WEP Warwick Evans Publishing

International Journal of Materials Science and Technology Studies

ISSN: 3006-7693 (Print), ISSN: 3006-3744 (Online) | Volume 1, Number 2, Year 2024
DOI: https://doi.org/10.62051/ijmsts.v1n2.01
Journal homepage: https://wepub.org/index.php/IJMSTS/index

Preparation of Graphene Doped Titanium Dioxide Compo -site and Study on Treatment of Laboratory Wastewater

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ABSTRACT

In this paper, five kinds of composite materials were prepared by using graphene oxide (or graphene) doped with TiO2. The photocatalytic properties of five kinds of composite materials, pure graphene oxide and pure TiO2 for methyl orange, phenol solution and rhodamine B simulated laboratory wastewater were studied respectively. The experimental results show that the photocatalytic performance of graphene oxide-doped titanium dioxide composites is better than that of graphene oxide-doped titanium dioxide composites. When graphene oxide is doped with titanium dioxide as catalyst, the higher the purity of graphene oxide, the better the photocatalytic effect of the composite material. Doped titanium dioxide is better than undoped titanium dioxide. When the amount of doped titanium dioxide is 0.3g, the photocatalytic effect is the best. Based on the comprehensive data, it can be clearly found that graphene oxide doped TiO2 composite is more suitable for degrading rhodamine system. By simulating the treatment of laboratory wastewater, it lays a theoretical foundation for the treatment of laboratory wastewater in the future.

KEYWORDS

Titanium dioxide; Grapheme; Photocatalysis; Composite material

1. INTRODUCTION

Human beings have entered the industrial age, industrial production technology has become increasingly mature, the production range has become wider, and the application of organic dyes has become more and more extensive, resulting in water pollution problems, which have caused a great threat to human health and the ecological environment. As a typical azo dye, methyl orange is widely used. The high chromaticity of the wastewater makes it difficult for light to penetrate the aquatic plants, which affects the absorption of light by aquatic plants, thus weakening photosynthesis and affecting the self-purification function of the water body. Dye wastewater has high alkalinity and high salt content, which can cause soil salinization, resulting in low crop yield or inability to grow [1].The pollution of water by phenol will cause harm. When the concentration of phenol in water is low, the water will be smelly, and fish and shellfish will be affected, while when the concentration of phenol is high, a large number of fish will die due to poisoning. Irrigating farmland with high concentration of phenol wastewater will also kill crops [2]. Due to the characteristics of high toxicity and low biodegradability of phenol, the treatment of water polluted by phenol has become an urgent problem [3]. Rhodamine B is a synthetic dye, which is toxic and will do harm to human body. If it comes into contact with the skin, it may stimulate the skin, cause skin discomfort, red rash, skin itching and other skin allergies. If you eat it carelessly, you may have adverse reactions such as headache and vomiting, or long-term consumption may cause pulmonary edema, which may be life-threatening.

At present, photocatalytic technology has a good application prospect in the field of sewage purification, so it is particularly important to develop cheap, efficient, non-toxic and easy to recycle photocatalytic materials [4]. As a photocatalyst, TiO_2 has the advantages of low cost, non-toxicity, high efficiency and high thermal stability, and is widely used in the fields of pollutant decomposition and hydrogen production [5]. However, due to the wide band gap (~3.2eV) of pure TiO_2, it can only be excited by ultraviolet light, so the utilization efficiency of sunlight is poor and the quantum efficiency of photocatalytic reaction is low [6]. In the past few years, efforts have been made to modify the band gap of TiO_2 and improve its photocatalytic activity under visible light illumination. For example, doping refers to the introduction of foreign atoms or complexes into the TiO_2 matrix to change the electronic structure in order to enhance its visible light absorption and carrier transfer [7]. Graphene oxide (GO) is an important derivative of graphene. It is a two-dimensional sp^2 conjugated structure material with carbon atoms. Its unique structure endows it high specific surface area, excellent electrical conductivity and mechanical properties, and it is considered as one of the important candidate materials for constructing three-dimensional porous structures. It has been widely studied in the field of photothermal materials. The composite materials obtained by doping titanium dioxide with graphene oxide (graphene) not only make up for the defects of titanium dioxide, but also improve the photocatalytic performance and enhance the purification ability of pollutants.

In this paper, the photocatalytic performance of graphene oxide doped titanium dioxide composite prepared by hydrothermal method. They were studied for methyl orange, phenol and Rhodamine B systems, which provided theoretical basis and reference significance for laboratory waste liquid treatment.

2. EXPERIMENTAL PART

2.1. Reagents and Instrumentst

The main reagents in the experimental process: graphene oxide (powder, analytically pure, Inner Mongolia Qingmeng Graphene Technology Co., Ltd.), graphene (self-prep -ared), graphene oxide (lamellar, self-prepared), anhydrous ethanol, titanium dioxide (analytically pure, Sinopharm Chemical Reagent Co., Ltd.), methyl orange (analytically pure, Tokyo Chemical Industry Development Co., Ltd.), rhodamine B (analytically pure, Tokyo Chemical Industry Development Co., Ltd.).

Main instruments and models: UV-1901 UV-visible spectrophotometer, KQ-200 KDE high-power numerical control ultrasonic cleaner, UV lamp, SHZ-D(III) circulating water vacuum pump, Quartz colorimetric dish, filter, CS101-2E Electric Drumming Dry Box, analytical balance and reaction kettle.

2.2. Preparation of Standard Solution

Preparation of 100 $mg \cdot L^{-1}$ (methyl orange, phenol, rhodamine B) standard concentr -ation solution: Analyze the balance of balance of 0.1001g-0.1003g (methyl orange, phenol, Rodin Ming B), put it each in 250mL beaker, add a proper amount of distilled water to dissolve it, stir it with a glass rod, transfer it to a capacity bottle of 1000mL, then wash the beaker and glass rod with distilled water for three times, add distilled water to set to the scale line and shake it, pour it into a dry and clean reagent bottle, put on the label for later use.

2.3. Preparation of Composite Materials

2.3.1. Preparation of Graphene-TiO_2 (A) and Graphene oxide (lamellar)-TiO_2(B)

Graphene -TiO_2(A) and Graphene oxide (lamellar)-TiO_2(B) composites were prepared by hydrothermal method. Weigh 0.03g of dried graphene(lamellar graphene oxide) and put it into a beaker, add 0.3g of TiO_2, add 20mL of deionized water, stir it ultrasonically for 2 hours, put it into a reaction kettle, set the temperature at 110°C, react for 5 hours, filter it after cooling, put the product into a crucible, dry it for 2 hours, weigh it after cooling, put it into a transistor, and label it and use it as A(B).

2.3.2. Preparation of Graphene Oxide (Powder) -TiO_2(C, D, E)

Graphene oxide -TiO_2 composites (C, D, E) were prepared by hydrothermal method. Add 0.03g of dried graphene oxide into a beaker respectively, then add 0.3g, 0.4g and 0.5g of TiO_2 respectively, add 20mL of deionized water respectively, stir with ultrasound for 2h, put it in a reaction kettle, set the temperature at 110°C, react for 5h, pump and filter it after cooling, put the product into the crucible, dry it for 2h, cool it and weigh it after cooling. put it into a transistor, label each preparation and reserve, respectively C, D, E. In order to provide sufficient composite materials for the experimental process, the quality of graphene oxide and TiO_2 can be expanded at the same time according to the ratio.

2.4. Study on Photocatalytic Properties

2.4.1. Research of the Maximum Wavelength

Wavelength scanning was selected, distilled water was used as a reference, different concentrations methyl orange solution were put into quartz colorimetric dishes and placed in the ultraviolet spectrophotometer. The wavelength range was set 365nm-580nm, and the maximum absorption peak was found. The maximum wavelength marked was 465nm, and the wavelength of this experiment was selected at 465nm.

Using the same method, phenol solutions of different concentrations were taken, and the wavelength range was 200nm-400nm, and the maximum absorption peak was sought, with the maximum wavelength at 269nm, which is chosen in this experiment.

Rhodamine B solutions with different concentrations were selected, and the wavelength range was 300nm-600nm to find the maximum absorption peak. The maximum wavelength was at 554nm, and the wavelength in this experiment was selected at 554nm.

2.4.2. Drawing curve of methyl orange standard solution

Accurately measure 1mL, 2mL, 3mL, 4mL, 5mL to 50mL volumetric bottles of methyl orange standard solution with a 10.00mL pipette, and use distilled water for constant volume for standby, with the concentrations of 2.0000 $mg{\cdot}L^{-1}$, 4.0000 $mg{\cdot}L^{-1}$, 6.0000 $mg{\cdot}L^{-1}$, 8.0000 $mg{\cdot}L^{-1}$ and 10.0000$mg{\cdot}L^{-1}$ respectively. The diluted methyl orange solution was placed in quartz colorimetric dishes from thin to concentrated and placed in an ultraviolet spectrophotometer. Distilled water was used as a reference and the wavelength was set at 465nm for quantitative determination. The relationship between absorbance and concentration was selected, and the measured data were listed in Table 1 and drew a standard curve, as shown in Figure 1.

Table 1. Measured data of standard curve of methyl orange solution

number	**1**	**2**	**3**	**4**	**5**
Absorbance /(A)	0.221	0.380	0.554	0.721	0.881
c/($mg{\cdot}L^{-1}$)	2.0000	4.0000	6.0000	8.0000	10.0000

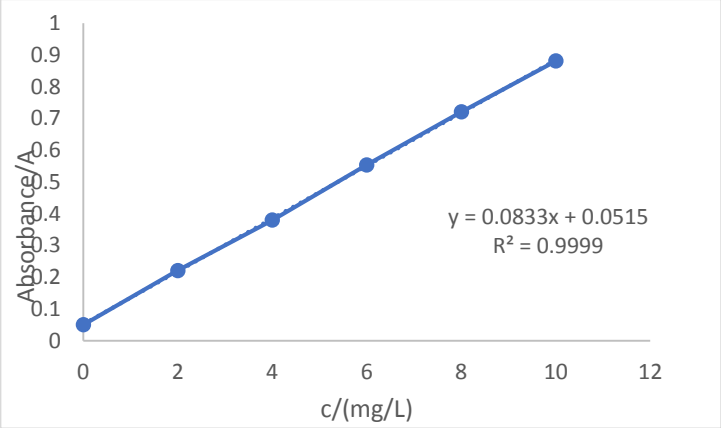

Figure 1. Curve of methyl orange standard solution

2.4.3. Study on Photocatalytic Properties of Methyl Orange Solution

The experiment of measuring photocatalytic Properties refers to the literature design of Zhang Qiaoling, Jiao Yurong and Pan Yanfei [8, 9, 10]. The specific process is as foll -ows:

Exploration of experimental conditions: Dilute the methyl orange solution with a con -centration of 100mg·L^{-1} to 5mg·L^{-1} and 10mg·L^{-1}, remove it to 100mL beaker, and add 100mg Graphene Oxide (Powder) -TiO_2(C). Ultraviolet lamp was used as the light sour -ce for irradiation, the first four sampling intervals were 15min, and the last three groups were sampled at intervals of 30min. Each sampling was measured with filter. The measured data are listed in Table 2, According to the data, the methyl orange solutions with different concentrations were compared with time under photocat -alysis, as shown in Figure 2.

Table 2. Comparison of methyl orange solutions with different concentrations under photocatalysis over time

Time	0min	15min	30min	45min	60min	90min	120min	150min
5mg·L^{-1}	5.0	0.3	0.0	0.0	0.0	0.0	0.0	0.0
10mg·L^{-1}	10.0	3.8	2.5	2.4	2.4	2.3	2.4	2.2

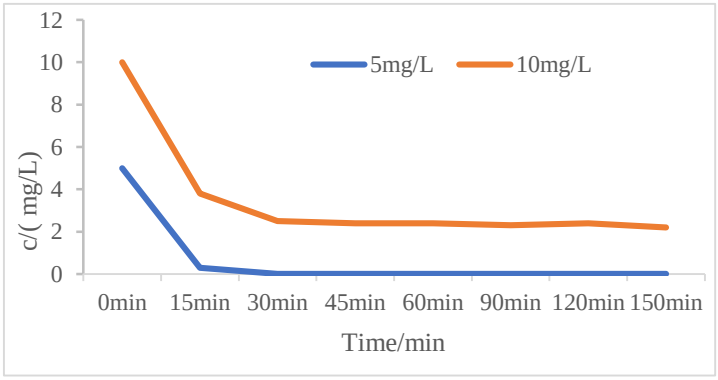

Figure 2. Comparison of different concentrations of methyl orange with time under the catalysis of the same light source

According to Figure 2, except for the different concentration of methyl orange, under the same experimental conditions, the degradation rate of methyl orange in 5mg·L^{-1} was 94% in 15min, and most of the methyl orange had been degraded, and the concentration of methyl orange was zero in 30min-150min. The degradation rate of 10mg·L^{-1} reached 62% in the first 15 minutes, and rose to 75% in 30 minutes, but it did not change much in 45 minutes, basically unchanged in 60 minutes, and changed little (2.5-2.2mg·L^{-1}) in 30 minutes-150 minutes, and only reached 78% in the last 150

minutes. Therefore, the best experimental conditions are that the concentration of methyl orange is 10mg·L^{-1} and the total time is controlled at about 30min.

Verification of experimental conditions: the standard methyl orange solution with a concentration of 10mg·L^{-1} was transferred to a 100mL beaker and 100mg of C composite was added. The ultraviolet lamp was used as the light source for irradiation, the control lamp distance was 10cm, and the time interval was kept to be 5min. Each group was measured 7 times, and filter membrane sampling was used for measurement each time. The measured data were listed in Table 3 for drawing, as shown in Figure 3.

Table 3. The changes of concentrations of methyl orange standard solution with time

Time	0min	5min	10min	15min	20min	25min	30min
5mg·L^{-1}	5.0	1.1	0.5	0.3	0.1	0.0	0.0
10mg·L^{-1}	10.0	4.3	4.2	3.8	2.6	2.5	2.4

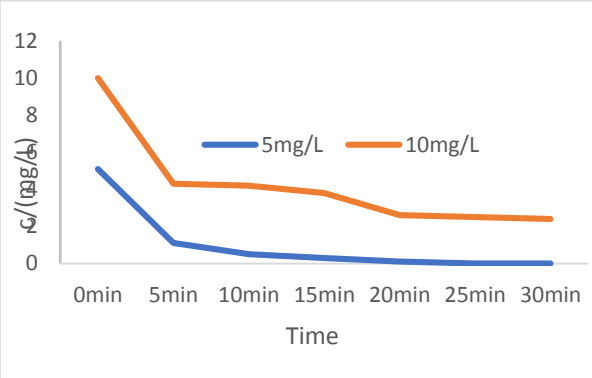

Figure 3. Changes of methyl orange solutions with different concentrations within 30min

It can be intuitively seen from Figure 3 that the degradation rate of methyl orange solution concentration is obvious, and the best time interval for determination of degradation rate is 5 minutes/time. However, the concentration of 10mg·L^{-1} methyl orange solution at 10min is similar to that at 5min, and the downward trend is not obvious. There are two possible reasons. First, before the solution is transferred into the quartz colorime -tric dishes, it was not cleaned with the liquid to be tested, and some of the upper liquid remained, which led to an increase in concentration. Second, the solution is not filtered clean enough when it is extracted. The suspension of composite material in the solution causes great interference to the test.

The effects of different photocatalysts on the photocatalytic performance of 10mg·L^{-1} methyl orange solution were determined: keeping the above conditions the same, different photocatalysts (A, B, C, D, E, pure TiO_2 and pure GO) were added to the methyl orange solution. Samples were taken at a time interval of 5 minutes, and each group was measured 6 times. All samples were filtered with filter membrane, and then the photocatalytic performance was tested. The measured data were listed in Table 4 and plotted, as shown in Figure 4.

Table 4. Experimental data of photocatalytic performance of different photocatalysts for methyl orange solution

Catalysts c time	0min	5min	10min	15min	20min	25min	30min
A	10.0	6.1	6.4	6.2	5.9	5.6	5.5
B	10.0	6.8	6.4	6.2	5.8	5.5	5.2
C	10.0	4.3	4.2	3.8	2.6	2.5	2.4
D	10.1	5.3	4.4	4.1	3.6	3.3	3.1
E	10.1	6.7	6.0	5.6	5.3	5.1	5.0
TiO_2	10.1	8.7	8.4	7.2	7.0	6.9	7.0
GO	10.0	3.7	2.1	1.3	0.6	0.1	0.0

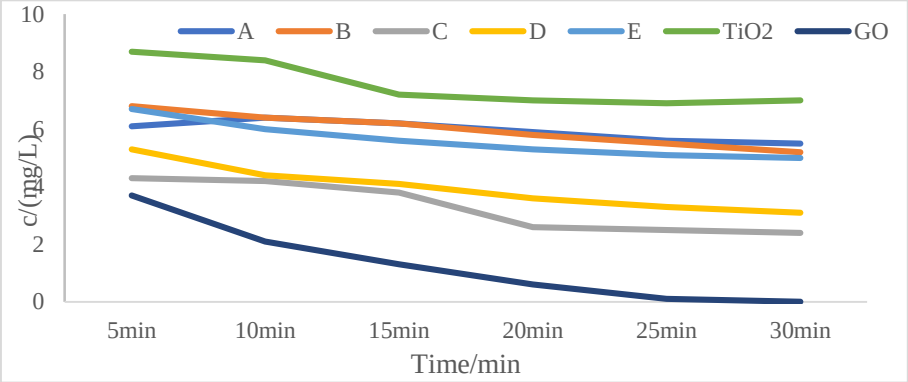

Figure 4. Photocatalytic performance of different photocatalysts for methyl orange solution

It can be seen from Figure 4 that the purity and aggregation state of group B and group C are different, and the doping amount of titanium dioxide in Group C, Group D and Group E is different. In addition, whether pure titanium dioxide and pure graphene oxide are doped or not, among the factors that affect the degradation effect of methyl orange, the experimental results show that pure graphene oxide has the best effect, which may be that it does not contain any impurities, has a large specific surface area and strong adsorption capacity, and directly adsorbs methyl orange solution molecules, and the solution becomes clear after degradation. Secondly, under the condition of different doping amount of titanium dioxide, 0.03g graphene oxide doped with 0.3g titanium dioxide has the best effect, and with the increase of titanium dioxide content, the performance effect gradually decreases. While the doping amount is the same, the analysis of pure graphene oxide has the best effect, compared to the self-made sheet effect is worse, there may be impurities or agglomeration caused by reduced adsorption. And homemade graphene and graphene oxide compared to graphene oxide, graphene oxide effect is better.

2.4.4. Drawing curve of phenol standard solution

The standard phenol solution was accurately measured in volume bottles of 10mL, 20mL, 30mL, 40mL to 50mL with a 10.00mL pipepipe, and use distilled water for constant volume for standby, with the concentrations of 20.0000 $mg{\cdot}L^{-1}$, 40.0000 $mg{\cdot}L^{-1}$, 60.0000 $mg{\cdot}L^{-1}$ and 80.0000 $mg{\cdot}L^{-1}$ respectively, and take another 30mL of stock solution into 100mL of dry and clean beaker, the concentration is 100$mg{\cdot}L^{-1}$. The diluted phenol solution was placed in a quartz colorimetric dish from thin to concentrated and placed in an ultraviolet spectrophotometer. Distilled water was used as a reference, and the wavelength was set at 269nm for quantitative determination. The relationship between absorbance and concentration was selected, and the measured data were listed in Table 5, and draw a standard curve, as shown in Figure 5.

Table 5. Measured data of standard curve of phenol solution

number	1	2	3	4	5
Absorbance /(A)	0.398	0.710	1.031	1.346	1.670
Concentration /(mg·L^{-1})	20.0000	40.0000	60.0000	80.0000	100.0000

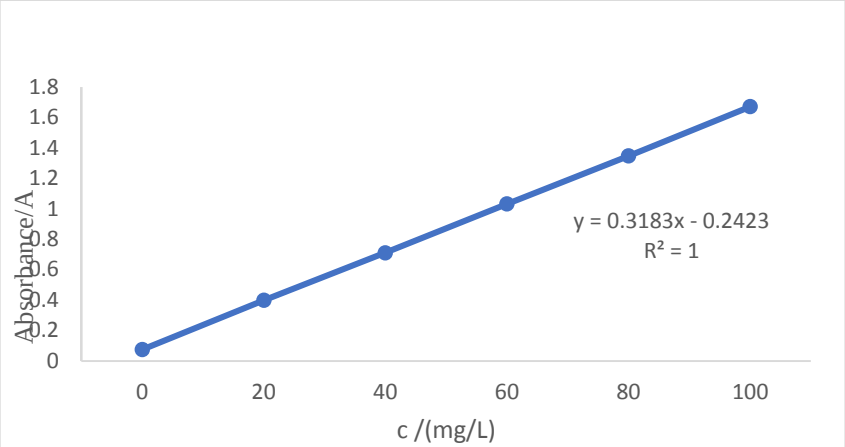

Figure 5. Curve of phenol standard solution

2.4.5. Effects of Different Photocatalysts on Photocatalytic Performance of Phen -ol Solution

The influence of different photocatalysts on the photocatalytic performance of phenol solution was determined: The standard solution of phenol with a concentration of 100mg·L^{-1} was diluted to 40mg·L^{-1}, and then moved to a 100mL beaker, keeping the above conditions the same. and add different catalysts respectively(A,B, C, D, E, pure TiO_2 and pure GO).The measured data of different catalysts were listed in Table 6 and plotted, as shown in Figure 6.

Table 6. Measured data of photocatalytic performance of different photocatalysts for phenol solution

Catalyst c Concentration time	0min	5min	10min	15min	20min	25min	30min
A	40.3	37.0	33.5	31.9	29.6	27.8	26.5
B	40.3	35.4	30.9	28.5	26.4	25.1	24.9
C	40.7	32.4	30.8	28.0	27.3	24.1	23.9
D	40.7	34.2	31.4	29.7	28.3	26.2	25.6
E	40.1	35.6	34.2	32.0	28.8	27.4	27.4
TiO_2	40.1	38.6	37.5	36.0	35.3	34.7	34.4
GO	40.0	37.5	37.0	36.3	36.1	35.6	34.9

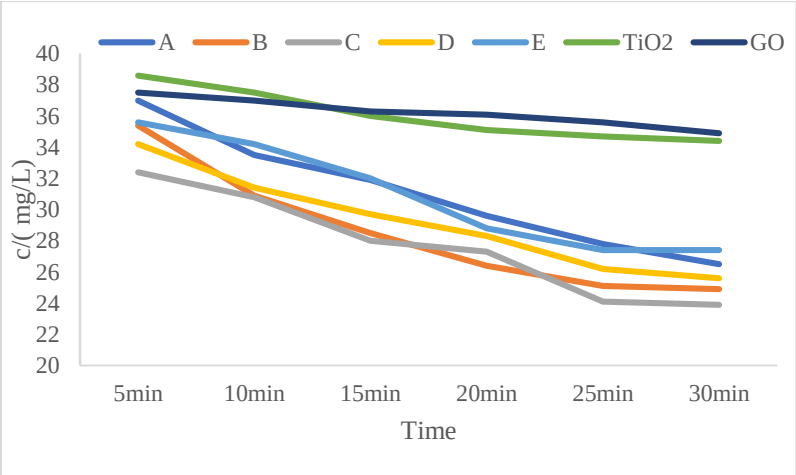

Figure 6. Photo-catalytic performance of different photocatalysts for phenol solution

From Figure 6, it can be found that the degradation effect of composites A, B, C, D and E is obviously higher than that of pure TiO_2 and pure GO, and the general trend of photocatalytic degradation effect from high to low is C>B>D>E>A>TiO_2>GO, in which graphene oxide titanium doped with 0.3g titanium dioxide B and C is the best for phenol degradation. The photocatalytic degradation effect of graphene oxide titanium doped with different titanium dioxide content: the degradation rate of C>D>E, pure TiO_2 and pure GO is poor, which shows that the composite material is very meaningful in catalysis.

2.4.6. Drawing curve of rhodamine B standard solution

Accurately measure 5mL, 10mL, 15mL, 20mL, 25mL to 50mL volumetric bottles of rhodamine B standard solution with a 10.000 mL pipette, and use distilled water for constant volume for standby, The concentrations were 10.0000 mg·L^{-1}, 20.0000 mg·L^{-1}, 30.0000 mg·L^{-1}, 40.0000 mg·L^{-1} and 50.0000mg·L^{-1} respectively. The diluted rhodamine B solution was placed in a quartz colorimetric dish from thin to concentrated and placed in an ultraviolet spectrophotometer. Distilled water was used as a reference, and the wavelength was set at 554nm for quantitative determination. The relationship between absorbance and concentration was selected, and the standard curve was drawn, as shown in Figure 7.

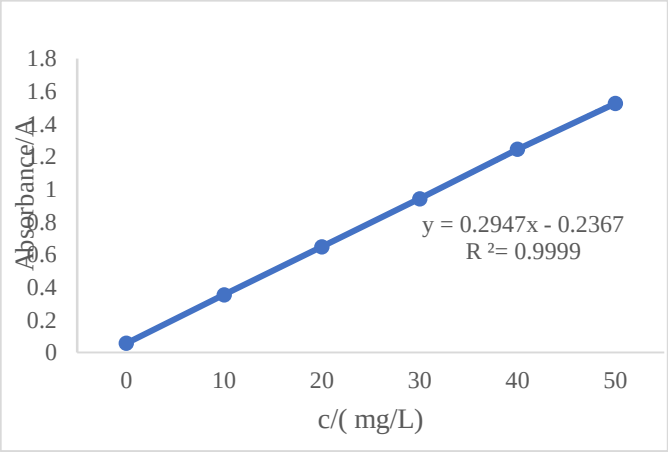

Figure 7. Curve of rhodamine B standard solution

2.4.7. Effects of Different Photocatalysts on Photocatalytic Performance of Rhodamine B Solution

Dilute the rhodamine B standard solution with a concentration of 100mg·L^{-1} to 20mg·L^{-1}, then transfer it to a 100mL beaker, and keep the above conditions the same. The different catalysts (A, B, C, D,

E, pure TiO_2 and pure GO (powder)) were added Rhodamine B. The measured data were listed in Table 7 and plotted, as shown in Figure 8.

Table 7. Data of photocatalytic performance of different photocatalysts for Rhodam -ine B solution

Catalyst c concentration degree time	0min	5min	10min	15min	20min	25min	30min
A	20.0	16.9	16.5	16.1	14.8	14.2	13.5
B	20.0	15.1	14.6	12.6	12.3	11.8	11.2
C	20.1	9.8	7.3	4.4	2.1	0.9	0.3
D	20.0	11.5	10.3	8.7	6.6	4.8	3.1
E	20.1	13.7	13.4	12.7	11.6	10.9	10.6
TiO_2	20.4	19.8	19.4	18.9	18.4	18.3	18.0
GO	20.4	18.4	17.1	16.6	16.3	16.1	15.9

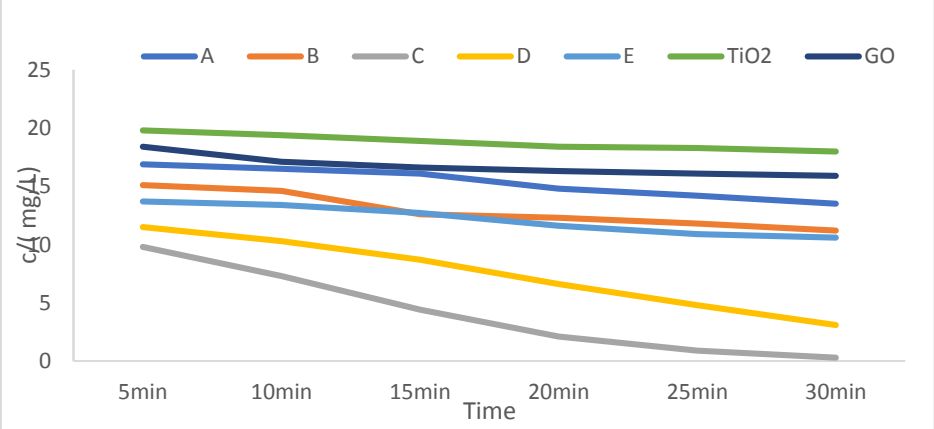

Figure 8. Photo-catalytic performance of different photocatalysts for rhodamine B solution

According to Figure 8, it can be found that the best effect is still C, except for the two groups of pure analysis. In contrast, the degradation rate of pure graphene oxide dop -ed titanium dioxide is more stable and the line is smoother, and the photocatalytic degradation effect of titanium oxide doped with different amounts of titanium dioxide is C>D>E, while the degradation rate of the two groups of self-made substances is reduced, but the degradation rate of B>A>GO>TiO_2.

3. CONCLUSIONS AND DISCUSSION

3.1. Comparison of Experimental Results Under the Same System

3.1.1. Comparison in methyl orange system

From Figure 4, the order of photocatalytic degradation obtained:GO>C>B>D> E>A>TiO_2, pure graphene oxide (GO) has the best photocatalytic performance, followed by C, that is, 0.03g graphene oxide doped with 0.3g titanium dioxide has the best photocatalytic performance, and with the increase of the amount of titanium dioxide, the photocatalytic performance becomes worse. Compared with the composites made of different purity graphene oxide doped with titanium dioxide, the pure graphene oxide doped with titanium dioxide has better effect. Compared with graphene oxide doped titanium dioxide composite, graphene oxide doped titanium dioxide composite has better effect.

3.1.2. Comparison in phenol system

According to the general trend of photocatalytic degradation effect obtained from high to low in Figure 6: C>B>D>E> A>TiO_2>GO, it can be found that the degradation effect of A, B, C, D and E composite materials is significantly higher than that of pure TiO_2 and pure GO, among which graphene oxide titanium doped with 0.3g titanium dioxide B and C is the best for phenol degradation. Poor degradation rates of pure TiO_2 and pure GO show that the composite material is of great significance in catalysis.

3.1.3. Comparison under Rhodamine B system

The degradation effect of photocatalyst degradation through Figure 8: C>B>A> GO >TiO_2, and the best effect is still Group C. Compared with the self-made graphene oxide doped titanium dioxide composite, the degradation rate of pure graphene oxide doped titanium dioxide composite is more stable and the lines are more gentle. The photocatalytic degradation effect of graphene oxide doped titanium dioxide with different amounts is CDE. The degradation rate of pure TiO_2 and pure GO is poor, while the homemade graphene is still better than graphene oxide.

To sum up, among the three systems studied, group C (0.03g graphene oxide doped 0.3g titanium dioxide composite) has the best photocatalytic effect, while pure TiO_2 has almost the worst photocatalytic effect. The photocatalytic effect of five kinds of doped titanium dioxide composite materials is better than that of pure titanium dioxide. Therefore, it is of practical significance to study the degradation of organic pollutants in water by graphene oxide doped titanium dioxide composite materials.

3.2. The Experimental Results of the Same Photocatalyst in Different Syste -Ms Were Compared

The photocatalytic effect of groups B and C (0.03g graphene oxide doped 0.3g titanium dioxide composite) was calculated according to the degradation rate:

The degradation effect of group B catalyst in different systems: the degradation rate of methyl orange solution is about 48%, the degradation rate of phenol solution is about 32%, and the degradation rate of Rhodamine B solution is about 50%.

The degradation effect of group C catalyst in different systems: the degradation rate of methyl orange solution is about 76%, the degradation rate of phenol solution is about 41%, and the degradation rate of Rhodamine B solution is about 98%.

According to the comparison of the photocatalytic effects of two kinds of composite photocatalysts on different systems, the degradation rate of the two kinds of composite photocatalysts was calculated, and it was analyzed that the degradation effect of the same catalyst on Rhodamine B solution was better, followed by methyl orange solution, and the degradation effect on phenol solution was the worst.

3.3. Discussion

In Figure 3, the data is abnormally large at the 30th minute of irradiation under ultraviolet lamp, which may be due to the increase of sampling times with the increase of time, and the difficulty of filtering the catalyst when the solution was removed increased with the decrease of volume. Particulate matter interferes with the test, resulting in some data anomalies. In addition, the data was artificially abnormal. Before moving the solution into the containment, the cleaning of the liquid on the upper group is partially residial, resulting in an occasional large concentration.

In Figure 4, the data of Group A fluctuated slightly at 10min, which may contain impurities, resulting in abnormal data. In addition, in the study of photocatalytic performance of methyl orange system, the photocatalytic effect of pure graphene oxide was better than that of TiO_2-doped composite

material. It may be that the amount of pure graphene oxide (0.1g) is large, which makes the orange solution become shallow immediately, fully indicating that its adsorption of methyl orange is very strong, but its price is expensive. It can also achieve good results when 0.03g of graphene oxide is used in composites. From the point of view of economic benefits, the choice of graphene oxide doped titanium dioxide is the best.

In Figure 6, the data of group C and group E are unstable and the curves are not smooth. The possible reason is that part of the graphene oxide was not evenly compounded during the composite reaction, resulting in fluctuations in the data and uneven lines.

In Figure 8, it can be clearly seen that the data of group A and group B fluctuated at 15min, which may contain impurities.

ACKNOWLEDGEMENTS

Foundation project: Research on preparation of graphene-doped titanium dioxide composites and treatment of laboratory Inner Mongolia (S202211427011) ,Jining Normal University 2022 Teaching Reform and Research Project (JGKT2022004).

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