

Deposition of Activated Carbon/TiO₂ on Polystyrene and Its Performance in Photodegradation of Methylene Blue

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Abstract. The composite material consisting of activated carbon (AC) and titanium dioxide (TiO₂) photocatalysts can be employed for degradation dye in water. Nevertheless, the dispersed-composite of AC/TiO₂ in water needs additional treatment to obtain clean water, which is considered less effective. This study aims to deposit AC/TiO₂ on polystyrene (PS) polymers with various concentrations of TiO₂ and study its ability to degrade methylene blue (MB). The synthesis of AC from orange peel waste involved carbonization at 600°C and chemical activation with 10% (w/v) ZnCl₂ solution. The AC was subsequently combined with TiO₂ using a straightforward mixing process lasting 30 minutes, with variations of TiO₂ are 10%, 15%, 20%, and 25%. AC/TiO₂ composite was coated to PS granules using the thermal milling method at 250°C for 1 hour. The scanning electron microscopy (SEM) analysis of the AC/TiO₂ composite shows the distribution of TiO₂ particles attached to the AC. UV-Vis characterization was carried out as a quantitative parameter to measure MB concentrations during photocatalytic activity. To see the performance of the PS-AC/TiO₂ composite, Pseudo-first-order kinetic was used as a photocatalytic activity model. The result shows that a concentration of 15% has the highest MB degradation for 8 hours of irradiation with a reaction rate constant of 0.22712 h⁻¹ and effectiveness of 83.75%. Hence, a film of AC/TiO₂ on PS granules is promising in photodegrading MB dye.

Keywords: activated carbon, TiO₂, polystyrene, thermal milling

Abstrak. Material komposit yang terdiri dari fotokatalis karbon aktif (AC) dan titanium dioksida (TiO₂) dapat digunakan untuk degradasi zat warna di air. Namun demikian, AC/TiO₂ komposit yang terdispersi di dalam air memerlukan pengolahan tambahan untuk mendapatkan air bersih, yang dinilai kurang efektif. Penelitian ini bertujuan untuk mendeposisikan AC/TiO₂ pada polimer polistiren (PS) dengan berbagai konsentrasi TiO₂ dan mempelajari kemampuannya dalam mendegradasi metilen biru (MB). Sintesis AC dari limbah kulit jeruk dilakukan melalui karbonisasi pada suhu 600°C dan aktivasi kimia dengan larutan ZnCl₂ 10% (b/v). AC selanjutnya digabungkan dengan TiO₂ melalui proses pencampuran langsung selama 30 menit, dengan variasi TiO₂ 10%, 15%, 20%, dan 25%. Komposit AC/TiO₂ dilapisi pada butiran PS menggunakan metode *thermal milling* pada suhu 250°C selama 1 jam. Analisis scanning electron microscopy (SEM) pada komposit AC/TiO₂ menunjukkan sebaran partikel TiO₂ yang menempel pada AC. Karakterisasi UV-Vis dilakukan sebagai parameter kuantitatif untuk mengukur konsentrasi MB selama aktivitas fotokatalitik. Untuk melihat kinerja komposit PS-AC/TiO₂ digunakan kinetika semu orde pertama sebagai model aktivitas fotokatalitik. Hasil penelitian menunjukkan bahwa konsentrasi 15% mempunyai degradasi MB tertinggi pada penyinaran 8 jam dengan konstanta laju reaksi sebesar 0,22712 jam⁻¹ dan efektivitas sebesar 83,75%. Oleh karena itu, film AC/TiO₂ pada butiran PS menjanjikan dalam fotodegradasi pewarna MB.

Kata kunci: karbon aktif, TiO₂, polisterina, *thermal milling*

INTRODUCTION

The textile industry uses synthetic dyes that can affect the environment and humans. Methylene Blue (MB) is a toxic, carcinogenic, and non-biodegradable dye often used in the textile industry [1]. The presence of this substance in water bodies must be eradicated due to its significant risk to human health and detrimental environmental effects. The adsorption technique using activated carbon (AC) is extensively used for the removal of MB [2]–[4], and it has some advantages, such as high specific surface area, excellent adsorbent, and versatility [5]. Another advantage is that AC can be easily synthesized from biomass containing lignocellulose, such as orange peel [6]. Orange peel contains lignocellulose, which is 69.1% cellulose, 5.4% hemicellulose, and 19.8% lignin [7]. Lignocellulosic in biomass is necessary for AC synthesis to convert its structure into carbon through high-temperature decomposition [8].

A single AC is inadequate as a wastewater treatment agent due to its limited capacity for adsorbing MB dye. To enhance the efficiency of the adsorbent, the adsorption technique was integrated with photocatalyst techniques [9]. Combining AC with metal oxide semiconductors, e.g., TiO₂ [10], ZnO [11], and Fe₃O₄ [12] have been studied. Among these semiconductors, TiO₂ in the anatase structure has a band gap of 3.23 eV [13], which is compatible with sunlight. It also has other advantages, such as inexpensive and photostable [14]. Nevertheless, the dispersion of TiO₂ in water leads to the formation of cloudy water after treatment due to its pigmentation, thereby reducing its efficacy in water treatment. To address this issue, it is necessary to modify the material by immobilizing a substance onto a polymer through a thermal milling method at heat-deflecting temperature (HDT) [15].

Polystyrene (PS) is a thermoplastic polymer that can be easily molded and allows for the attachment of other materials onto its surface. Previous research has been carried out regarding the surface immobilization of polystyrene with AC [16] and TiO₂ [17]. Exploring the activity of these two materials on PS surfaces is an exciting research endeavour. This material has the potential to be developed as a photocatalyst, offering the advantage of not requiring filtration at the end of the water treatment.

This study reports the photocatalytic activity of PS-AC/TiO₂. The AC derived from the orange peel will be combined with TiO₂ using a straightforward mixing technique. Then, AC/TiO₂ is coated on the surface of PS grains using the thermal milling method. We then analyze PS-AC/TiO₂ using scanning electron microscopy (SEM) and assess its effectiveness in the photodegradation of MB.

METHOD

Tools and Materials

The tools used in this study were a 100 mesh sieve, furnace (Carbolite; England), grinder, filter paper, mixer, oven (Cosmos; Indonesia), pH meter, thermal milling cylinder, UV-Vis spectrophotometer (Shimadzu UV-2600; Jepang), and scales. The materials used were distilled water, Siamese orange peel, high-impact polystyrene (HIPS) granules (100% purity; Trinseo; Indonesia), TiO₂ with anatase structure (technical grade from Ex Korea TK 4534), and ZnCl₂ (98% purity; Merck; Germany).

Synthesis Process

The AC synthesis process was followed in our previous study [18]. After cleaning and washing the orange peel, the orange

peels are dried under sunlight and carbonized to create the charcoal using a furnace at 600°C for 1 hour of holding time [19]. Charcoals are grounded and then sieved to 100 mesh. ZnCl₂ with a concentration of 10% (w/v) is used to activate the charcoal with the mass ratio of charcoal, and ZnCl₂ was 1:2 by weight per volume. After 24 hours, the AC is washed using distilled water until neutral (pH=7) and dried using an oven at 150°C for 3 hours [20]. We used **Equation 1** [21] to count the yield of AC.

$$\text{Yield (\%)} = \frac{m_1}{m_0} \times 100 \quad (1)$$

where m_1 is the dry mass of AC (g), and m_0 is the dry mass of the orange peel (g)

To make the composite, AC was mixed with TiO₂ with 10, 15, 20, and 25% variations using a mixer for 30 minutes. Then, AC/TiO₂ was coated on the PS surface using a thermal milling method at 250°C for 60 minutes. The mass ratio of AC/TiO₂ and PS was 1:4, respectively. After thermal milling, PS-AC/TiO₂ was rinsed with distilled water to remove the unattached AC/TiO₂ and dried at 100°C for 30 minutes.

Experimental Procedure

The experiment was conducted at FMIPA Tanjungpura University, located at coordinates 0°03'29" S 109°20'43" E. The PS-AC/TiO₂ was examined for its performance in degrading MB. Firstly, MB solution as artificial wastewater is prepared with a concentration of 10 ppm. Then, 15 grams of PS-AC/TiO₂ is added to 500 mL of artificial wastewater. The photodegradation process under sunlight starts from 08.00-16.00 WIB (8 hours), with sampling for measurement every 2 hours. Subsequently, samples are analyzed using a UV-Vis spectrophotometer with a maximum wavelength of 663 nm. Evaluating PS-

AC/TiO₂ effectiveness (E) in the degradation of MB is used in **Equation 2** [22].

$$E (\%) = \left(\frac{A_0 - A_t}{A_0} \right) \times 100 \quad (2)$$

A_t is the maximum absorbance during irradiation, and A_0 is the initial maximum absorbance.

Pseudo-First Order Kinetics Model

The MB degradation rate of the adsorption-photocatalytic process can be modeled using pseudo-first-order kinetics [23], as seen in **Equation 3**.

$$\frac{C_0}{C_t} = e^{-K_{ap}t} \quad (3)$$

The concentration value is directly proportional to the absorbance value, so the absorbance value represents the concentration value. C_0 and C_t are at $t = 0$ and t time, respectively. K_{ap} is the degradation rate constant, and t is the time during the adsorption-photocatalytic process.

RESULT AND DISCUSSION

Synthesis and Characteristic Material

Synthesizing AC from lignocellulose biomass involves some processes, i.e., preparation, carbonization, and activation. Carbonization is crucial because carbon compounds increase, resulting in lignocellulose decomposition in orange peel. The average mass of charcoal produced in this study was 23.45%, indicating lost mass. This is common in the carbonization step because the orange peel is dehydrated from water and releases volatile matter from the cellulose structure [24].

Using the ZnCl₂ compound as an activator has decreased AC mass by as much as 20.64%. This result suits with the theory of AC production from lignocellulose

material decreasing by 20-30% [25]. This yield decreased by 2.81% from the carbonization process because of the impurities dissolved in ZnCl₂.

The AC/TiO₂ deposited on the PS using the thermal milling method was successfully synthesized, covering the PS surface in a film that maintains the PS shape, as seen in **Figure 1**. When the concentration of TiO₂ is raised, the color of PS-AC/TiO₂ becomes greyish (**Figure 2**). This is due to the white color of TiO₂, which affects the composite color as a whole.

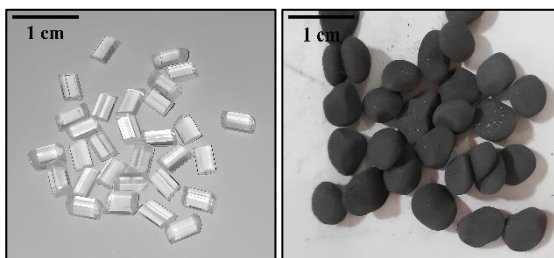


Figure 1. Images of PS before coated (left) and PS after coated (right)

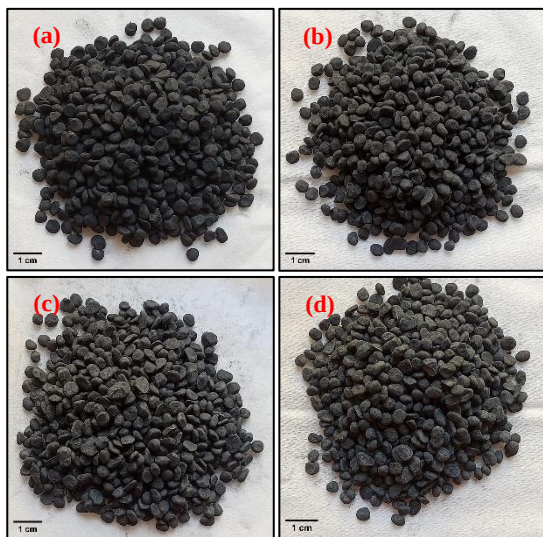


Figure 2. PS-AC/TiO₂ with varied of TiO₂ (a) 10%, (b) 15%, (c) 20%, and (d) 25%

Results of AC/TiO₂ immobilization on PS with different TiO₂ concentrations are shown in **Table 1**. Immobilization is interpreted as the amount of AC/TiO₂

attached to the surface of PS granules. According to **Table 1**, the immobilization value increases with increasing TiO₂ concentration. The 25% concentration exhibited the greatest immobilization value compared to the other concentrations. The addition of TiO₂ particles to the AC increases small-sized particles, as TiO₂ particles are smaller than activated carbon (**Figure 3**). Smaller particles tend to adhere more to the polystyrene surface due to their increased surface area.

Table 1. Deposition of AC/TiO₂ on PS

No	TiO ₂ concentration (%)	Immobilization (gram)
1	10	0.4
2	15	0.3
3	20	0.6
4	25	0.8

SEM Analysis

SEM images in **Figure 3** show the distribution of TiO₂ particles on the surface of AC. Using a simple mixing method to produce an AC/TiO₂ composite does not provide a homogenous dispersion of TiO₂. There are vacancy areas (uncoated by TiO₂) on the surface of AC, and on the other side, the distribution of TiO₂ particles tends to overlap. **Figure 3(a)** shows the concentration of 10%, the distribution of TiO₂ is not visible and difficult to identify because the given mass is too small. In higher concentrations of 15, 20, and 25%, TiO₂ can be identified, and its distribution on the AC surface is shown in **Figure 3 (b)-(d)**, respectively.

We also analyzed the film of PS-AC and PS-AC/TiO₂ of 15% to see the effect of adding TiO₂ semiconductor on AC. We only decided to characterize 15% composition because it has good photocatalytic activity in the breakdown of MB, as we will describe later in this article. **Figure 4(b)** shows the color of the PS-AC/TiO₂ composite turning greyish compared with PS-AC (**Figure**

4(a)), which is pitch black. A greyish color appeared in **Figure 4(b)** due to adding 15% of TiO_2 . The SEM images are consistent with **Figure 2**, which shows the addition of TiO_2 , making composites turn grey due to the white pigment of TiO_2 . Through SEM

analysis, the AC/ TiO_2 film on PS grains can be observed in detail, showing a homogeneous distribution of TiO_2 attached to the AC surface, resulting in a surface enriched with photocatalytic activity.

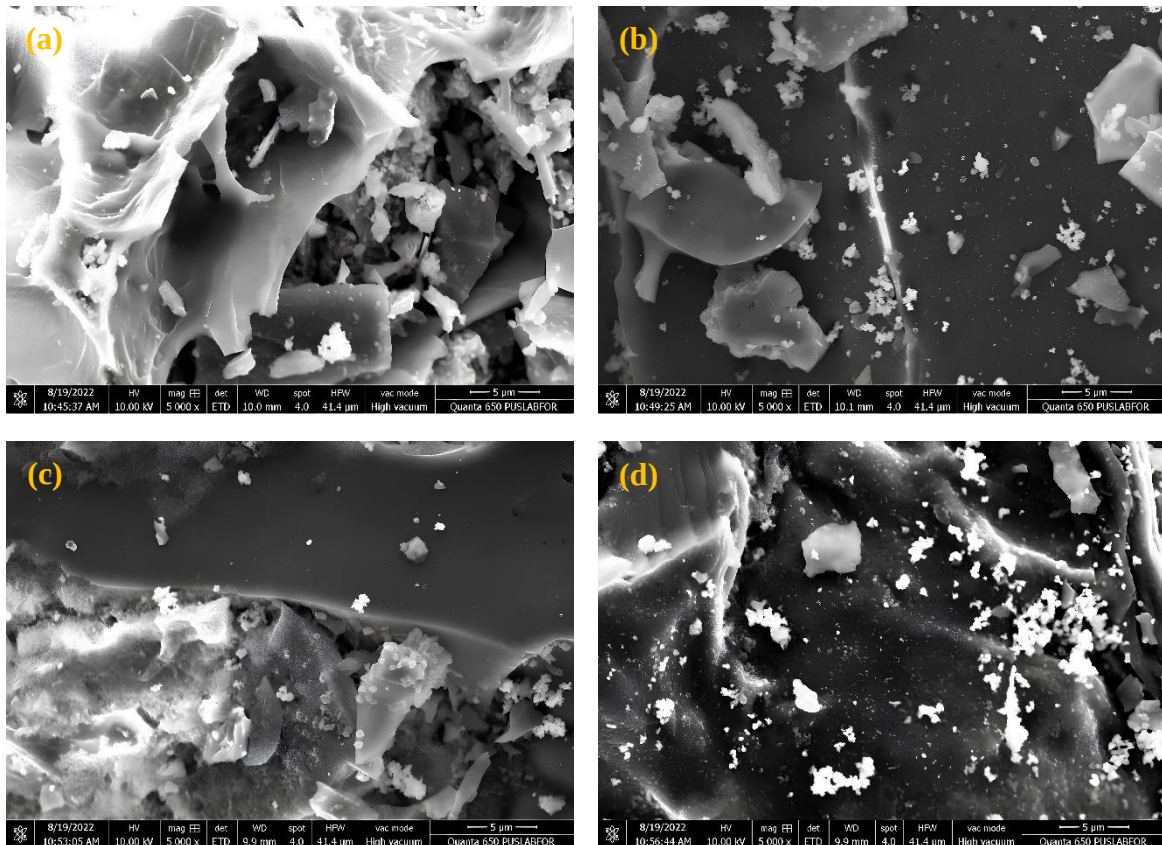


Figure 3. Micrograph of AC/ TiO_2 : (a) 10%, (b) 15%, (c) 20%, and (d) 25%

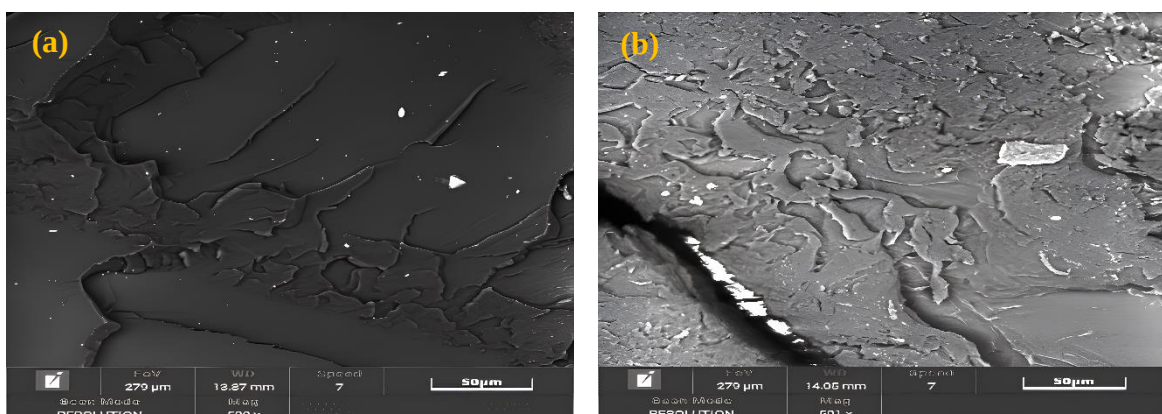


Figure 4. The cross-sectional area of the film (a) PS-AC, (b) PS-AC/ TiO_2 (15%)

Adsorption-Photocatalytic Test

Figure 5(a) presents a comparison of the PS-AC/TiO₂ model in MB degradation based on TiO₂ concentration. The most significant decrease occurred at a concentration of 15%, demonstrating notable efficacy within the initial 4-hour period. At the beginning of the process, the fast adsorption of MB dye in the early stages (0-4 hours) is caused by the availability of open surfaces and active sites on the AC surface [26]. The photocatalytic activity of TiO₂ also plays an important role in producing a significant decrease in the first 4 hours due to the high intensity of sunlight at that time (12 pm), which is the highest intensity of sunlight ranging from 11 to 2 pm [27]. Despite the optimal performance of the 15% PS-AC/TiO₂, an anomaly occurred during the 4-6 hours period (12-2 pm), and MB concentration unexpectedly increased by 46.21%. We suspect that the anomaly might be due to the desorption of MB from the AC. Additionally, in MB treatment, AC not only has adsorption capability but also

has a desorption capability [28]. Releasing back of adsorbate (MB in this study) from AC pores into water happen as a result of thermal desorption [29], leading to a more significant amount of MB dye discharged into the water [30]. High temperatures provide sufficient energy for MB molecules that have been adsorbed on AC to overcome the attractive forces between AC and MB, allowing MB to be released back into the surrounding environment. Therefore, the desorption process in this investigation was influenced by high environmental temperatures between 12-2 pm. Moreover, the temperature on the experiment day reached 33°C, higher than the usual environmental temperature of 29°C. This anomaly is also confirmed at PS-AC, as seen in **Figure 5(b)**. No significant change exists in the 4-6 hour period (12 to 2 pm) because AC adsorption capability decrease in high temperatures [30]. It is more convincing that the AC in the 15% PS/AC-TiO₂ composite underwent desorption due to the high temperature.

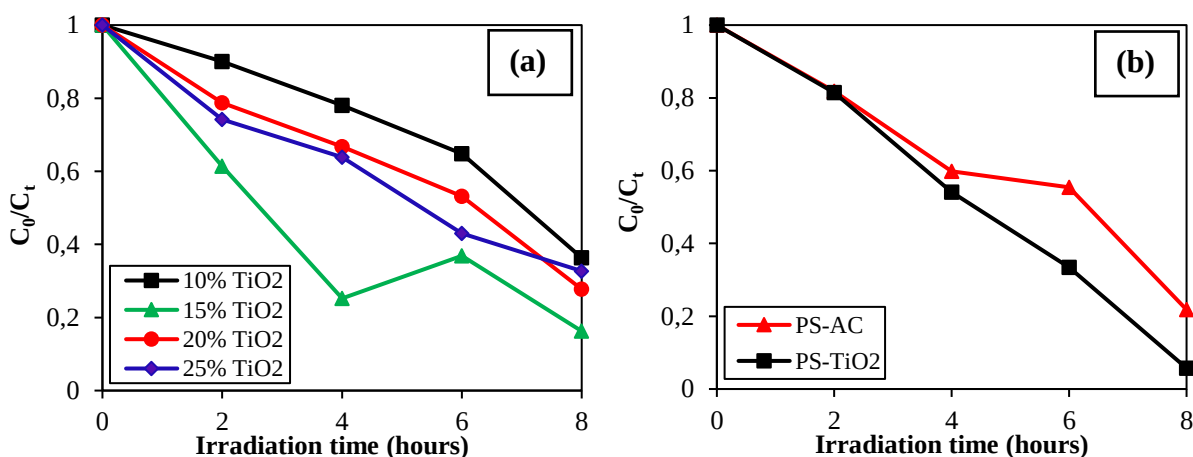


Figure 5. MB photodegradation kinetics after 8 hours irradiation (a) PS-AC/TiO₂ and (b) PS-AC vs PS-TiO₂

Unlike AC has limited to adsorbing and releasing MB, the photocatalytic process (black line) in **Figure 5(b)** is stable in degrading MB dyes. Therefore,

TiO₂ photocatalyst significantly improved the AC properties in this study. During irradiation, TiO₂ particles interacted with sunlight ($h\nu$) to produce electrons (e) and

holes (h^+). H_2O or O_2 trapped the holes on the surface of the TiO_2 particle, resulting in H^+ and HO radicals. HO attacked the MB molecule by a hydroxyl addition or hydrogen extraction effect, and MB was converted to CO_2 and H_2O via several pathways [22].

Performance of PS-AC/ TiO_2

The final results of the adsorption-photocatalytic process after 8 hours of irradiation are shown in **Figure 6**. The injection of PS-AC/ TiO_2 composite can reduce MB dye, as shown in **Figure 6 (a)**, which shows both qualitative and quantitative parameters. After 8 hours of treatment, the absorbance values of the four treated samples (brown bars) decreased relative to their pre-treatment value (blue bars). By the quantitative data, **Figure 6(a)** also shows the photo of MB

as qualitative parameters, which indicates the colour shift from untreated (IC) to treated following treatment with PS-AC/ TiO_2 with TiO_2 concentration variation. To evaluate the performance of the PS-AC/ TiO_2 composite, a pseudo-first-order kinetic model (**Equation 3**) was used to determine the best TiO_2 concentration in MB breakdown. K_{Ap} is a reaction rate constant that is directly proportional to the efficiency of AC/ TiO_2 in degrading MB [31]. The K_{Ap} for the four variations were calculated with values of 0.12657, 0.22712, 0.16036, and 0.13974 h^{-1} obtained from the AC/ TiO_2 composites with TiO_2 concentration of 10, 15, 20, and 25%, respectively, as shown in **Figure 6 (b)**. The 15% concentration has the highest K_{Ap} value. Therefore, it is the most optimal parameter in this research, with an efficiency of 83.75%.

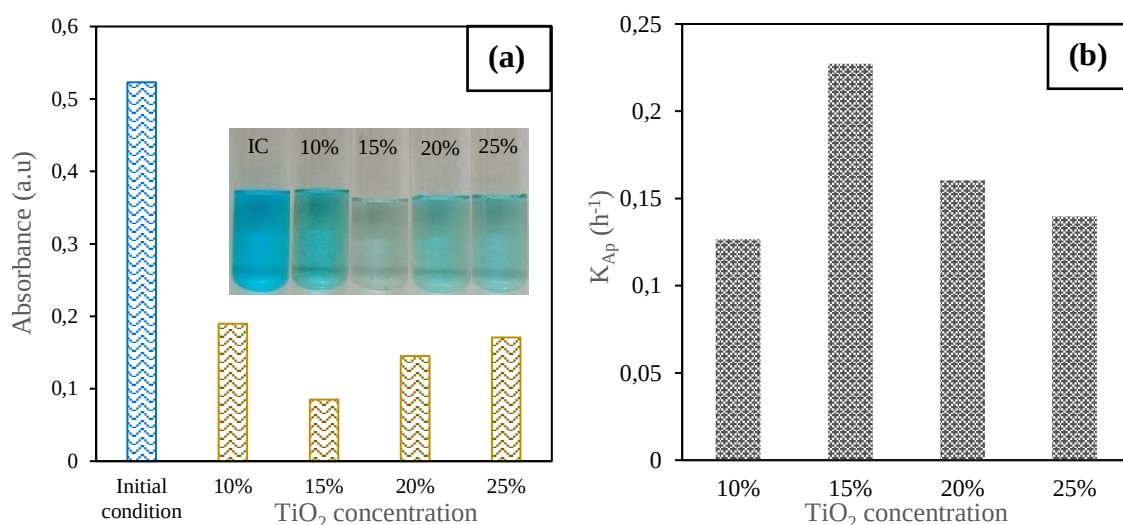


Figure 6. Performance of PS-AC/ TiO_2 based on absorbance value (left) and degradation rate constant (K_{Ap}) (right axis)

CONCLUSION

A film of AC from orange peel and TiO_2 composite deposited on the surface of PS grains has been successfully synthesized using the thermal milling

method and investigated on photodegradation of MB. Adding TiO_2 to AC can improve the performance of AC, which is less stable in MB photodegradation. After 8 hours of irradiation, based on variations in TiO_2 ,

the best concentration was obtained at 15% with a reaction rate constant of 0.22712 h⁻¹ and effectiveness of 83.75%. With high effectiveness results, the PS-AC/TiO₂ composite is promising to be used as an adsorbent-photocatalyst material to degrade MB without needing post-treatment.

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