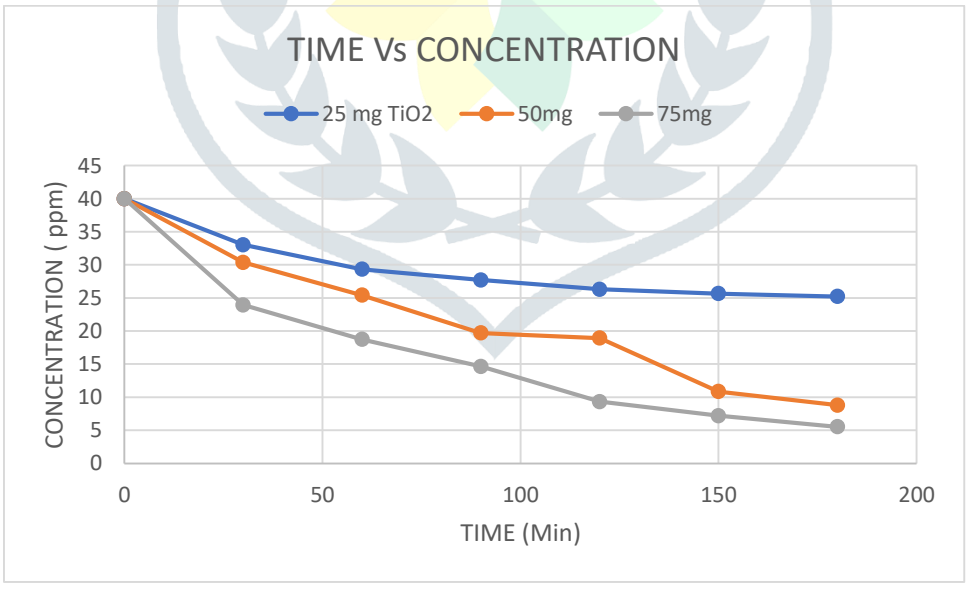


Figure 9: Concentration of Direct Dye of 40 ppm with TiO2 catalyst

From Figure 9 the photo-catalytic activity of the TiO_2 catalyst at a dosage in 40 ppm concentrated Direct Black Dye is demonstrated. The concentration values fluctuate over time, indicating that as time progresses, the concentration diminishes. The concentration of Direct Black Dye decreases

with an increase in catalyst dosage over time. The initial concentration of the dye, which is 40 ppm, reduces to 5.5 ppm as both catalyst dosage and time increase

Table 2: Effect of TiO_2 catalyst for Degradation of Acetate Red:

Time (min)	Concentration			% Degradation		
	25 mg TiO2	50 mg TiO2	75 mg TiO2	25 mg TiO2	50 mg TiO2	75 mg TiO2
0	40	40	40	0	0	0
30	39	38	37	2.5	5	8.5
60	37	34	34	7.5	15	18
90	35	31	32	12.5	22.5	22
120	32	29	26	20	27.5	35
150	30	27	19	25	32.5	52
180	27	25	15	30	32.4	65

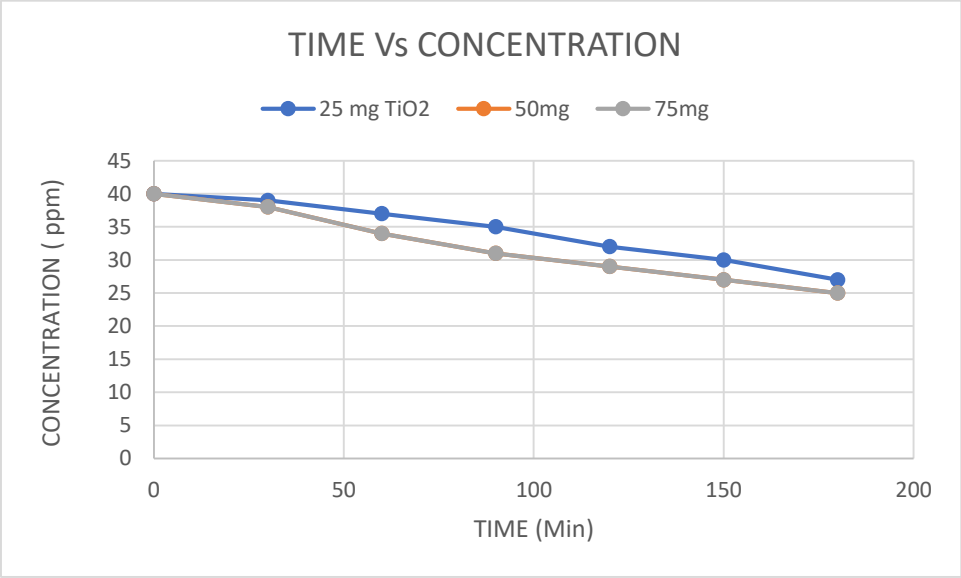


Figure 10: Concentration of acetate red Dye of 40 ppm with TiO_2 catalyst

From Figure 10 photo-catalytic activity of the TiO_2 catalyst was evaluated at a dosage in a solution containing 40 ppm of Acetate Red Dye. The concentration values over time indicated that as time progressed, the concentration diminished, suggesting that further reduction may occur with extended

duration. Additionally, the concentration of Acetate Red Dye decreased from an initial level of 40 ppm to 15 ppm as the dosage of the catalyst was increased.

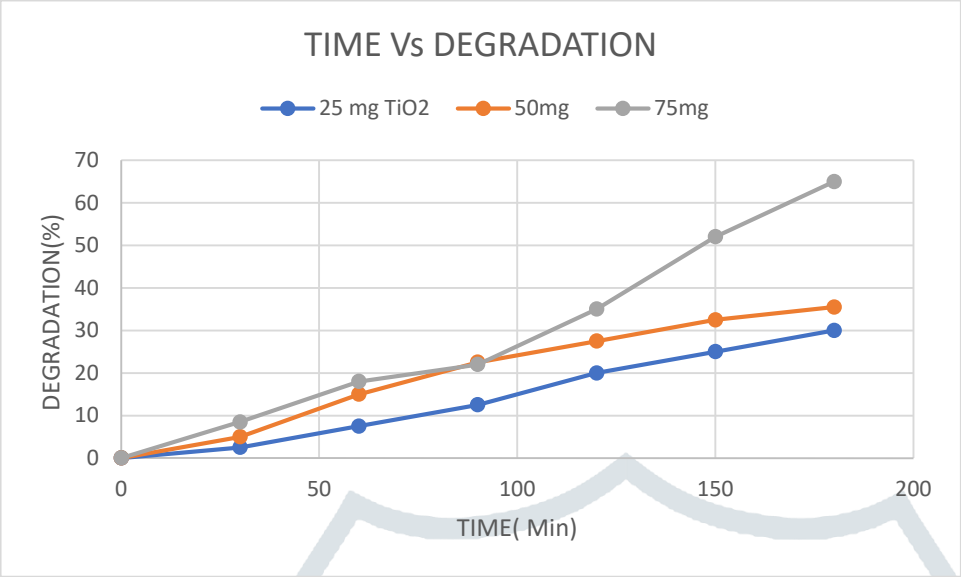


Figure 11: Degradation of acetate red Dye of 40 ppm with TiO_2 catalyst

Figure 11 The photo-catalytic activity of the TiO_2 catalyst at a dosage of 40 ppm concentrated Acetate Red Dye is demonstrated. The values of percentage degradation over time indicate that as time progresses, the percentage of degradation also increases. The degradation percentage of Acetate Red Dye rises with an increase in catalyst dosage, achieving only 65% degradation over a duration of 3 hours.

Table 3: Effect of Cobalt doped TiO2 catalyst for Degradation of direct dye (black):

Time (min)	Concentration		% Degradation	
	50 mg TiO_2	75 mg TiO_2	50 mg TiO_2	75 mg TiO_2
0	40	40	0	0
30	40	40	1.2	7.2
60	39.5	37.1	3.2	11
90	38.7	35.5	8.5	18
120	36.6	32.8	10	25
150	36	30	14.7	32
180	34.1	27	26	45

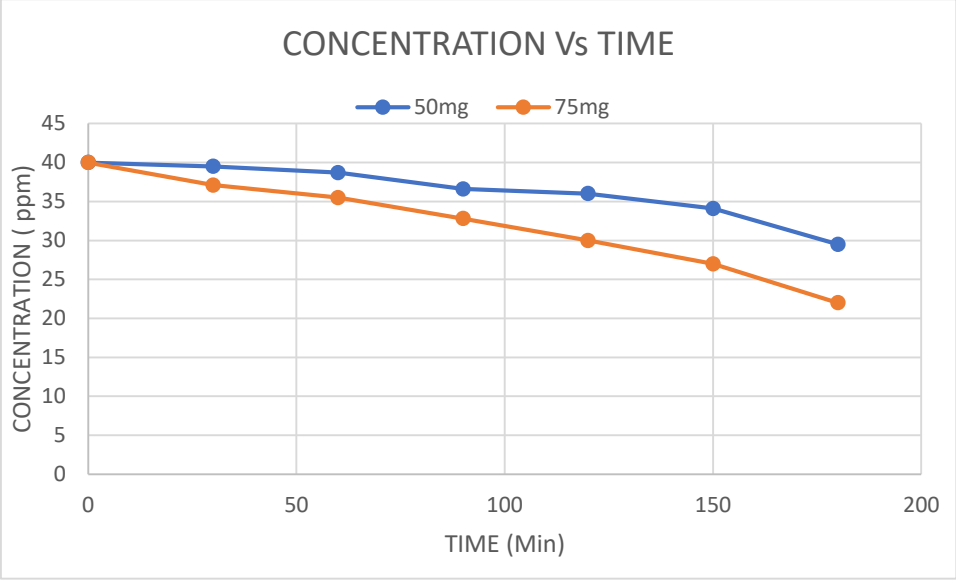


Figure 12: Concentration of Direct Black Dye of 40 ppm with Cobalt doped TiO_2 catalyst

Figure 12 Shows the photo catalytic activity of Cobalt doped TiO_2 catalyst dosage in 40 ppm concentrated Direct Black Dye . The concentration values of Direct Black dye exhibit minimal variation over time with respect to the dosage of cobalt-doped TiO_2 catalyst. .

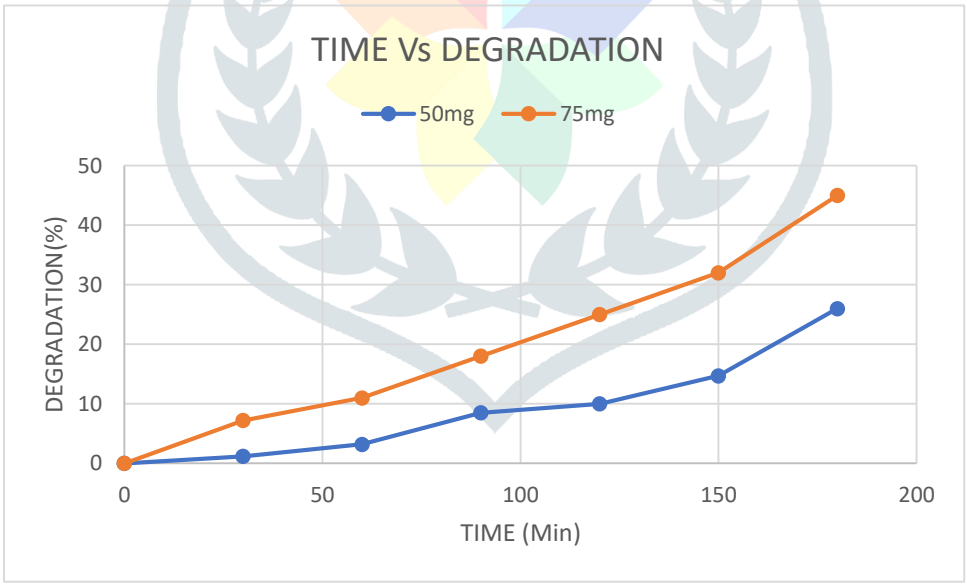


Figure 13: Degradation of 40 ppm Direct Black dye with Cobalt doped TiO2 catalyst

The photo-catalytic performance of the Cobalt doped TiO_2 catalyst was evaluated at a concentration of 40 ppm of Direct Black Dye (Figure 13). The percentage of degradation exhibited an upward trend over time, with only a slight enhancement in degradation observed as the catalyst dosage increased. Notably, the degradation percentage of Direct Black Dye reached a maximum of 46% after a period of 3 hours.

CONCLUSIONS:

TiO_2 and Cobalt-doped TiO_2 catalysts are synthesized using the sol-gel method, followed by characterization through techniques including X-ray diffraction (XRD), scanning electron microscopy (SEM), and Fourier-transform infrared spectroscopy (FTIR) to enable heterogeneous photocatalysis.

The investigation into the degradation of Direct dye has revealed that the degradation percentages for a concentration of 40 ppm, when utilizing varying dosages of TiO2 catalyst at 25 mg, 50 mg, and 75 mg, are 36.97%, 78%, and 86.11%, respectively.

The degradation of Acetate red dye has been investigated, revealing that the degradation percentages for a direct dye concentration of 40 ppm, when utilizing varying dosages of TiO2 catalyst (25 mg, 50 mg, and 75 mg), are 25%, 32.5%, and 52%, respectively. %Degradation of dye water is increasing as catalyst dosage is increasing.

The observation that absorbance values do not decrease when co-doped TiO2 is subjected to treatment with direct dye leads us to conclude that CO-doped TiO2 exhibits limited efficiency in the removal of direct dye.

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