

# EXPLORING TiO2-PP AS A REUSABLE FLOATING PHOTOCATALYST FOR HUMIC ACID AND IRON REMOVAL IN PEAT WATER

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## ABSTRACT

In this study, we fabricate a  $TiO_2$ -PP catalyst and analyze its use for peat water photodegradation. The photocatalyst is a thin layer of  $TiO_2$  deposited on the surface of polypropylene (PP) grains by the thermal milling method. Scanning electron microscopy (SEM) and energy-dispersive x-ray spectroscopy (EDS) images indicate that the fabrication successfully deposited  $TiO_2$  particles on the PP grain surface homogeneously well. The results show that photocatalysis of peat water using  $TiO_2$ -PP with solar irradiation is more effective than the UV lamp. After photocatalysis for 40 hours with solar irradiation, the humic acid content in peat water decreases significantly, accompanied by a decrease in Fe concentration. When being reused,  $TiO_2$ -PP photocatalyst shows performance above 92% in the fourth iteration, while in the fifth iteration, the performance decreases to 83%. These results show that  $TiO_2$ -PP has the potential to be applied as a reusable floating photocatalyst to reduce the humic acid and iron content in peat water.

Keywords: peat water; humic acid; iron; photocatalyst; polypropylene; titanium dioxide

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## INTRODUCTION

Indonesia has a substantial peatland expanse stretching from eastern Sumatra to southern Papua<sup>[1]</sup>. These peatland areas are distributed from coastal regions deep into the hinterlands. The limited service of clean water provided by Perusahaan Daerah Air Minum (PDAM)<sup>[2]</sup> forces the residents in peatland areas to use peat water for their daily needs<sup>[3]</sup>. Peat water contains high levels of organic compounds and iron content. Among these organic compounds are humic acids, consisting of humic acid, fulvic acid, and humin<sup>[4]</sup>. The presence of humic acids results in a low pH value, a brownish color, and the emergence of a pungent smell in peat water. Meanwhile, the iron content, besides affecting color and smell, can also lead to an oily layer on the water surface. Therefore, peat water must be treated to make it suitable for sanitation and consumption.

Several methods of wastewater treatment<sup>[5]</sup> and peat water treatment<sup>[6-8]</sup> have been studied and developed. One promising method involves employing a heterogeneous photocatalysis process using titanium dioxide (TiO<sub>2</sub>). This method is recognized for effectively mineralizing organic compounds under light radiation<sup>[9]</sup>. TiO<sub>2</sub> is a semiconductor material that has achieved broad utility not only in water treatment but also in the field of solar cells <sup>[10-11]</sup>. TiO<sub>2</sub> nanoparticles are commonly chosen as photocatalysts in water treatment applications due to their excellent performance in degrading organic pollutants. Additionally,  $TiO_2$  nanoparticles are water-insoluble, non-toxic, and demonstrate substantial photocatalytic activity attributed to their expansive surface area and robust chemical stability <sup>[12]</sup>.  $TiO_2$  is a semiconductor characterized by a band gap energy of 3.2 eV <sup>[13]</sup>, signifying that the photocatalytic process using these nanoparticles necessitates irradiation within the ultraviolet (UV) wavelength spectrum.

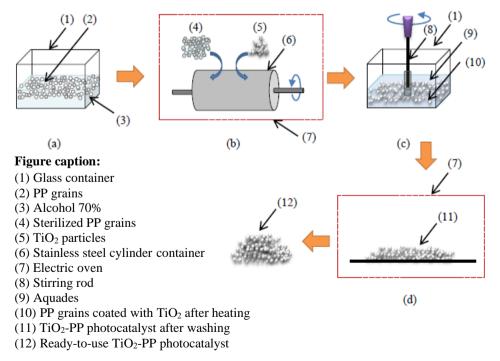
Although  $TiO_2$  nanoparticles are promising for peat water treatment, several challenges are associated with their direct application. Due to their high density,  $TiO_2$  powder tends to sink in water, leading to suboptimal light exposure and a consequent decrease in the photocatalytic material's performance. An additional concern arises from the need for post-treatment to separate  $TiO_2$  powder from the water when they are mixed.

This study addresses the outlined issues by engineering  $TiO_2$ , used as a photocatalyst, and testing its application on peat water. Using thermal milling,  $TiO_2$  will be deposited onto the polypropylene (PP) surface to prevent sinking and ease its separation from the treated water. This research also explores the effectiveness of the  $TiO_2$ -PP photocatalysis process under the irradiation of UV lamps and sunlight. The performance of  $TiO_2$ -PP as a reusable photocatalyst will also be assessed.

# METHODS

## Fabrication of TiO<sub>2</sub>-PP

 $TiO_2$ -PP was fabricated by immobilizing  $TiO_2$  nanoparticles onto the surface of the PP grain. PP was chosen as the buffer material for  $TiO_2$ based on its mass density, light transmittance properties, and melting point characteristics. Due to PP's lower density than water, the resulting  $TiO_2$ -PP would float, ensuring optimal light exposure. Additionally, this



**Figure 1.** TiO<sub>2</sub> coating process on PP surface: (a) PP cleaning, (b) thermal milling deposition of TiO2 particles on PP grains (TiO2-PP), (c) TiO2-PP re-cleaning, and (d) drying.

photocatalyst would be easily separate from the treated water. Prior to fabrication, PP grains

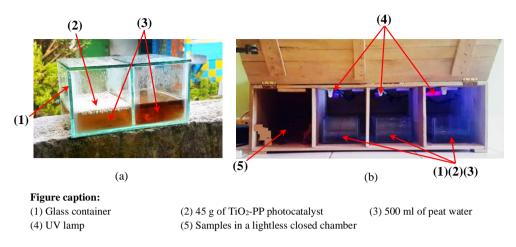


Figure 2. Irradiation of peat water using (a) sunlight exposure and (b) UV lamp

were cleaned with 70% alcohol. Subsequently, the deposition was conducted through thermal milling. PP and TiO<sub>2</sub> were introduced into a rotating cylindrical container and heated to a temperature of  $175^{\circ}$ C for 40 minutes. This process melted the surfaces of the PP grains, facilitating the immobilization of TiO<sub>2</sub> powder onto them, resulting in the formation of TiO<sub>2</sub>-PP beads. Before being used in the photocatalysis process, TiO<sub>2</sub>-PP was first cleaned with 70% alcohol and then dried. The schematic details of this process are shown in Figure 1.

## Application of TiO<sub>2</sub>-PP to Peatwater

Following the fabrication of  $TiO_2$ -PP, the subsequent step is applying the photocatalyst to peat water. In this step, 45 g of  $TiO_2$ -PP was mixed with 500 ml of the sample and placed in a transparent container. Sunlight and UV lights were separately used to initiate the photocatalysis process. For comparison, two control samples of peat water were also prepared: one with  $TiO_2$ -PP but without exposure to light and another without  $TiO_2$ -PP but exposed to sunlight.

During photocatalysis under UV light irradiation, the samples were enclosed in a light-tight box to exclusively expose them to UV radiation. Visual representation of the samples during the photocatalysis process is depicted in Figure 2.The samples were solar irradiated from March to May 2021, accumulating an exposure duration of 40 hours (8 hours daily, from 08:00 to 16:00, over 5 days). UV lamp irradiation was 40 hours, with data measurements taken at 8-hour intervals. The UV intensity of the lamp was adjusted to approximate the UV intensity of sunlight measured during the photocatalysis process to facilitate the comparison of photocatalytic degradation results with two irradiation sources.

The utilized UV lamp is an ultra-electronic support stand (TL UV T5 8W) with an operating voltage range of 170-250 Volts. The intensities of UV light from both sources during the 40-hour photocatalysis process are presented in Table 1. We analyzed the results of the TiO<sub>2</sub>-PP application to peat water for five iterations to assess the photocatalyst's performance as a reusable photocatalyst.

Irradiation Time (WIB)	Average UV Intensity (mW/cm <sup>2</sup> )			
	Sunlight	UV Lamp		
8.00	0,78	1,19		
9.00	0,67	1,19		
10.00	0,62	1,15		
11.00	1,38	0,93		
12.00	1,64	1,21		
13.00	1,71	1,14		
14.00	2,32	1,22		
15.00	0,69	1,20		
16.00	0,20	0,88		
Average	1,11	1,12		
Intensity				
40 h (mW/cm <sup>2</sup> )				

 Table 1. UV intensity measurements during 40 hours of photocatalysis comparing sunlight and UV lamp

The performance of the photocatalyst can be quantitatively expressed by the percentage of undegraded substance in the sample, denoted as R%. This quantity is derived from Lambert-Beer's law, which establishes a proportional relationship between the absorbance of a solution and its concentration decrease. Mathematically, the expression for R% can be written as<sup>[14]</sup>

$$R\% = \frac{A}{A_0} \times 100 \,, \tag{1}$$

Here,  $A_o$  represents the maximum absorbance value of peat water under initial conditions, while *A* represents the maximum absorbance value of peat water after the photocatalysis process. Another parameter that can indicate photocatalyst performance is the ability of TiO<sub>2</sub>-PP to reduce dissolved substances in peat water, denoted as  $K_f$ %. The value of this parameter is calculated with the following equation<sup>[15]</sup>.

$$K_{\rm f}\% = \frac{A_0 - A}{A_0} \times 100\% \tag{2}$$

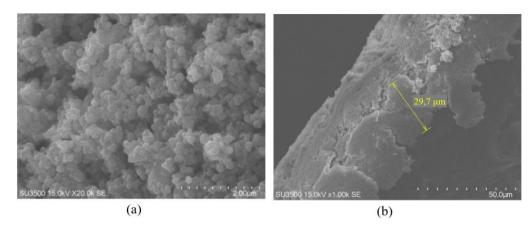
For each variation, measurements were performed on five samples subjected to identical treatments to ensure the precision and consistency of the result. The values used in the calculations are the average measurements obtained from these five samples.

#### **Characterization of TiO2-PP and Post-Treated Peat Water**

Characterization was conducted to analyze  $TiO_2$ -PP and the peat water after photocatalysis. The characterization of  $TiO_2$ -PP aimed to investigate surface morphology and catalyst composition using scanning electron microscopy (SEM) and energy dispersive x-ray spectroscopy (EDS), respectively. The peat water was characterized using UV-Vis spectroscopy to evaluate the concentration of dissolved substances following the photocatalysis process.

#### **RESULTS AND DISCUSSION**

Figure 3(a) shows an SEM image of the  $TiO_2$  surface with a magnification of 20,000 times. This image illustrates the effective and uniform coating of  $TiO_2$  particles on the surface of PP grains. The thickness of the deposited  $TiO_2$  can be ascertained from the cross-sectional image of the TiO<sub>2</sub>-PP photocatalyst magnified at 1,000 times [Figure 3(b)]. The TiO<sub>2</sub> coating on the surface of PP grains appears to have a thickness of approximately 29.7  $\mu$ m. Since the TiO<sub>2</sub> particles used have an average diameter of 187 nm, approximately 158 layers of TiO<sub>2</sub> particles are deposited on the surface of the PP grains.



**Figure 3.** Characterization of the photocatalyst in the form of (a) SEM image of the TiO<sub>2</sub>-PP surface at 20,000 times magnification and (b) cross-sectional image of TiO<sub>2</sub>-PP at 1,000 times magnification revealing a TiO<sub>2</sub> layer thickness on the PP surface of 29.7  $\mu$ m.

As a point of comparison, we performed an EDS analysis on TiO<sub>2</sub>-PP, and the findings are presented in Figure 4. It is evident that 84.14% of TiO<sub>2</sub> is deposited on the surface of PP (see caption in Figure 4). The content of C, representing 15.86% of the total, arises from open voids on the surface of PP that TiO<sub>2</sub> does not cover. Hence, the EDS spectrum corroborates the SEM image, indicating further evidence of a well-coated TiO<sub>2</sub> layer on the surface of PP. The formation of open voids on the PP surface does not adversely affect the photocatalytic performance. On the contrary, it can optimize the photocatalysis process, as the light incident on the open voids on one side of the PP surface can pass through to the other side, thereby exciting more TiO<sub>2</sub>.

Figure 5 illustrates the visual changes observed in peat water samples subjected to different treatments and irradiation. Figure 5(a) shows that peat water untreated with  $TiO_2$ -PP photocatalyst exhibited no discernible qualitative changes following a 40-hour exposure to

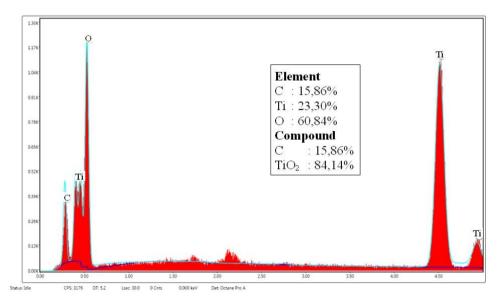
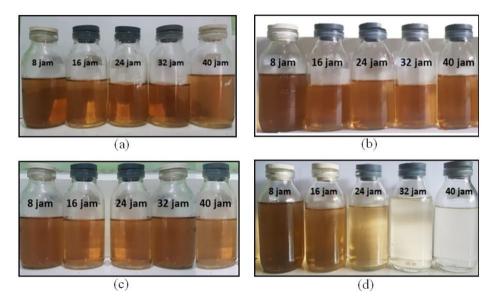


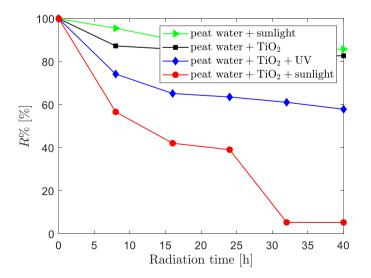
Figure 4. EDS spectrum of TiO<sub>2</sub>-coated PP surface.

sunlight. A similar observation was made for peat water treated with  $TiO_2$ -PP but not exposed to irradiation [Figure 5(b)]. For samples irradiated with UV light [Figure 5(c)], the reddish-brown color of the peat water gradually faded with increasing irradiation time. However, this change was relatively insignificant. A more pronounced alteration in color was exclusively witnessed in samples treated with  $TiO_2$ -PP subjected to sunlight irradiation, as evidenced in Figure 5(d). This emphasizes the substantial influence of the radiation source on photocatalytic degradation.



**Figure 5.** Results of irradiation on peat water for various treatments: (a) peat water with non-irradiated TiO<sub>2</sub>-PP photocatalyst, (b) peat water without photocatalyst irradiated under sunlight for 40 hours, (c) peat water with TiO<sub>2</sub>-PP photocatalyst irradiated with UV light for 40 hours, and (d) peat water with TiO2-PP photocatalyst irradiated under sunlight for 40 hours.

Quantitatively, the results presented in Figure 5 can be validated by examining the percentage of undegraded substances in the samples, denoted as R% [equation (1)]. The absorbance value, A, required to calculate this parameter is obtained from the UV-Vis



**Figure 6.** Photocatalytic degradation comparison, *R*%, between peat water samples treated with TiO<sub>2</sub>-PP photocatalyst under various irradiation conditions.

spectrum of the samples<sup>[16-17]</sup> at a wavelength of 275 nm, which corresponds to the characteristic wavelength of humic acid  $(250-275 \text{ nm})^{[18]}$ .

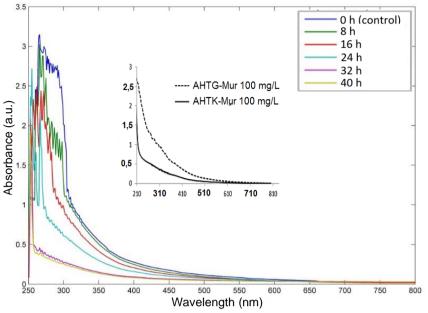


Figure 7. UV-Vis spectrum changes during the photodegradation of peat water using  $TiO_2$ -PP photocatalyst irradiated under sunlight (insert: degradation of humic acid compounds<sup>[16]</sup>).

A graphical representation of R% for each sample is depicted in Figure 6. R% values were calculated by averaging values of A from measurements conducted on 5 samples for each experimental condition. This graph demonstrates that, between 8 and 32 hours, the samples subjected to TiO<sub>2</sub>-PP catalyst and exposed to sunlight (as indicated by the red curve) exhibit the highest photodegradation rate, characterized by the steepest average curve slope. Additionally, photodegradation under UV irradiation (represented by the blue curve) exhibits comparatively less effective performance when contrasted with sunlight irradiation. These findings are consistent with those shown in Figure 5.

The minor photocatalytic activity observed in the samples without irradiation (black curve) is attributed to light exposure when the sample container [refer to Figure 2(b)] was opened for data measurements every 8 hours. Meanwhile, the slight decrease of R% values for the samples with the absence of the photocatalyst (green curve) is presumed to occur due to the reduction of soluble iron (Fe). This reduction is associated with the oxidation of Fe at the water's surface upon contact with air, leading to the formation of iron hydroxide, which subsequently precipitates. This precipitation causes the water to be slightly more transparent, leading to a minor decrease in the R% values.

The enhanced efficiency of the photocatalysis process under solar irradiation can be explained as follows: the semiconductor nanoparticles employed as catalysts in this study consist of anatase  $TiO_2$  with a band gap of 3.2 eV. In the production of  $TiO_2$ -PP, the thermal milling process exclusively coats  $TiO_2$  onto the surface of PP, preserving its original band gap value. Consequently, the photocatalysis process exhibits optimal efficiency when subjected to UV light with suitable energy.

Solar radiation emits UV light with a continuous spectrum spanning a broad range (3-30 eV) owing to thermal radiation from the sun's temperature of approximately 5,800 K. This characteristic makes sunlight an excellent source for the excitation of photocatalysts,

ensuring highly efficient outcomes. In contrast, UV light generated by lamps results from the electrical discharge within low-pressure mercury gas, producing a narrower and less continuous spectrum than sunlight. This disparity can lead to a mismatch between the energy levels of the lamp's UV spectrum and the band gap of TiO<sub>2</sub>, resulting in an inefficient excitation process.

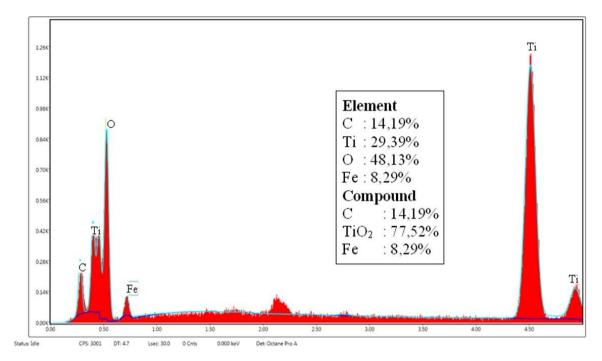


Figure 8. EDS Spectrum of TiO<sub>2</sub>-PP after the fifth application on peat water.

Spectrum changes in the TiO<sub>2</sub>-PP-treated samples subjected to 40 hours of sunlight irradiation are illustrated in Figure 7. This absorbance spectrum reveals a conspicuous blue shift and decreasing peak intensity as irradiation time progresses. The reduction in peak intensity signifies a decrease in the concentration of dissolved substances responsible for the discoloration of peat water. Notably, the spectrum closely matches the absorbance spectrum of humic acid as delineated in reference <sup>[19]</sup>, suggesting humic acid's susceptibility to degradation.

The decline in humic acid content also leads to a reduction in iron levels in the water. This phenomenon is explained by the fact that humic acid's capacity to interact with metals stems from its negatively charged functional groups, including -OH phenolic, -COOH, -O- (enolate), -O- (ether), and C=O (carbonyl)<sup>[20]</sup>. Building upon Hayes' conceptual framework<sup>[21]</sup>, the adsorptive action of humic compounds results in ionizing their polyelectrolyte surfaces, facilitating both the chemisorption and physisorption of iron<sup>[22]</sup>. As the photocatalysis process unfolds, the degradation of humic acid causes the liberation of previously bound iron, transforming it into free Fe ions amenable to adsorption on TiO<sub>2</sub><sup>[23]</sup>. EDS spectrum measurements on the surface of the TiO<sub>2</sub>-PP photocatalyst in Figure 8 validate this phenomenon. Notably, there is an 8.29% iron content in TiO<sub>2</sub>-PP, an increase that was present after initiating photocatalysis (Figure 4). Furthermore, a decrease in surface oxygen content on TiO<sub>2</sub>-PP is observed (compare Figure 4 with Figure 8), attributed to generating OH radicals through the interaction of oxygen with water.

The decrease in iron content in peat water following 40 hours of photocatalysis is evident when examining the Fe concentrations across different samples exposed to various treatments, as detailed in Table 2. The Fe concentration was measured using atomic absorption spectroscopy (AAS). Notably, the Fe content in peat water treated with  $TiO_2$ -PP and exposed to 40 hours of sunlight (rightmost column in Table 5) exhibited a substantial reduction, reaching 89.85% lower levels than the untreated reference peat water (leftmost column in Table 2).

Sample Type	peat water without the catalyst and irradiation	peat water + TiO <sub>2</sub> -PP + UV lamp (40 h)	peat water + TiO2-PP no irradiation	peat water + TiO <sub>2</sub> -PP + sunlight (40 h)
Fe Content	1,38	1,29	1,32	0,14
(mg/L)				

Table 2. Iron content in peat water for various treatments as measured by AAS.

The reliability of  $TiO_2$ -PP as a reusable photocatalyst was evaluated through its successive application in peat water, undergoing 40 hours of sunlight irradiation for five cycles. After each iteration, comprehensive characterizations were conducted on both the peat water samples and TiO<sub>2</sub>-PP to assess the efficacy of the photocatalysis process.

The performance of the photocatalysis process was examined by analyzing parameter values that reflect TiO<sub>2</sub>-PP's capability to reduce dissolved substances in peat water, denoted as  $K_f$ % (Equation 2). The absorbance value, denoted as A and required for computing this parameter, was measured using the method described in the previous section (A measurements for R% calculation). Table 3 presents the average A values over time for each successive use of TiO<sub>2</sub>-PP. Subsequently,  $K_f$ % values were calculated based on the number of reuse iterations, and the results are graphically depicted in Figure 9.

Usage number	Average value of A at every 8-hour interval (a.u.)						
	0 hour	8 hour	16 hour	24 hour	32 hour	40 hour	
1	2,89	1,637	1,217	1,130	0,154	0,154	
2	2,89	1,559	1,522	0,546	0,186	0,15	
3	2,89	1,645	1,272	0,630	0,154	0,156	
4	2,89	1,602	1,340	1,326	0,225	0,206	
5	2,89	1,644	1,586	1,400	0,916	0,465	

Table 3. Average absorbance, A, as a function of time for each iteration of TiO2-PP usage.

As shown in Table 3, after 40 hours, absorbance values remain relatively stable during the first four cycles of photocatalyst reuse. However, a notable increase in absorbance is observed during the fifth usage cycle. This trend is also prominently depicted in the  $K_f$ % versus the number of iterations graph (Figure 9). The photocatalytic performance of TiO<sub>2</sub>-PP exhibits consistency, evidenced by invariably high  $K_f$ % value, exceeding 92% throughout the initial four reuse cycles. In contrast, during the fifth cycle of operation, there is a significant decline in the  $K_f$ % value, reaching approximately 83%.

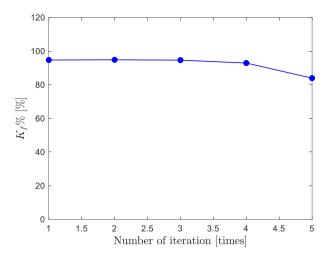


Figure 9. The photocatalyst's capability to decompose organic compounds in peat water for each number of iterations.

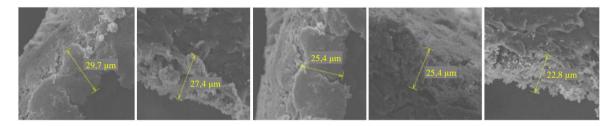


Figure 10. SEM cross-sectional images of TiO<sub>2</sub>-PP grains, taken at 1,000× magnification, for measuring film thickness across five iterations of photocatalyst usage: (a) initial (29.7 μm), (b) second (27.4 μm), (c) third (25.4 μm), (d) fourth (25.4 μm), and (e) fifth (22.8 μm).

The decline in the photocatalytic efficiency of  $TiO_2$ -PP is likely ascribed to several factors. Firstly, there is a noticeable thinning of the  $TiO_2$  layer as particles detach from the PP surface. This phenomenon is discernible in the SEM cross-sectional images of  $TiO_2$ -PP grains in each reuse cycle [Figure 10(a)–(e)]. During photocatalysis, UV excitation renders the originally nonpolar  $TiO_2$  polar. Consequently, some loosely coated  $TiO_2$  particles on the PP surface detach due to the attractive forces with polar water molecules. These detached  $TiO_2$  particles settle at the bottom and receive less light exposure than those near the water surface, reducing photocatalytic effectiveness. Another contributing factor to the declining performance of  $TiO_2$ -PP photocatalysis could be the adsorption of substances present in the water, such as Fe, onto the  $TiO_2$  surface. This interaction can impede the optimal excitation of UV light, compromising the photocatalytic process's efficiency.

#### CONCLUSIONS

We investigate the efficacy of TiO<sub>2</sub>-PP photocatalyst in treating peat water. SEM and EDS analyses were conducted to characterize the catalyst, and UV-Vis spectrophotometry was employed to analyze peat water. The fabrication process employed thermal milling to coat TiO<sub>2</sub> particles onto the surface of the PP. SEM images and EDS spectra analysis confirmed the uniform and successful coating of TiO<sub>2</sub> particles on the PP surface. The application of TiO<sub>2</sub>-PP in peat water under sunlight irradiation demonstrated a notably effective photocatalytic performance, surpassing that observed with UV lamp irradiation. This can be attributed to the broader and more continuous wavelength spectrum of sunlight UV, which contrasts with the narrower UV lamp spectrum. AAS measurements on the samples

underscored the remarkable effectiveness of the TiO<sub>2</sub>-PP photocatalytic process in reducing iron (Fe) concentrations in peat water by 89.85%.

In applying TiO<sub>2</sub>-PP as a reusable photocatalyst, it consistently maintained performance levels exceeding 92% through the first four uses. However, in the fifth application, its efficiency declined to 83%. The decline in performance is attributed to the gradual thinning of the TiO2 layer, leading to particle detachment from the PP surface. Additionally, the adsorption of Fe onto TiO<sub>2</sub> during the catalytic process may have contributed to the hindrance of UV light excitation. These findings unveil the significant potential of TiO2-PP as a floating and reusable photocatalyst, offering an effective solution for reducing humic acid and iron content in peat water.

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