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TiO_2/g-C_3N_4@HPBC Photoanode in PMFC for Shipboard Oily Wastewater Degradation

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Abstract

This study addresses the problems of high energy consumption, high cost, and incomplete removal existing in traditional treatment methods for oily wastewater from ships, and proposes and constructs a Photocatalytically-assisted Microbial Fuel Cells (PMFC) device for efficient and green treatment. The device couples photocatalysis with Microbial Fuel Cell (MFC) technology, utilizing ·OH and $\cdot O_2^-$ generated by semiconductor photocatalysts under illumination to synergistically degrade oil pollutants with microbial metabolism. Specifically, pine cone shell biochar (PBC) modified by high-temperature carbonization (350°C) and H_2O_2 oxidation (HPBC) is used as the anode substrate, which significantly improves its hydrophilicity (contact angle reduced to 44.637°) to facilitate the attachment of photocatalysts and microorganisms. Then, a TiO_2/g-C_3N_4 heterojunction photocatalyst (TiO_2/g-C_3N_4@HPBC) is loaded to construct a composite photoanode. A two-chamber PMFC reactor is built, using an emulsified diesel solution prepared with an inorganic salt medium as the anolyte, a potassium ferricyanide solution as the catholyte, and a composite microbial community as the anode microorganisms. The results show that the maximum output voltage of 0.6389V and oil degradation rate of 76.34% of PMFC under illumination are significantly higher than the maximum output voltage of 0.5832V and oil degradation rate of 71.72% under dark conditions, confirming that the photocatalytic effect effectively improves the power generation performance and pollutant degradation efficiency of the system. The PMFC device provides an energy-saving and efficient potential solution for the treatment of oily wastewater from ships.

Keywords

PMFC, Shipboard Oily Wastewater, TiO_2/g-C_3N_4.

1. Introduction

In recent years, with the rapid development of the shipping industry, pollution of marine environments caused by ship wastewater has aroused widespread concern from all sectors. Among ship wastewater, oily wastewater has become a key research object in related fields due to its huge generation volume, high treatment difficulty, and severe harmfulness. Pollutants in ship oily wastewater are mostly toxic and prone to forming oil films on the water surface, which hinder oxygen dissolution into water bodies, thereby harming the growth, development, and reproduction of aquatic organisms. If directly discharged, it will inevitably cause extremely serious pollution to the marine environment, and ultimately bring considerable adverse effects on human health. Traditional treatment methods for ship oily wastewater, including physical, chemical, and biological processes, have disadvantages of long treatment time, high energy consumption, excessive costs, and incomplete pollutant removal. Therefore, developing green and low-cost efficient methods for treating ship oily wastewater is particularly important.

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Photocatalytically-assisted Microbial Fuel Cells (PMFC) belong to green technologies, which can protect the environment while saving energy, realize the combination of biological energy and light energy, give full play to the advantages of the two clean energies, and improve power generation efficiency and pollutant degradation efficiency [1].

At present, there are mainly three ways to realize the application of photocatalytic technology in microbial fuel cells. The first is to construct photocatalytic electrodes using semiconductor materials to couple with a two-chamber MFC system. The second is to use MFC power generation to drive photoelectrocatalytic pollutant treatment. The third is to combine MFC with photocatalytic technology for wastewater treatment, where each process is carried out independently and connected in sequence.

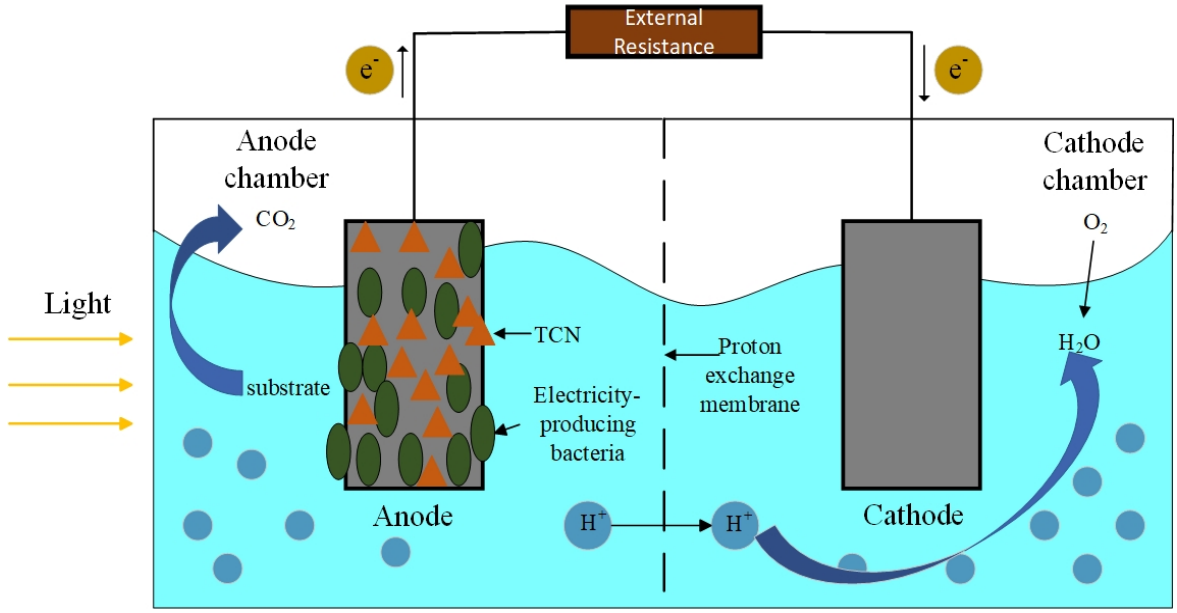

Figure 1. Schematic diagram photocatalytic microbial fuel cells

The method of constructing photocatalytic electrodes using semiconductor materials and coupling them with a dual-chamber MFC system is currently a common approach.The coupled battery configuration only needs to add a light source on the side of the photoelectrode, and the rest of the structure is basically the same as the two-chamber MFC configuration, which is composed of an anode chamber, a cathode chamber, and a proton exchange membrane separating the two chambers. The cathode and anode are connected through an external load. Wang et al. proposed a microbial fuel cell system with a tightly coupled photocatalytic anode for the synergistic degradation of the refractory substance 2,4,6-trichlorophenol (2,4,6-TCP) [2]. At an initial concentration of 200 mg/L, the removal efficiency within 10 hours was 79.3%, which was higher than that of MFC (66.0%) and photocatalysis (56.1%). The corresponding power density increased from 17.6 W/m^3 to 19.8 W/m^3. Si et al. enhanced the degradation of the antibiotic metronidazole (MNZ) by coupling a photocatalytic and microbial fuel cell system[3]. By attaching the photocatalyst TiO_2 to the bioanode to construct the battery system, the experimental results showed that compared with the traditional microbial fuel cell, the removal rates of metronidazole (MNZ) and total organic carbon (TOC) increased by 1.30 times and 1.24 times, respectively.

In semiconductor materials, graphitic carbon nitride (g-C_3N_4) has become a research hotspot in the field of photocatalysis due to its visible light-responsive bandgap structure (2.69 eV), excellent thermochemical stability, and simple synthesis process. It is worth noting that when

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this material is used to construct a heterojunction system with TiO_2, the interfacial synergistic effect can effectively inhibit the recombination of photogenerated carriers, thereby significantly improving the quantum efficiency of TiO_2-based photocatalysts [4]. This heterostructure design provides a new technical path for expanding the light absorption range and optimizing the charge separation efficiency, showing good application prospects in the fields of environmental purification and energy conversion.

According to the band theory, crystalline materials are divided into conductors, semiconductors, and insulators. The core of photocatalytic reactions lies in semiconductor materials. Semiconductors exhibit optical properties different from those of metals and insulators. Their band structure is composed of a high-energy conduction band (CB) and a low-energy valence band (VB). The region between the conduction band and the valence band is called the forbidden band, where there are no other energy levels[5].. Therefore, the energy difference between the highest energy level of the valence band and the lowest energy level of the conduction band in a semiconductor is called the band gap (Eg) Different from metals, which have continuous electron energy levels, insulators have too large a band gap, making it difficult for electrons in the valence band to obtain enough energy to transition to the conduction band to form carriers. In contrast, semiconductors have a smaller band gap, which allows semiconductor photocatalysts to be excited to exhibit photocatalytic activity when illuminated by light with energy equal to or exceeding the band gap [6].

This process can be summarized as follows: when a semiconductor material is photoexcited, electrons in the valence band (VB) absorb photon energy and transition to the conduction band (CB), forming photogenerated electrons (e^-) and leaving holes (h^+) in the valence band. These photogenerated carriers have two competitive paths: on the one hand, part of the electron-hole pairs will quickly recombine, leading to energy quenching; on the other hand, the uncombined electrons migrate to the catalyst surface under the drive of an electric field and undergo reduction reactions with H_2O/O_2 to generate H_2O_2 and superoxide radicals ($\cdot O_2^-$), while the holes oxidize $H_2O/\cdot OH$ to produce hydroxyl radicals ($\cdot OH$). These two radicals can efficiently degrade organic pollutants [7].

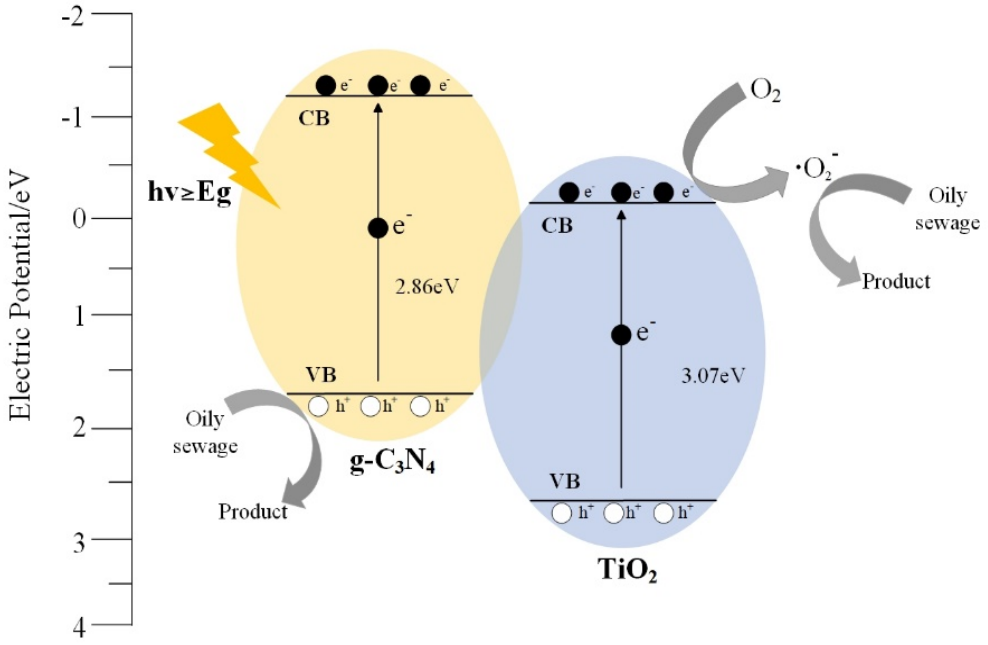

Figure 2. Schematic diagram of photocatalytic degradation of ship oily wastewater by TiO_2/g-C_3N_4 composite

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2. Experimental Preparation

2.1. Preparation of MFC Anode.

Compared with traditional carbon felt, biochar exhibits unique comprehensive advantages as an anode material. The high-temperature pyrolysis process endows it with a developed porous structure and abundant surface functional groups, which not only significantly increases its specific surface area but also enhances substrate adsorption capacity—critical for improving electron transfer efficiency. The three-dimensional skeleton structure derived from natural biomass provides an ideal habitat for microorganisms, while its endogenous mineral components can participate in microbial energy metabolism. These materials can achieve environmentally friendly degradation after use, avoiding secondary pollution. Based on the consideration of resource recycling, this study selects pine cone shells with wide sources and low cost as raw materials, in line with the development direction of green energy technology.

The scales of pine cone are cut into 1 cm×1 cm thin slices, which are placed in absolute ethanol for ultrasonic vibration for 30 minutes to remove surface impurities, then ultrasonically cleaned with deionized water for 30 minutes. After that, the slices are taken out and dried in an electric hot-air drying oven at 90℃ for 6 hours. The dried pine cone shells are put into a box-type resistance furnace for vacuum carbonization at 400℃ for 30 minutes, and then the samples are taken out after cooling to room temperature with the furnace to prevent the biochar from being oxidized when exposed to air at high temperature.

To improve the performance of pine cone shell biochar (PBC), an oxidation modification treatment is carried out, which aims to enhance its specific surface area and porosity by increasing surface oxidation functional groups (such as hydroxyl, carboxyl and aldehyde groups), so as to strengthen its adsorption capacity and improve the adhesion effect of photocatalyst. The specific operation is as follows: mix 5 grams of biochar with 50 milliliters of 10% H_2O_2 solution, ultrasonicate for 30 minutes, then let stand for 6 hours, after that rinse the biochar with deionized water to remove residual H_2O_2, and finally dry at 80°C for 2 hours. H_2O_2@PBC is prepared, which is abbreviated as HPBC hereinafter.

The wettability of material surfaces can be quantitatively characterized by contact angle measurement. This experiment uses an optical imaging system to record droplet morphology in real time. Specifically, the sample to be tested is horizontally fixed on the test platform, and a static water droplet is generated on the material surface via a microsyringe to form a solid-liquid-gas three-phase interface. The contact angle θ is calculated by mathematically fitting the droplet profile based on the Young-Laplace equation. According to the thermodynamics equilibrium principle, when $\theta < 90°$, it indicates that the material surface energy is higher than the liquid surface tension, showing hydrophilic characteristics (the smaller the θ, the stronger the hydrophilicity); conversely, $\theta > 90°$ reflects lower material surface energy, exhibiting hydrophobic properties (the larger the θ, the stronger the hydrophobicity). 90° serves as the critical wettability threshold, directly determining the spreading behavior of liquids on solid surfaces. The figure below shows contact angle images of PBC at different carbonization temperatures.

The contact angle of water droplets on biochar reflects the hydrophilic ability of the material. As shown in the figure, pine cone shells carbonized at different temperatures and modified samples are presented. The water droplet contact angle measurement shows that:

(1) Figure (a) carbonized pine cone shell has a contact angle of 85.880°, indicating certain hydrophobicity;

(2) Figure (b) carbonized pine cone shell shows a reduced contact angle of 74.491°, with weakened hydrophobicity;

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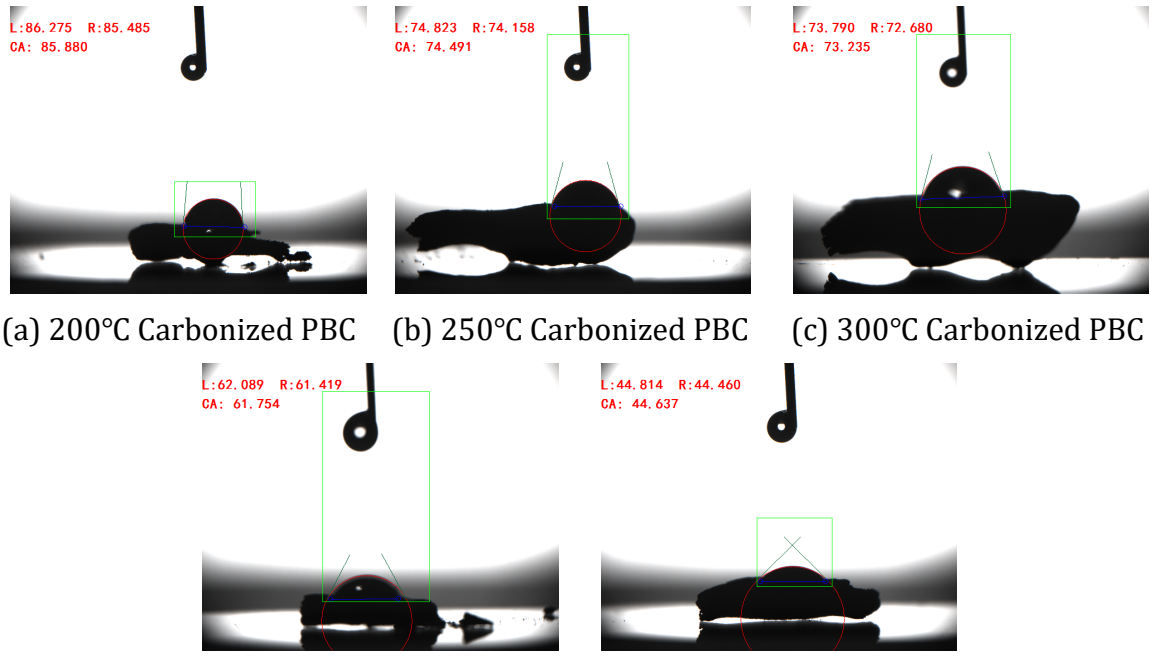
(3) Figure (c) carbonized pine cone shell has a contact angle of 73.235°, with further decreased hydrophobicity;

(4) Figure (d) carbonized pine cone shell exhibits a contact angle of 61.754°, demonstrating a significant improvement in hydrophilicity;

(5) Figure (e) H_2O_2-modified carbonized pine cone shell has a contact angle as low as 44.637°, with remarkably enhanced hydrophilicity.

This indicates that the hydrophilicity of pine cone shells increases with the rise of carbonization temperature. H_2O_2 modification introduces more hydrophilic functional groups (such as hydroxyl groups) to the surface, improving hydrophilicity and facilitating microbial adhesion, thus enabling the formation of a favorable microbial biofilm in microbial fuel cells.



(a) 200℃ Carbonized PBC (b) 250℃ Carbonized PBC (c) 300℃ Carbonized PBC

(d) 350℃ Carbonized PBC (e) H_2O_2-Modified 350℃ PBC

Figure 3. Contact Angles of PBC at Different Calcination Temperatures

2.2. Preparation of Photocatalytic Anode.

g-C_3N_4 was synthesized via thermal polymerization by placing 5 g of urea in an alumina crucible with a lid, which was then placed in a box-type resistance furnace. The temperature was raised at a rate of 5 °C/min to 500 °C and maintained for 2 h. After natural cooling to room temperature, the sample was taken out and thoroughly ground in an agate mortar to obtain a pale yellow g-C_3N_4 powder.

For the preparation of TiO_2/g-C_3N_4@HPBC (denoted as TCN@HPBC hereinafter), 1 g of g-C_3N_4 powder and 0.5 g of nano-TiO_2 were ground in an agate mortar for 30 min. The mixture was added to 50 mL of absolute ethanol and dispersed by sonication for 30 min, followed by the addition of 0.6 g of HPBC and further sonication for 1 h. The HPBC was then dried at 80 °C for 2 h in a thermostatic drying oven, and this procedure was repeated twice. Finally, the HPBC was placed in a muffle furnace, heated to 380 °C at a rate of 2 °C/min, calcined for 1 h, and cooled with the furnace to yield TCN@HPBC.

2.3. SEM Analysis of Photocatalytic Anode.

Scanning electron microscopy (SEM) relies on the principle of electron beam-sample interaction, achieving micro-nano scale morphological characterization through signal

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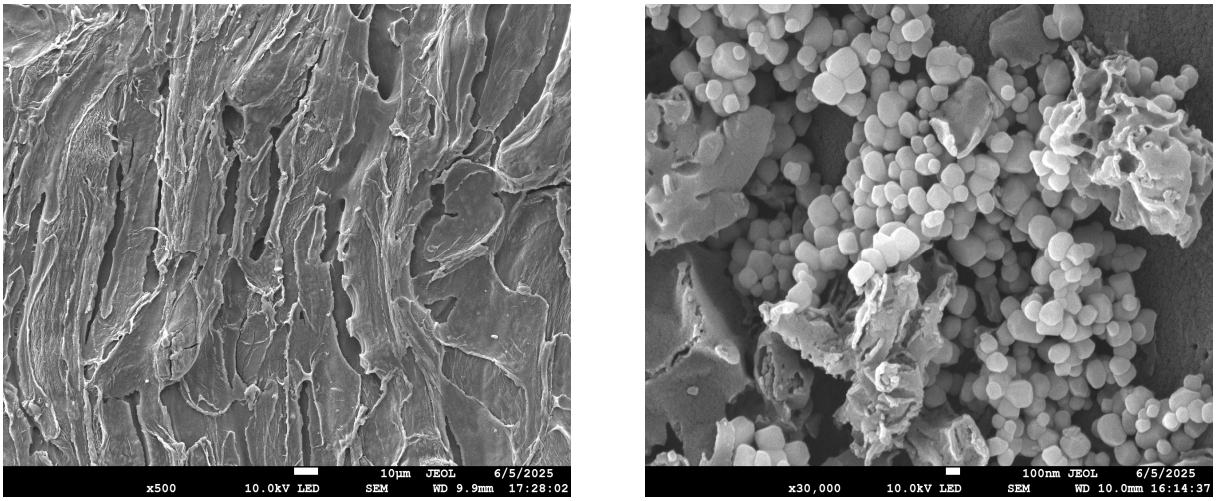
acquisition and image reconstruction. In the experimental process, samples were first fixed using conductive adhesive bonding, followed by Au-Pd alloy coating via an ion sputtering apparatus to enhance surface conductivity. Subsequently, under vacuum conditions, multi-stage magnification scanning was performed by regulating the electron optical system, ultimately acquiring secondary electron images of the sample surface topology.



(a) HPBC (b) TiO_2/g-C_3N_4@HPBC

Figure 4. Photoelectrode SEM image

From the figure, it can be observed that TiO_2 exhibits an irregular spherical shape, while the surface of g-C_3N_4 is relatively smooth with typical two-dimensional layered characteristics. As the substrate, biochar provides a relatively rough and porous surface, enabling TiO_2 to disperse on the surface of g-C_3N_4 and form a hierarchical structure of TiO_2/g-C_3N_4@HPBC.

2.4. Cultivation of Electricity-Generating Strains and Preparation of Anolyte.

The construction procedure of the anodic composite microbial community for microbial fuel cells is as follows: First, single-strain activation cultivation was conducted for *Clostridium butyricum*, *Shewanella*, *Rhodopseudomonas palustris*, *Bacillus subtilis*, and *Saccharomyces cerevisiae*. Subsequentially, the five activated strains were inoculated into an inorganic salt medium for combined cultivation under the conditions of 35°C, a shaking speed of 180 r/min, and a cultivation duration of 24 h, so as to facilitate the rapid proliferation of microorganisms and establish a stable composite microbial community system.

The inorganic salt medium used had the following formulation: ethylenediaminetetraacetic acid disodium salt (0.01 g/L) served as the metal ion chelating agent, a phosphate buffer system (potassium dihydrogen phosphate 3 g/L and dipotassium hydrogen phosphate 1.5 g/L) maintained pH stability, ammonium nitrate (2 g/L) acted as the nitrogen source, and trace inorganic salt components (anhydrous calcium chloride 0.01 g/L, magnesium sulfate heptahydrate 0.1 g/L) were added. The medium was prepared with distilled water, and the initial pH was adjusted to 7.5 to optimize the microbial growth environment.

The anolyte used in the experiment was an emulsified diesel solution prepared with an inorganic salt medium. The specific preparation method of the anolyte was as follows: on a sterile bench, the inorganic salt medium was prepared according to the above-mentioned reagent ratio, and the medium was placed in a pressure steam sterilizer at 121 °C for 15 min to kill miscellaneous bacteria. Then, 2 g of 0# diesel and an appropriate amount of Span 80 emulsifier were injected into the medium, and the solution was fixed to 1 L. The mixture was sufficiently stirred with a magnetic stirrer to achieve the effect of emulsified diesel, thereby

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obtaining an anolyte with an oil concentration of 2 g/L. Potassium ferricyanide, as an excellent electron acceptor, can significantly improve the electrochemical performance of the cathode[8]. Therefore, in this experiment, potassium ferricyanide was fully mixed and dissolved with deionized water to prepare a 30 mM potassium ferricyanide solution as the catholyte of the MFC.

2.5. Construction and Startup of PMFC Device.

The traditional dual-chamber device was constructed under aseptic conditions on a laminar flow bench. The MFC battery casing was sterilized by ultraviolet irradiation for 2 h, after which 50 mL of anolyte and catholyte were respectively injected into the two electrode chambers. Specifically, 1 mL of composite microbial culture solution was added to the anolyte. The prepared 1 cm×1 cm TCN@HPBC photoelectrode was immersed in the anolyte as the anode. A 20W LED lamp was positioned outside the anode chamber to drive photocatalytic reactions. The cathode used a carbon rod (0.5 cm diameter, 15 cm length) as the electrode.

For the anode chamber, two small holes were designed: one for inserting the anode electrode, and the other sealed with a silicone plug to maintain an anaerobic environment for microbial activity. In the cathode chamber, one hole was for placing the carbon rod, while the other was kept open to the atmosphere, ensuring effective combination of electrons, protons, and ambient oxygen to facilitate the oxidation reaction.

3. PMFC Device for Treating Ship Oil-Contaminated Wastewater

3.1. Output Voltage Performance Analysis of PMFC Device.

The output voltage test serves as a crucial metric for evaluating the electricity-generating capacity of a PMFC device. During the test, a CT4008 battery detection system was employed. A 1000 Ω resistor was externally connected across the PMFC device, and the system was configured to detect the voltage at set time intervals. The collected voltage data was automatically saved to the system. Concurrently, a dark control group of PMFCs was established for comparative experiments.

The output voltage of the PMFC under continuous operation is presented in Figure. 5 This figure illustrates the dynamic evolution of voltage over time in a photocatalytic microbial fuel cell (PMFC) under both illuminated and dark conditions.

Under illumination, during the initial phase, the voltage remained stable between 0.5 and 0.7 V. This phenomenon can be primarily attributed to the rapid proliferation and growth of the inoculated microorganisms in the anolyte at the onset of the device operation, with substantial adhesion to the anode material, thereby enhancing the electrical conductivity of the battery. Simultaneously, the photocatalytic reaction stimulated the semiconductor catalyst to generate electron-hole pairs, propelling the steady progression of the electrode reactions and consequently elevating the voltage. Subsequently, the voltage began to decline appreciably. This decline may stem from the continuous depletion of internal substrates as the reaction advances, reducing the substances participating in the electrochemical reactions. Alternatively, it could be due to the gradual attenuation of the photocatalytic material's activity under prolonged illumination, which impedes electron transport and electrode reaction efficiency. In the later stage, the voltage decline decelerated and approached a plateau, signifying the establishment of a new equilibrium in the internal reactions of the battery, where the substrate consumption rate and reaction rate achieved relative stability.

Under dark conditions, the initial voltage was slightly lower than that under illumination, at approximately 0.58 V, followed by a rapid decline. This is because without the energy provided by light, the microbial fuel cell relies mainly on microbial metabolism of substrates for power generation, leading to faster substrate consumption. In the intermediate stage, the voltage

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continued to drop. Although the decline rate slowed down compared to the previous stage, it did not stop, reflecting the gradual weakening of microbial substrate metabolism or the increase in internal battery impedance, which interfered with electron transfer and voltage output. In the later stage, the voltage dropped below 0.2 V and stabilized, at which point the substrate was nearly exhausted and microbial metabolic activity was extremely weak.

Comparing the curves under the two conditions, it can be seen that the voltage of the PMFC under illumination was consistently higher than that under dark conditions throughout the entire time span. This fully demonstrates that photocatalysis can provide additional energy to the microbial fuel cell, promote electron transfer and electrode reactions, improve the power generation performance of the battery, and extend the duration for maintaining high voltage output.

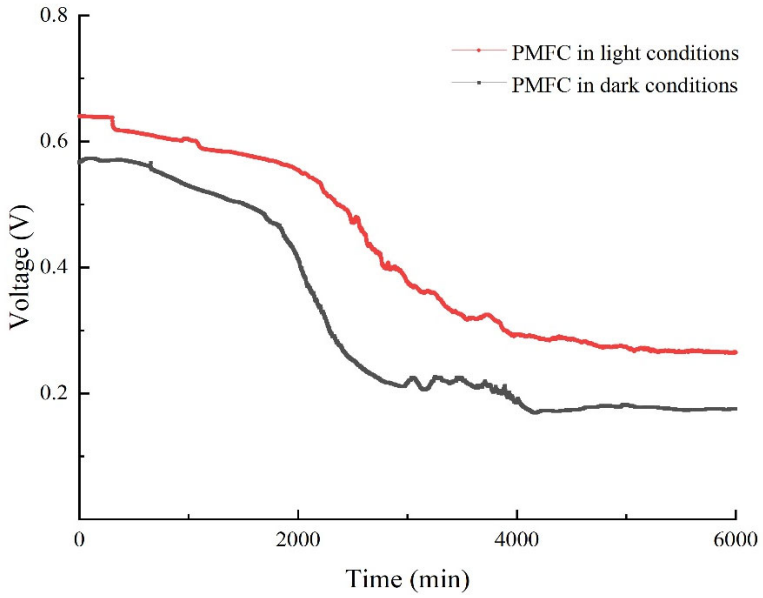

Figure 5. Output voltage of PMFC under illuminated and dark conditions

3.2. Degradation Performance of PMFC Device for Shipboard Oily Wastewater.

In this experiment, the oil degradation rate refers to the change rate of oil content in oily wastewater before and after treatment by the MFC device under specified conditions, which is an important parameter to measure the device's ability to purify oily sewage. The oil content was detected and analyzed by a UV - 1800 ultraviolet spectrophotometer. Before detecting the oil degradation rate, the standard curve of emulsified oil should be drawn first. The specific method is to prepare emulsified diesel from marine diesel and Span 80 emulsifier. Using emulsified diesel as the solute and petroleum ether as the solvent, different concentrations of petroleum ether emulsified oil solutions (1.0, 5.0, 10.0, 20.0, 40.0, 60.0, 80.0, 100.0 mg/mL) were obtained by diluting at different multiples as standard solutions. The standard curve of emulsified oil was drawn based on the principle of testing the absorbance of solutions with different concentrations at the wavelength of 256 nm by an ultraviolet spectrophotometer. The corresponding curve formula is $Y=0.01841x-0.00245$, $R^2=0.99914$, as shown in the Figure. 6 below.

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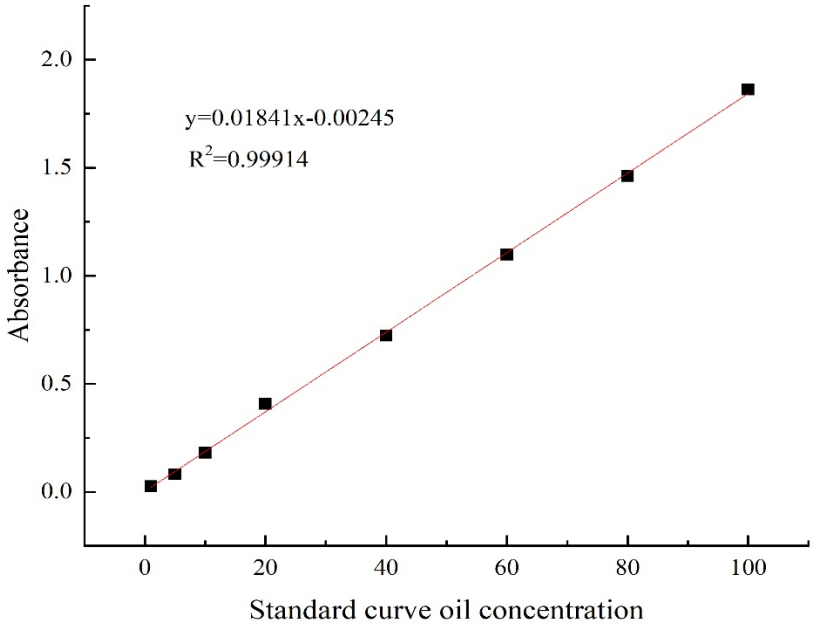

Figure 6. Standard curve of emulsified diesel

For the oil degradation rate test of microbial fuel cells: 10 mL of anolyte was taken, to which 0.6 g of NaCl and 5 mL of petroleum ether were sequentially added. The solution was transferred to a test tube and thoroughly mixed by vortex oscillation for 10 minutes followed by ultrasonic vibration for 30 minutes. The mixture was centrifuged at 5670 rpm for 10 minutes to separate water, impurities, and petroleum ether. After solution stratification, the supernatant was extracted, treated with anhydrous sodium sulfate, and filtered to obtain an emulsified oil solution extracted by petroleum ether. The volume was adjusted to 1 mL with petroleum ether, and the oil content in the degraded water sample was detected using a UV-1800 ultraviolet spectrophotometer at a wavelength of 254 nm.

The oil degradation rate of the MFC device can be calculated according to Formula (1).

$$\eta = \frac{C_0 - C}{C_0} \times 100\% \quad (1)$$

Where η represents the oil degradation rate of the PMFC device, C_0 is the content of emulsified oil in the oily wastewater before degradation, and C is the content of emulsified oil in the oily wastewater after degradation.

The PMFC device with TCN@HPBC anode material was subjected to a continuous 7-day degradation test under dark and illuminated conditions. According to the detection results in Figure. 7, the oil degradation rates of the PMFC device under different lighting conditions differ significantly. The degradation rate under dark conditions is 71.72%, while that under illuminated conditions reaches 76.34%. Experiments show that when other influencing factors are the same, lighting determines the strength of the PMFC device's oil removal capability. This is mainly because photogenerated electrons migrate to the catalyst surface under the action of an electric field, undergoing redox reactions with water and oxygen to produce H_2O_2 and $\cdot O_2^-$. Meanwhile, holes with strong oxidizing properties react with water molecules to generate hydroxyl radicals. Both $\cdot OH$ and $\cdot O_2^-$ have extremely strong oxidation capabilities, which can effectively oxidize and degrade various organic pollutants.

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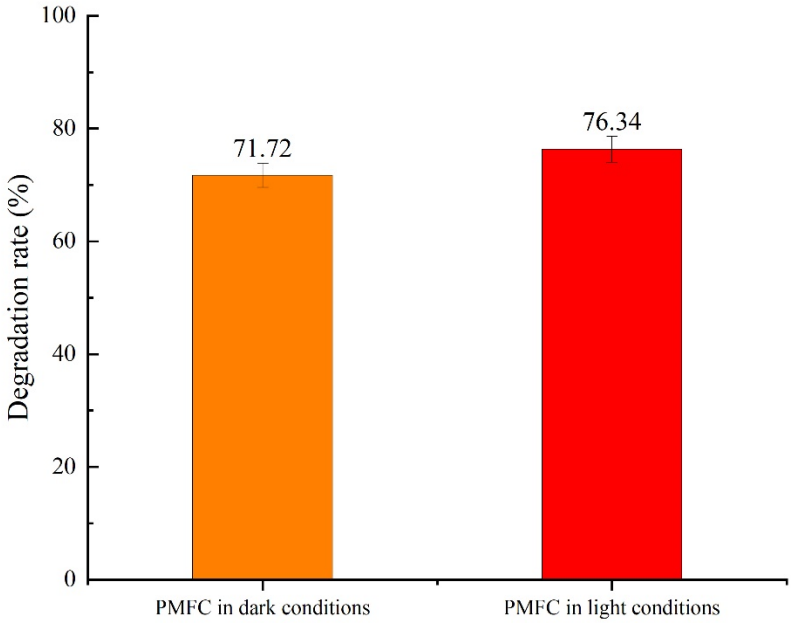

Figure 7. Degradation rates of emulsified diesel by PMFC device under illuminated and dark conditions

4. Conclusion

In this study, a photocatalytic microbial fuel cell (PMFC) device with a composite photocathode was successfully developed. The device is constructed based on a TiO_2/g-C_3N_4 (TCN) heterojunction photocatalyst supported on H_2O_2-modified pine cone biochar (HPBC), namely TCN@HPBC. This novel PMFC is designed for efficient treatment of ship oil-contaminated wastewater. Experimental results show that the modification significantly enhances the hydrophilicity of the anode material, with a contact angle as low as 44.637°, which is beneficial for microbial adhesion. The constructed two-chamber PMFC device exhibits superior performance under illumination: the output voltage stably remains at 0.5-0.7 V for a longer period, and the degradation rate of ship oil-contaminated wastewater reaches 76.34%, significantly higher than 71.72% under dark conditions. This fully confirms the key synergistic effect of photocatalysis - photo-generated electrons and holes excited by light effectively promote the generation of strongly oxidizing radicals (·OH, $\cdot O_2^-$). These radicals, in conjunction with microbial metabolism, not only significantly enhance the degradation efficiency of organic pollutants but also improve the electron transfer efficiency and overall power generation performance of the system. The PMFC device demonstrates good performance and application potential in treating ship oil-contaminated wastewater, providing a new approach for the development of green and efficient ship wastewater treatment technologies.

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