Idea Lab IDEA-2015-CH-01
Photocatalysis

Idea Lab Project

Submitted By
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Under the mentorship of
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Contents

Report

Declaration

We do hereby declare that the technical project report submitted is original, and is the outcome of the independent research carried out by us and contains no plagiarism. The research is leading to the discovery of new techniques of scientific facts already known. This work has not been submitted to or supported by any other University or funding agency.

We do hereby further declare that the text, diagrams or any other material taken from other sources (including but not limited to books, journals and web) have been acknowledged, referred and cited to the best of our knowledge and understanding.

Date:

Place:

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NIRMA UNIVERSITY
INSTITUTE OF TECHNOLOGY
IDEA LAB
CHEMICAL ENGINEERING DEPARTMENT

1. Idea Lab Project ID: IDEA-2015-CH-01

2. Project Title: Photocatalysis

3. Period of Project: 05/10/15 to 23/04/2016

4.
   a) Name of Student (Roll No.): Brijesh Saparia (13BCH012)
      Department: CHEMICAL

   b) Name of Student (Roll No.): Vishal Patel (13BCH045)
      Department: CHEMICAL

5. Project Start Date: 05/10/15

6. (a) Total Amount Approved: Rs. 20,000/-

   (b) Total Expenditure: Rs. 19846.24/-

   (c) Report of the work done:

      i. Brief objective of the project:

      The main objective is to prepare a photocatlyst which can efficiently remove the color of the dye and degrade it by using solar light. But the energy of visible radiation is less and UV radiation being limited in the solar spectrum, we need to develop a catalyst which efficiently works in the visible region. Due to the band gap limitation of a single photocatayst, this is not practically possible. So to achieve this we need to prepare a composite photocatalyst which can work efficiently in the visible region too and can degrade the dye molecule.

      ii. Work done:

      We have prepared composite catalyst with composition 100% Ag_2CO_3, 94% Ag_2CO_3-06% SiC, 91% Ag_2CO_3-09% SiC, 88% Ag_2CO_3-12%SiC, , 85%

Ag_2CO_3-15%SiC and 82% Ag_2CO_3-18%SiC. And performed experiment for color removal of dye (methylene blue).

iii. Results achieved from the work :

From the above recorded observations, we conclude that amongst the composite catalysts prepared, the best results in terms of dye degradation were shown by 9% Sic-91%Ag2CO3. It is also seen that this composite catalyst works even better than 100% TiO2 and 100% Ag2CO3 and 100% SiC. This proves the formation of the z-scheme photocatalyst.

iv. Has all the objectives been achieved as per plan:

Yes

v. Technical difficulties experienced in implementing the project :

Changing intensity of sunlight, Filtering, Sampling,etc

vi. Summary :

So, by preparing Z-scheme composite catalyst we can effectively degrade industrial dye effluent which contains dye stuff in that by using sunlight. It can work more efficiently than single photocatalyst.

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1.1 Introduction

Industrial effluents always cause a major environmental issue. Dyes are extensively used in textile and printing industry. Ever increasing growth of industrialization and urbanization causes gigantic problem of environmental pollution. An industry consumes large quantity of water for their processes. The effluents from dye industry are highly colored, toxic and carcinogenic. The textile industries are responsible for pollution; large amount of waste water is released through cloth dyeing and washing processes. The effluents from these industries are highly organic and toxic in nature with non bio degradable properties. Removing color from wastes is often more important than other colorless and organic substances, because the presence of small amount of dyes (below 1 ppm) is clearly visible and influences the water environment considerably. Therefore, it is necessary to find an effective method of wastewater treatment in order to remove color from textile effluents.[1]

This invariably corresponds to a release of about 615 tons per day into the environment and ecological system. Due to their toxicity and recalcitrance, these dyes are hazardous to the environment and even when they are present in very low concentrations, can present serious carcinogenic effects.[1]

1.1.1 Textile dyes and environmental concerns[2]

Although dyes constitute a small portion of the total volume of waste discharged in textile processing, color removal from effluent is a major problem for textile industry because of several reasons:

1. The presence of even a small fraction of dyes in water is highly visible due to high tinctorial value of dyes and affects the aesthetic merit of streams and other water resources.

2. Most dyes have complex aromatic structure resistant to light, biological activity,ozone and other degradative environments and hence not readily removed by typical waste treatment processes.

3. Possible long term effects of a few dyes and dye degradation products are becoming of increasing concern. The possible mutagenic, carcinogenic and allergenic effects of dyes have been established.

4. Unless and otherwise properly treated dyes can significantly affect photosynthetic activity in aquatic life due to reduced light penetration and may also be toxic to certain forms of aquatic life due to presence of metals and chlorides in them. There is a little evidence that dyes found in water courses are toxic to fish and other wild life at the concentrations likely to be present. Most of the studies on toxicity of dyes and pigments are concerned with the hazards due to occupational exposure of employees to dyes in the user industry.

5. Dyes have also been known to interfere with certain municipal waste water treatment operations i.e UV disinfection etc.

1.1.2 Advantage of Advanced Oxidation Process over other Conventional Process[3],[1]

This a modern process used for the water treatment and colour removal. Advanced oxidation processes (AOPs) are therefore mostly based on the generation of highly reactive radical species (especially the hydroxyl radical HO**.**) that can react with a wide range of compounds, including compounds that are otherwise difficult to degrade, e.g. dye molecules.

Photocatalysis, which is one of the Advanced Oxidation Processes, is a new method used to mineralize dye compounds . The utilization of photocatalysis has been attracting increasing attention. It may be developed to a useful process for the reduction of water pollution caused by dyeing compounds because of their mild conditions required and their efficiency in the mineralization. It also has the potential ability to oxidize most of the organic contaminants to CO_2 and H_2O

1.2 Literature Survey

Phtocatalysis, the term can be generally used to describe a process in which light is used to activate a substance the photocatalyst which modifies the rate of chemical reaction without being involved itself in the chemical transformation.

Semiconductores are particularly useful as photocatalyst because of a favourable combination of electronic structure, light absorption properties, charge transport characteristics and excited state lifetime. In semiconductor photocatalysis excitation of an electron from the valence band to conduction band is accomplished by absorption of a photon of energy equal to or higher than the band gap energy of the semiconductor. This light induce generation of an electron-hole pair is a pre-requisite step in all semiconductor mediated photocatalytic processes.

Interfacial electron transfer i.e. transfer of an electron to or from substrate adsorbed onto the light activated semiconductor is probably the most step in photocatalytic process and efficiency determines to a large extend the ability of the semiconductor to serve as photocatalyst for given redox reaction. The efficiency of electron transfer reaction is, in turn a function of the position of semiconductor's conduction and valence bond-edges relative to the redox potentials of the adsorbed substances. For a desired electron transfer reaction to occur , the potential of the electron acceptor species should be located below(more positive then) the conduction band of the semiconductor , whereas the potential of the electron donor species should be located above(more negative than) the valence band of the semiconductor.

1.2.1 Mechanism[5],[6]

The steps involved in visible light assisted photocatalytic reactions are explained below.

1. Separation of charge carriers by photons absorption of photocatalyst.
   When any photocatalyst is subjected to a radiation of suitable frequency, it absorbs the radiation and the electron hole pairs are created due to energy gain and excitation.
   $$A + h\upsilon \rightarrow A(e^{-}_{CB} + h^{+}_{VB})$$
2. Migration of photo-excited electrons and holes through surface of the photocatalyst. Due to instability the electron from the conduction band gets transferred to the valence band which generated the electron hole pairs.

3. Generation of intermediate species such as hydroxyl radical ($OH^{\bullet}$), superoxide anion radical ($O_2^{\bullet-}$) and hydrogen peroxide (H_2O_2). As the band gap energy of lies in the visible region, the catalyst can harvest photon energy efficiently from the naturally available solar source and can then react with the dye molecule to degrade it.

$$e^-_{CB} + O_2 \rightarrow O_2^{\bullet-}$$

$$h^+_{VB} + OH^- \rightarrow OH^{\bullet}$$

$$O_2^{\bullet-} + H_2O \rightarrow HO_2^{\bullet} + OH^-$$

$$HO_2^{\bullet} + H_2O \rightarrow OH^{\bullet} + H_2O_2$$

$$H_2O_2 \rightarrow 2OH^{\bullet}$$

$$OH^{\bullet} + Dye \rightarrow CO_2 + H_2O$$

$$h^+_{VB} + Dye \rightarrow \text{Degradation Products}$$

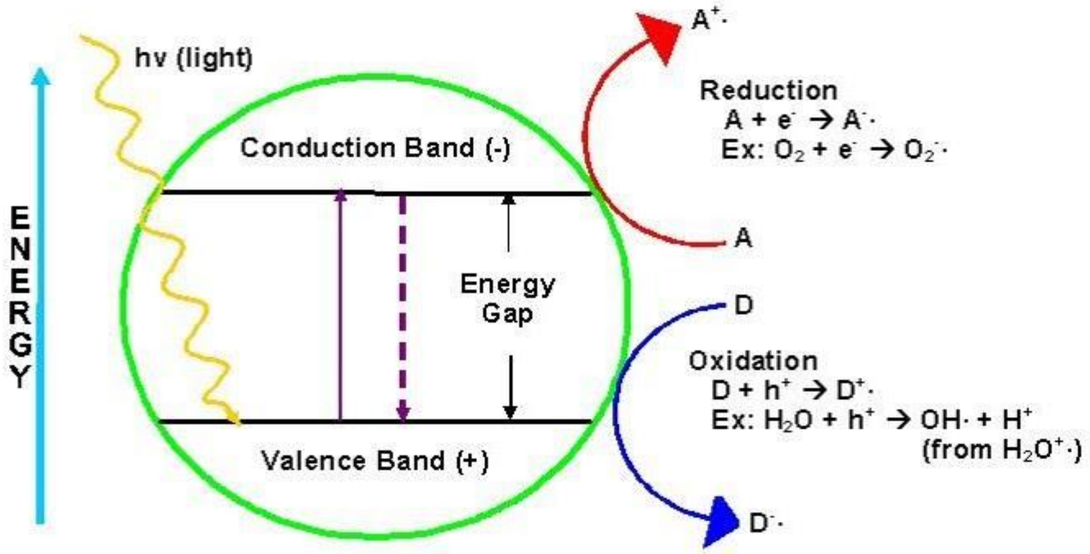


Figure 1 Photocatalyst Mechanism[7]

1.2.2 Solar Photocatalysis [8]

The problem with the photocatalytic process is that when the natural sunlight is being used it consists of a large variety of wavelengths but most of the catalysts which are used have a large band gap which requires high energies and thus small wavelengths are needed. But the UV radiation from the sun's radiation is only about 5 %. Due to this the rate of degradation of the dye molecules is slow. But if the band gap of the catalyst is small then it can work well with the radiation of low energy. According to the equation

$$E = \frac{hc}{\lambda}$$

If the energy required is low it means that the wavelength of that radiation is large. So we can efficiently use the visible region of the solar radiation. So there is a need to develop a efficient catalyst with a small bandgap and which can used in the visible region.

The potential required for the oxidation of hydroxide radical to hydroxyl radical is 2.4V. So the potential of the conduction band of the catalyst should be more than 2.4V. Similarly the potential required for the reduction of oxygen to superoxide anion is -0.33V so the valence band of the catalyst should have a potential less negative than this. To fulfill this condition if we select a single photocatalyst, the band gap required to achieve this is too high and the required energy cannot be provided by the visible region radiation. To overcome this problem we select a combination of 2 catalyst such that one catalyst which can easily reduce and the other which can easily oxidize and the two have small band gap overlapping each other.

1.2.3 Z-Scheme

In this mechanism the photogenerated electron in the valence band of the semiconductor are transferred into the conduction band and annihilate the holes of the catalyst with lower valence band potential. Here again the charge separation take place and the electrons are transferred to the conduction band of that catalyst. Finally the holes are present in the catalyst whose valence band potential is relatively higher than the other and the electrons are present in the conduction band of the catalyst with more negative potential. Here the reactive radicals are generated and the dye molecule gets converted into simpler form.[9],[11]

An example of this catalyst is SnO_{2-x}/g-C_3N_4

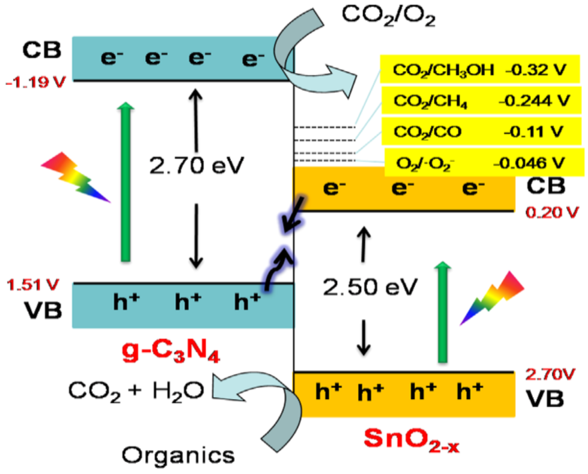


Figure 2 Z-scheme

1.3 Major Objective Proposed

The main objective is to prepare a photocatlyst which can efficiently remove the color of the dye and degrade it by using solar light.

Since the conventional methods are not so effective as they convert one type of pollution to the other and hence are responsible for secondary pollution. Photocatalysis being one of the latest advanced oxidation technique is very efficient in completely degrading the dye molecule to very simple compounds like Carbon dioxide and water. But the present catalysts which are being used works efficicnetly only in UV region. Since solar energy is available free of cost, if we can use that it would contribute toward the green engineering technique. But the energy of visible radiation is less and UV radiation being limited in the solar spectrum, we need to develop a catalyst which efficiently works in the visible region. Due to the band gap limitation of a single photocatayst, this is not practically possible. So to achieve this we need to prepare a composite photocatalyst which can work efficiently in the visible region too and can degrade the dye molecule.

1.4 Objective Achieved

We have achieved our objective. We have prepared composite catalyst which is working more efficiently than single photocatalyst in solar light for methylene blue's degradation.

1.5 Technical Difficulties Faced

Changing Intensity of sunlight, Filtering problem or sampling problem (dye was getting absorb in filter paper)

1.6 Experimental Setup and Results

The catalyst SiC/Ag_2CO_3 will work according to the Z-scheme mechanism.

3.1.1 Synthesis of Photoctalyst

In order to prepare this composite photocatalyst, the two catalyst selected were SiC and Ag_2CO_3. The VB and CB potential of SiC are 1.5V, -1.5V and that of Ag_2CO_3 are 2.68V, 0.68V respectively and hence can effectively work in visible light as a photocatalyst for color removal of dye.

In order to understand the effect of concentration of both SiC and Ag_2CO_3 the catalyst were prepared in the varying proportion like 100% Ag_2CO_3, 94% Ag_2CO_3-06% SiC, 91% Ag_2CO_3-09% SiC, 88% Ag_2CO_3-12%SiC, , 85% Ag_2CO_3-15%SiC, 82% Ag_2CO_3-18%SiC and TiO_2

Materials :

The materials used were Silver Nitrate($AgNO_3$), sodium Carbonate(Na_2CO_3), SiC and distilled water. SiC was directly used without any further purification.

Preparation of Ag_2CO_3:

The Ag_2CO_3 sample was synthesized by a simple ion-exchange method. In a typical procedure for synthesing 100% Ag_2CO_3, $AgNO_3$ solution(0.1N) was placed in a beaker and an aqueous solution of sodium carbonate(0.05N) was added dropwise using a burrete under constant stirring at room temperature. After this, a constant stirring for 24hrs was provided to obtain a yellowish green precipitate. The solution was filtered and washed several times with distilled water till showed neutral pH. The Ag_2CO_3 obtained was then dried in oven for 3-4hours.[14],[15]

Synthesis of Ag_2CO_3/SiC composite catalyst

For the synthesis of composite catalyst, the required amount of SiC powder was dissolved in water and ultrasonicated for 2hr at 175-180V. After this $AgNO_3$ was added to this solution and was added to this solution and was continuously stirred for 60min. Similar procedure of adding Na_2CO_3 to the solution was employed. After giving a constant stirring for about 24hr the solution was filtered and the precipitate obtained was washed with distilled water and then dried in microwave oven for about 15min.[14]

For the preparation 3gram of different combinations procedure remains same as above, only amount of raw material changes.

Table 1 Amount of raw material for different composite catalysts

Catalyst	$AgNO_3$	SiC	$NaHCO_3$
100% Ag_2CO_3	4.93gm	-	2.436gm
94% Ag_2CO_3-06% SiC	3.427gm	0.18gm	1.693gm
91% Ag_2CO_3-09% SiC	3.317gm	0.27gm	1.638gm
88% Ag_2CO_3-12%SiC	3.208gm	0.36gm	1.585gm
85% Ag_2CO_3-15%SiC	3.09gm	0.45gm	1.526gm

3.1.2 Experiment for color removal

The dye solution prepared for the experiment contained 25mg in 700 ml of distilled water which is equivalent to 25 ppm solution. We prepared such 6 samples of the dye solution and added 1.6 grams of the 94%Ag_2CO_3-06%SiC, 91%Ag_2CO_3-09%SiC, 88%Ag_2CO_3-12%SiC, 85% Ag_2CO_3-15%SiC,100% Ag_2CO_3 And TiO_2 respectively.

The 6 samples were first kept in dark with constant stirring for 2 hour. This was done to check the extent of adsorption. The two readings were taken in the interval of 60 min. The stirring was done at 750 rpm.

The samples were then kept in the sunlight. In order to prevent evaporation losses of the solution, the samples were kept in the chilling water bath maintained at 15°C. The stirring was done at 750 rpm. The initial intensity of the light at the time of placing the samples was recorded as 104 mW/cm^2. The first 4 readings were taken in the interval of 30 min and the intensity of light at that time was 100, 102, 110,114 mW/cm^2 respectively and the last reading was taken after 3 hours from the initial time of placing the samples and the intensity was recorded as 108 mW/cm^2. The samples were placed in the sunlight at 11 am and were withdrawn at 3 pm.

The UV analysis for all the readings was done and recorded.

Figure 3 Setup in Dark

Figure 4 Setup in Sunlight

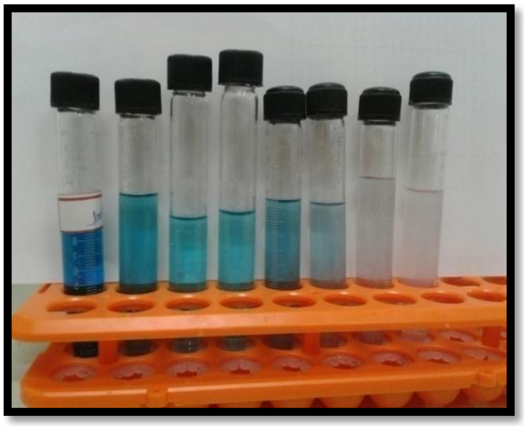


Figure 5 94% Ag_2CO_3-6% SiC

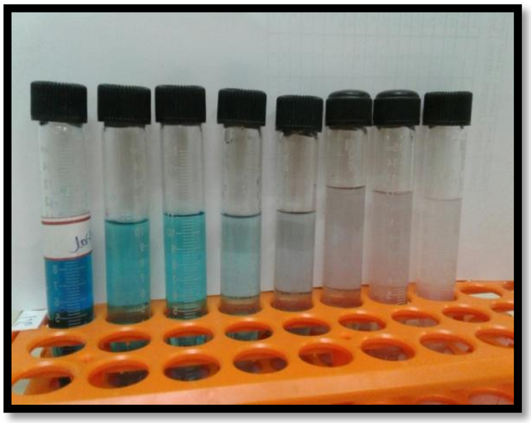


Figure 6 91% Ag_2CO_3-09% SiC

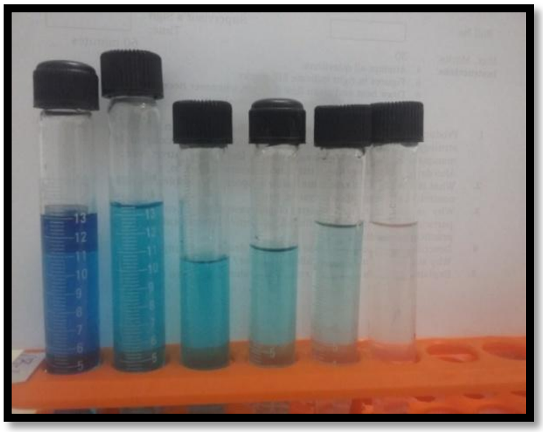


Figure 7 88% Ag_2CO_3-12% SiC

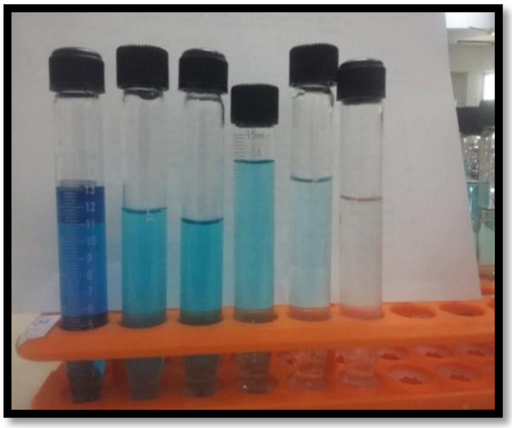


Figure 8 85% Ag_2CO_3-15% SiC

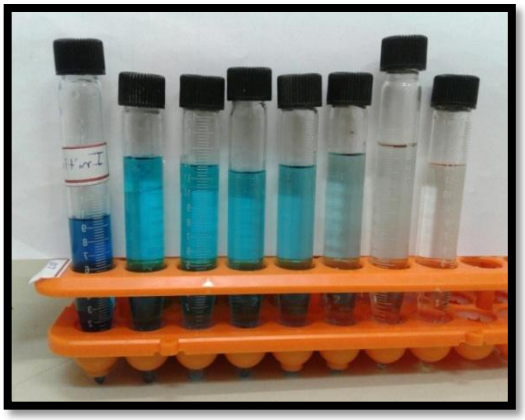


Figure 9 TiO_2

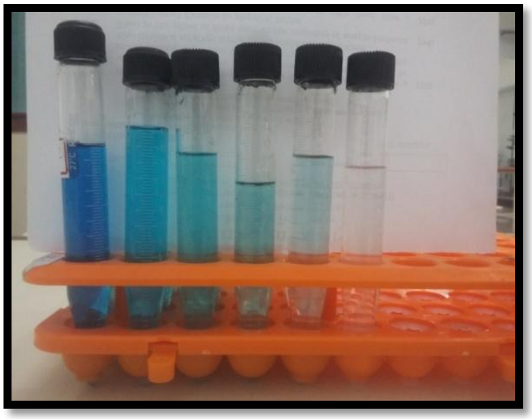
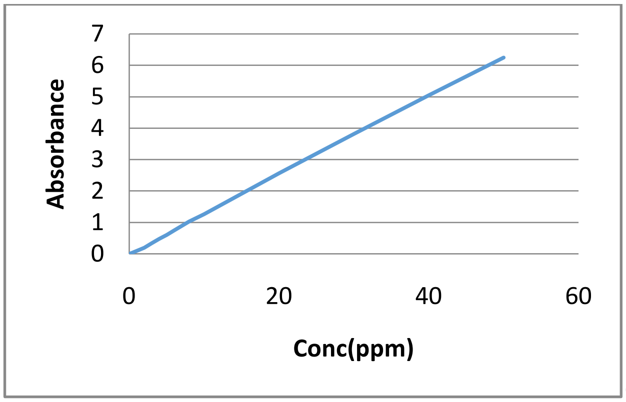


Figure 10 100% Ag_2CO_3

3.1.3 Results



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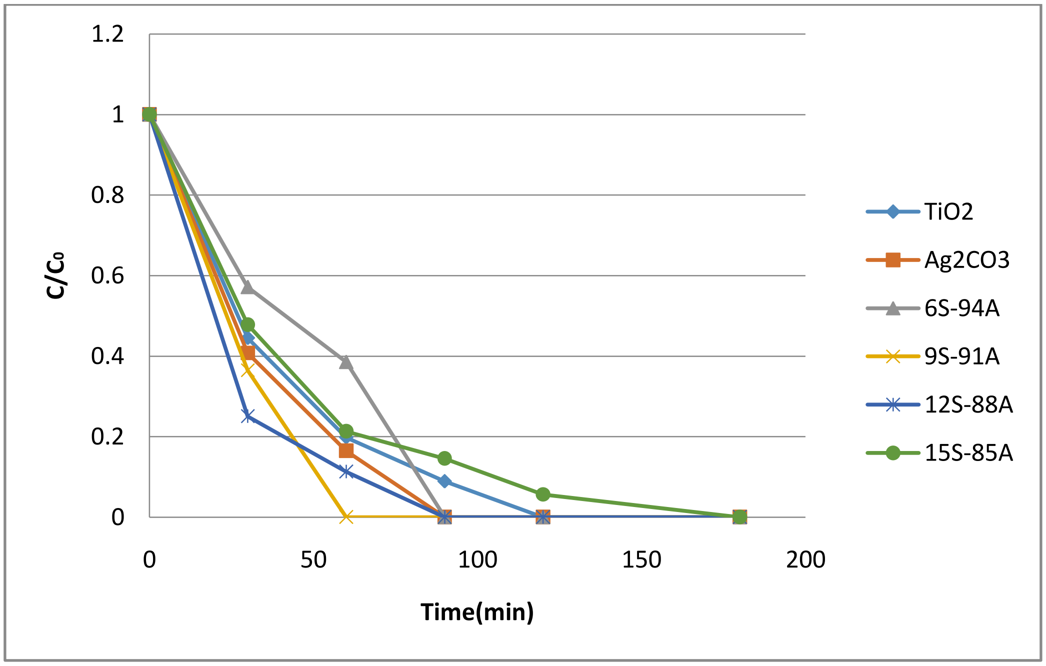

(12)

Figure 11 Calibration Curve, Figure 12 C/C_0 vs time(min)

Table 2 C/C0 with respect to time

time	TiO2	Ag2CO3	6S-94A	9S-91A	12S-88A	15S-85A
0	1	1	1	1	1	1
30	0.444824	0.407446	0.571429	0.364379	0.250291	0.477925
60	0.197952	0.164426	0.385445	0	0.112922	0.213024
90	0.088737	0	0	0	0	0.145695
120	0	0	0	0	0	0.056291
180	0	0	0	0	0	0

1.7 Results

From the above recorded observations, we conclude that amongst the composite catalysts prepared, the best results in terms of dye degradation were shown by 9% Sic-91%Ag2CO3. It is also seen that this composite catalyst works even better than 100% TiO2 and 100% Ag2CO3 and 100% SiC. This proves the formation of the z-scheme photocatalyst.

1.8 Budget Analysis

1. Budget Sanctioned : 20,000/-
2. Budget Utilized : 19,846/-

Sr. No	Name of Product	Amount	Bill No.	Date	Consumables/ NonConsumables	Dead Stock Num
1	Conical Flask 500mL	2*154= 308	PC/15-16/RI/439	09/03/16	Consumables	NA
2	Conical Flask 1000mL	495				
3	Measuring Cylinder 10mL	4*82= 328				
4	Beaker 1000mL	2*205= 410				
5	Beaker 250mL	2*85= 170				
6	Beaker 600mL	3*110=330				
7	Stirring Rod 6 * 200	2*11= 22				
8	Culture Tube Flat Bottom 15mL	41*22= 902				
9	Silver Nitrate AR 25Gm (35% Disc)	4,707				
10	Burrete10mL	2*240= 480				
11	Crystallizing Dish 1000mL	1,100				
12	Conical Flask 25mL	41*52= 2,132				
13	Plastic Laboratory Tray 450*350*75	430				
	Total	11,814				
	Discount(20%)(except 9th)	3068.8				
	After Disc.	8745.1				
	VAT (5%)	437.3				
	Final Amount	9182				
14	Pipette Auto Variable (500microL) (Disc. 16%)	3,950	R0642/15-16	23/02/16	Consumables	NA
15	Filter Paper-Sup,Qua-9.0cm (Disc. 13%)	4*1,600 =6,400				

16	Pipette Measuring-B.G.-10mL (Disc. 28%)	2*95= 190	R0642/15-16	23/02/16	Consumables	NA
17	Burrete for Teflon-B.G.-25mL (Disc. 28%)	3*225= 675				
	Total	11,115				
	Discount	1,706.2				
	After Disc.	9,508.8				
	VAT(5%)	475.44				
	Final Amount	9,984				
18	Gloves GAMAX	2*75= 150	AA11672	29/03/16	Consumables	NA
19	Gloves	2*40= 80				
20	Extension Cord 2MTR LTK-454	450	{HE}18615{15-16}	29/03/16	Consumables	NA
	Total	680				
	Grand Total	9182 + 9984 + 680 = **19,846**				

1.9 Conclusion and Future Work

Thus we conclude that the prepared z-scheme photocatalyst 9% Sic- 91% Ag2CO3 works efficiently than TiO2 and Ag2CO3 and is efficient catalyst for the methylene blue degradation. It works well in the solar region so rather than using a single photocatalyst, we can use such composite catalyst which works in the sunlight. Also the cost of treatment of waste water polluted due to dye can be decreased. In future, we will do the parametric study on this catalyst which will include pH, catalyst loading, dye concentration, rpm.

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