

Preparation and Performance Study of Photocatalytic Composite Material Biochar/TiO₂ Based on Experimental Method

Chunxia Ren^{1*}

¹ College of Chemistry and Chemical Engineering, Taishan University, Tai'an, China

Email Address

13583831365@163.com (Chunxia Ren)

*Correspondence: 13583831365@163.com

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Abstract:

This article investigates the preparation and properties of composite material biochar/TiO₂ through experimental methods. Including the experimental design, analysis of experimental results, and experimental conclusions. In the specific experiment, pineal shells were used as raw materials to prepare porous pine shell biochar materials through HCl modification, KOH activation, and high-temperature pyrolysis; composite treatment of biochar material and nano TiO₂ particles to complete the preparation of composite material biochar/TiO₂. The morphology, structure, and performance characterization of the composite material were tested using scanning electron microscopy, specific surface area detection, and Raman spectroscopy; The photodegradation of Rhodamine B aqueous solution was tested using a UV visible spectrophotometer. According to experimental testing, the composite material can efficiently degrade Rhodamine B solution within 120 minutes, with a photodegradation rate exceeding 90%. It has a good treatment effect on organic dye wastewater of this type.

Keywords:

Photocatalysis, Composite Materials, Biochar, Nano Tio₂, Organic Dye Wastewater Treatment

1. Introduction

Rhodamine B belongs to trimethylene derivatives and is a major dyeing material in the manufacturing of silk fabrics and biological products. Therefore, such materials have great toxicity, and if their wastewater is directly discharged into water, it will have serious adverse effects on water bodies and organisms. However, due to the difficulty of treating such wastewater, the high cost of traditional treatment techniques, and the low qualification rate of treated wastewater, the preparation of green and low-cost treatment materials is a key focus in the field of wastewater treatment. Through existing practical research, it is known that using HCl as a modifier, KOH as an activator, and pineal shell as a raw material, pineal shell biochar can be prepared by

high-temperature pyrolysis. By effectively combining biochar with nano TiO_2 particles, composite materials can be prepared to carry out photodegradation treatment of toxic organic compounds in such wastewater, achieving good wastewater treatment results in a simpler and more economical way. To achieve this goal, the researchers decided to study the preparation of composite material biochar/ TiO_2 and its application performance in rhodamine B wastewater treatment through experiments.

2. Experimental Design

2.1. Basic materials

In this experiment, the selected basic materials include the following. (1) The common pineal shell in the current market. (2) Anhydrous ethanol, KOH, and Rhodamine B produced by Tianjin Damao Chemical Reagent Factory. (3) N-methylpyrrolidone produced by Tianjin Zhiyuan Chemical Reagent Co., Ltd. (4) P25 nanoscale TiO_2 particles produced by Tianjin Tianli Chemical Reagent Co., Ltd. (5) NaOH produced by Tianjin Kaitong Chemical Reagent Co., Ltd.

2.2. Main equipment

In this experiment, the main instruments and equipment selected include the following. (1) The 82-2 constant temperature magnetic stirrer produced by Shandong Juancheng Hualu Electric Heating Instrument Co., Ltd. (2) DZG-6050D vacuum drying oven produced by Shanghai Senxin Experimental Instrument Co., Ltd. (3) The 2066 high-speed centrifuge produced by Guangzhou Jidi Instrument Co., Ltd. (4) The Geimini 300 scanning electron microscope produced by ZEISS in Germany. (5) The ASAP 2020 PLUS specific surface area and porosity analyzer produced by Micromeritics in the United States. (6) LabRAM HR Evolution Raman Spectrometer produced by Horiba Corporation in Japan. (7) The UV-2300 ultraviolet-visible spectrophotometer produced by Shanghai Tianmei Scientific Instrument Co., Ltd.

2.3. Preparation of experimental materials

In this experiment, the main materials prepared include biochar and composite material biochar/ TiO_2 .

The basic preparation method and steps for porous pine shell biochar materials are as follows. (1) Cut the cleaned pineal gland into small pieces and control the cutting area reasonably. (2) Soak small pieces of pineal gland in KOH solution, replace KOH at regular intervals, and control the total soaking time. (3) Take out the small pieces of pineal gland shell, adjust the pH value with HCl solution to match the test standard, and then perform drying treatment under certain temperature conditions. (4) Place the dried small pieces of pineal shells into a quartz boat and perform nitrogen anaerobic sintering treatment in a tube furnace under high temperature conditions. (5) At a faster temperature rise rate, further increase the temperature of the tube furnace to reach the set final sintering temperature, and under this condition, carry out the final sintering treatment on the pinecone shell and control the sintering time. In this way, the rational preparation of pineal shell porous carbon (referred to as biochar) material samples can be achieved. Table 1 shows the basic parameter control during the preparation of porous pine shell biochar material in this experiment:

Table 1. Basic parameter control during preparation of porous pine shell biochar material in this experiment.

Serial number	Project	Parameter	Serial number	Project	Parameter
1	Small area of pineal gland shell	1cm ²	7	Nitrogen anaerobic sintering temperature	400°C
2	KOH solution replacement frequency	12h/time	8	Nitrogen anaerobic sintering time	1h
3	KOH soaking time	24h	9	Temperature rise rate of tubular furnace	10°C/min
4	Pineal shell pH standard	7	10	Maximum temperature rise of tubular furnace	750°C
5	Pinecone shell drying temperature	80°C	11	Final sintering time of pine nut shells	2h
6	Pinecone shell drying time	12h			

The basic preparation method and steps for composite material biochar/TiO₂ are as follows: (1) Add 2g of nano TiO₂ to a 200mL NaOH solution and perform 10 minutes of ultrasonic treatment to prepare nano TiO₂ dispersion. (2) Disperse 20mL of nano TiO₂ dispersion and 100mg of biochar in 100mL of water for 15 minutes of ultrasonic treatment, followed by 5 hours of magnetic stirring. (3) The obtained solution is subjected to centrifugal treatment, filtration treatment, cleaning treatment, and drying treatment in sequence, and then placed in a vacuum drying oven for continuous drying at 70 °C for 12 hours. (4) Take out the obtained sample, place it in a tube furnace, and conduct a 2-hour pyrolysis treatment with N₂ at 400 °C. In this way, the rational preparation of composite material biochar/TiO₂ can be achieved.

2.4. Test method

The main focus of this experiment is to investigate the catalytic effect of composite material biochar/TiO₂ in the photodegradation treatment of organic pollutants in Rhodamine B dye wastewater, in order to determine whether composite material biochar/TiO₂ can be used as a catalyst in its photodegradation treatment. Based on the experimental objective, the researchers mainly set the following experimental methods and steps during the specific experiment. (1) Prepare 100mL of Rhodamine B aqueous solution with a concentration controlled at 20mg/L. (2) Add 100mg of composite material biochar/TiO₂ to the solution as a catalyst and perform magnetic stirring for 30 minutes in a dark environment. (3) Use a 300W xenon lamp to irradiate the stirred solution, take a sample every 15 minutes, and test its absorbance using a UV visible spectrophotometer. (4) Based on the measured changes in the absorbance of the solution, evaluate the photocatalytic removal effect of the composite material biochar/TiO₂ on Rhodamine B to determine its photocatalytic activity and whether it is suitable for use as a photocatalyst in this type of water pollution treatment [1].

3. Analysis of Test Results and Discussion

3.1. SEM analysis of composite material biochar/TiO₂

SEM stands for Scanning Electron Microscopy (SEM) observation method. In this experiment, the SEM analysis of composite material biochar/TiO₂ was mainly carried out using the Geimini 300 scanning electron microscope. The basic method is to use

the scanning electron microscope to observe the morphology and structure of the composite material biochar/TiO₂ obtained in this preparation. After KOH modification and HCl activation treatment, further pyrolysis treatment is carried out on pine fruit shells, and rich pores with pore sizes ranging from 100-200nm will be uniformly generated on their surfaces. This rich and uniform porous biochar structure can provide effective sites for photocatalyst loading in the photocatalytic degradation of Rhodamine B polluted water.

Through this experiment, it was found that with the help of scanning electron microscopy, the porosity of the composite material biochar/TiO₂ can be clearly observed, indicating that it has a high pore density, and each pore presents a uniform distribution state. All nano TiO₂ particles composite in it adhere uniformly on the surface and inside the pores of the porous biochar material, and all particles exhibit irregular shapes. Further analysis reveals that the fundamental reason for the existence of this structural feature is that when photoelectrons reach the surface of the composite material biochar/TiO₂, TiO₂ can first transfer a portion of photon energy and release charge carriers. During this process, due to the presence of porous biochar in the composite material of biochar/TiO₂, it will hinder the interaction between nano TiO₂ particles through uniform and discrete distribution, preventing them from aggregating together [2].

3.2. Isothermal curve analysis of porous biochar in N₂ adsorption desorption process

According to existing practical research, it is known that in the photocatalytic degradation process of Rhodamine B, if porous biochar materials have a larger specific surface area, there will be more active sites with adsorption for photocatalytic degradation reactions, which is also more conducive to loading photocatalysts.

In this experiment, to investigate the specific surface area of porous biochar prepared from pine nut shells, the researchers decided to use N₂ physical adsorption desorption method at 196 °C to determine its specific surface area and pore structure. And with the support of the BET (specific surface area) calculation equation, the isothermal curve is obtained to determine the adsorption amount in the monolayer, thereby achieving further acquisition of important information such as its specific surface area.

Through experiments, it was found that under different relative pressure conditions, the adsorption desorption parameters of porous biochar for N₂ will form isothermal curves. After calculation, it was determined that the specific surface area of the porous biochar prepared based on pineal shell was 158.35m²/g, and the overall pore structure was good. It can effectively load the photocatalyst onto the surface, providing more active sites for the photocatalytic degradation of Rhodamine B and creating favorable conditions for the preparation of composite biochar/TiO₂ [3].

3.3. Raman spectroscopic analysis of porous biochar

In this experiment, Raman spectroscopy analysis revealed that there are two typical catalytic characteristic peaks of rutile TiO₂ in the composite material biochar/TiO₂ spectrum, one located at 517/cm and the other at 637/cm; There are also two characteristic peaks in carbon materials, one located at 1289/cm and the other at 1617/cm, corresponding to the D and G bands in the carbon layer. As the D band is graphitized carbon, it can be proven that the carbon layer has a high degree of

graphitization and good electrical conductivity. From this, it can be determined that the porous biochar prepared based on the pineal shell in this experiment has a high carrier separation efficiency in the photocatalytic degradation reaction. Therefore, this composite material is more conducive to the efficient photocatalytic degradation reaction of Rhodamine B.

3.4. Analysis of photocatalytic performance of composite material biochar/TiO₂

In order to clarify the photocatalytic performance of the composite material biochar/TiO₂ prepared this time, researchers used the composite material biochar/TiO₂ to adsorb and degrade Rhodamine B solution under light conditions, and analyzed the photocatalytic performance of the composite material biochar/TiO₂ by examining the removal effect of Rhodamine B.

Through observation of experimental phenomena and analysis of experimental data, it was found that the absorbance of Rhodamine B solution showed a significant upward trend at a wavelength of 550nm, indicating a significant increase in reaction rate under these conditions. Therefore, it can be determined that the composite material biochar/TiO₂ has good photocatalytic performance in the photocatalytic degradation of Rhodamine B at a wavelength of 550nm. Furthermore, through further observation and analysis, it was found that under the conditions of using composite material biochar/TiO₂ as a photocatalyst, there is a significant correlation between its catalytic effect and light exposure time. The longer the light exposure time, the more obvious the catalytic effect [4]. When the illumination time reaches 120 minutes, the adsorption degradation rate of Rhodamine B will exceed 90%. Table 2 shows the variation of Rhodamine B absorbance with light exposure time based on composite material biochar/TiO₂ photocatalyst in this experiment:

Table 2. Changes in Rhodamine B absorbance with light exposure time based on composite material biochar/TiO₂ photocatalyst in this experiment.

Serial number	Illumination time	Absorbance	Serial number	Illumination time	Absorbance
1	0min	0Abs	6	75min	0.76Abs
2	15min	0.27Abs	7	90min	0.82Abs
3	30min	0.42Abs	8	105min	0.94Abs
4	45min	0.50Abs	9	120min	1.28Abs
5	60min	0.68Abs			

After the preparation and photocatalytic performance analysis of the photocatalytic composite material biochar/TiO₂, the researchers have drawn the following experimental conclusions. (1) The biochar in this composite material has a uniform porous structure, which can uniformly adsorb nano TiO₂ particles on its surface with its strong adsorption performance, and create good dispersion conditions for it with the porous structure to ensure sufficient contact between it and the reactants. (2) The biochar obtained through this experimental method is a porous structure with a large specific surface area. It has high carrying capacity for TiO₂ photocatalyst and adsorption capacity for photocatalytic degradation reactants, which can create favorable conditions for the smooth and efficient progress of photocatalytic degradation reaction. (3) The composite material biochar/TiO₂ prepared in this experiment can form typical photocatalytic and adsorption characteristic peaks in Raman spectroscopy analysis, and can be used as an adsorption photocatalyst in photocatalytic degradation reactions. (4) In the photocatalytic degradation of organic

wastewater based on Rhodamine B dye, the composite material biochar/TiO₂ exhibits good photocatalytic effect at a wavelength of 510nm. Using it as a catalyst and controlling the illumination time at 120min or above can effectively remove Rhodamine B pollutants from wastewater.

4. Conclusions

In summary, in the photocatalytic degradation treatment of organic wastewater based on Rhodamine B dye, nano TiO₂ particles can be used as photocatalysts alone, but due to their own aggregation effect, the amount of photocatalyst used in practical applications is usually large, and the photocatalytic effect is not ideal. Based on this, researchers can use the common pineal shell in the market today as a carbon source, modify it with KOH and activate it with HCL, and then produce porous biochar through high-temperature sintering. Then, it can be combined with nano TiO₂ particles to form a composite material biochar/TiO₂. This can effectively prevent the aggregation of nano TiO₂ particles and provide more reaction sites for photocatalytic degradation reactants under the combined action of porous structure and adsorption performance, achieving good photocatalytic effect.

Conflicts of Interest

The author declares that there is no conflict of interest regarding the publication of this article.

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