MOLEKUL
eISSN: 2503-0310

Articles
https://doi.org/10.20884/1.jm.2025.20.1.13714

Synthesis of TiO_2-Activated Carbon from Coffee Dregs by Hydrothermal Method for Photodegradation of Diazinon

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Received November 22, 2024; **Accepted** February 20, 2025; **Available online** March 20, 2025

ABSTRACT. The photocatalytic activity of TiO_2 can be enhanced by binding it to materials with high adsorption capacity. The synthesis began with the mixture of 5 mL TTIP, 30 mL ethanol, and H_2O 30 mL stirred for 1 hour. Activated carbon made from coffee grounds in varying amounts (5 g, 10 g, and 15 g) was then added to the TTIP solution, and the mixture was stirred for 2 hours. The mixture was then placed in an autoclave, heated at 180°C for 12 hours, dried, and then calcined at 500°C for 3 hours. SEM morphological analysis showed that TiO_2 particles were attached to the surface of the activated carbon, which was further confirmed by EDX data showing the presence of Ti and O elements in the synthesized material. Furthermore, crystallinity and gap analyses demonstrated that the material was exclusively in the anatase phase. The largest surface area, 286.10 m^2/g, was observed on titanium dioxide-activated carbon/10 (TiO_2-AC/10), exhibiting a mesoporous structure and microporous features. Photocatalytic tests for diazinon degradation demonstrated that TiO_2-AC/10 exhibited the most significant photocatalytic activity of 65.18%. The composite material's degradation capability diminished by 9-13% from the initial to the third cycle. The residual titanium elements within the composite material maintained stability, suggesting the integrity and durability of TiO_2 particles affixed to the surface of activated carbon.

Keywords: TiO_2-AC, Coffee Dregs, Diazinon, Photocatalysis, Reusability

INTRODUCTION

Titanium dioxide (TiO_2) is a semiconductor that functions as a photocatalyst material, absorbing energy from ultraviolet (UV) light. This material can be applied in various industries, including the paint, plastic, paper, pharmaceutical, and textile industries (Sheilatina, *et al.*, 2020). The TiO_2 crystal structure phase comprises brookite, anatase, and rutile phases, with band gap values of approximately 3.0–3.2 eV and notable activity in UV light. The anatase phase exhibits superior photocatalytic activity compared to the brookite or rutile phase. The advantages of anatase TiO_2 include high thermal stability and effective UV light absorption, which enhances photocatalytic activity (Sutisna et al., 2023).

TiO_2 can be obtained commercially or synthesized from TiO_2 precursors, one of which is *titanium tetraisopropoxide* (TTIP). Synthesis of TiO_2 from precursors has the advantage of better control of the size of TiO_2 (Ribeiro *et al.,* 2020). Zeng et al. (2021) reported that TiO_2 from TTIP precursors in the anatase phase has a smaller band gap value than commercial TiO_2 P25. The band gap value of TiO_2 from TTIP is 3.07 eV, while commercial TiO_2 P25 is 3.21 eV. TiO_2 synthesis is carried out using a hydrothermal method, which has the advantages of good crystallinity, uniform particle size, and good size distribution. The hydrothermal method is crystal growth due to the heating of the material by water vapor that occurs in an autoclave (Sheilatina *et al.*, 2020).

TiO_2 exhibits a deficiency in low adsorption power and photocatalytic activity. This can be rectified by incorporating activated carbon as an adsorbent to create composite materials (Barakat et al., 2023). The addition of activated carbon increases the composite material's surface area and adsorption efficiency while simultaneously decreasing the band gap energy. This is evidenced by the findings of Zeng et al. (2021), which demonstrated that the TiO_2-AC composite exhibited a band gap of 2,32 eV, compared to the 3.0-3,3 eV observed in commercial TiO_2. Sutisna et al. (2023) successfully synthesized TiO_2-AC with a mass variation of 5-20 g activated carbon, and the optimum degradation occurred at 10 g activated carbon, resulting in a 95% decrease in methylene blue within 60 minutes. Coffee dregs, which contain high levels of carbon and cellulose, have been identified as a promising source of activated carbon for composite

support materials (Sukhbaatar et al., 2021; Oko et al., 2021).

Activated carbon can be produced by adding activators such as hydrochloric acid (HCl) which increases absorbency by reducing water content and removing metal impurities (Puspaningrum *et al.*, 2023). Oko et al. (2021) showed that the temperature and concentration of 1M HCl affect the characteristics of activated carbon, with an ash content of 2.15% and a moisture content of 1.49% at 400°C. TiO_2-AC composites are widely applied in photodegradation with various organic environmental pollutants that are difficult to decompose, including diazinon. Diazinon is an organic pollutant often used as an insecticide and is classified as a hazardous compound because it is in class II by WHO (*World Health Organization*) (Khoiriah *et al.*, 2020). TiO_2-AC photocatalyst from coconut shells successfully reduced diazinon by 67.13% after 8 hours of UV irradiation (Salsabillah et al., 2022). Based on this study, one-step synthesis of TiO_2-AC from coffee grounds with variations in activated carbon mass using the hydrothermal method will be carried out and characterized using XRF, XRD, UV-Vis DRS, BET, and SEM-EDX to evaluate its photocatalytic activity in diazinon photodegradation.

EXPERIMENTAL SECTION

Synthesis of Activated Carbon

The synthesis of activated carbon is based on the research of Deswardani et al. (2022), with minor modifications. The coffee grounds were washed with distilled water until the pH was neutralized, after which they were dried. The coffee grounds were converted into carbon using a furnace at 400°C for 90 minutes, allowing them to cool. Subsequently, the carbon was sieved until it reached a size of approximately 150-200 mesh. The carbon was activated by immersing it in a 1M HCl solution (100 g in 100 mL) for 48 hours. The solution was decanted, and the carbon was washed with distilled water until the pH reached a neutral level. It was then dried. Subsequently, the resulting activated carbon samples were evaluated for moisture and ash content to ascertain their quality. The moisture and ash content were determined by weighing 1 gram of activated carbon. The carbon was subjected to a moisture content test in an oven at 105°C for 2 hours, while the ash content test was conducted by heating in a furnace at 650°C for 4 hours.

Synthesis TiO_2-Activated Carbon (TiO_2-AC)

TiO_2-AC synthesis was carried out by hydrothermal method based on Sutisna et al. (2023) through mixing 5 mL TTIP, 30 mL ethanol, and 30 mL distilled water, stirred for 1 hour, then added activated carbon (mass variations of 5, 10, and 15 g) and stirred for 2 hours. The mixture was then subjected to autoclaving at 180°C for 12 hours. Following this, the mixture was washed to a neutral pH, dried, and reheated at 500°C for 3 hours. TiO_2 synthesis was carried out similarly, except for using activated carbon.

Characterization of TiO_2-AC

The resulting TiO_2 and TiO_2-AC materials were characterized using scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDX), X-ray diffraction (XRD), ultraviolet-visible diffuse reflectance spectroscopy (UV-Vis DRS), X-ray fluorescence (XRF), and surface area analysis (SAA). The characterization aimed to determine the morphology of the particle surface, the crystal phase, the band gap value, the elemental analysis, and the surface area of the material. All characterization data of TiO_2-AC materials were compared for each mass variation of activated carbon.

Photocatalytic Test

The TiO_2-AC photocatalytic test was conducted following the methodology proposed by Salsabillah et al. (2022) for the photodegradation of a 30 ppm diazinon solution. A total of 1 gram of TiO_2-AC was added to 200 mL of a diazinon solution, which was then irradiated using 5 UV lamps (10 watts) in a chamber and stirred at 200 rpm. The experiment was conducted over seven hours, with samples collected at one-hour intervals (up to 4 mL) and allowed to stand for 24 hours to precipitate the material. The photocatalysis tests were conducted with the following variations: diazinon with UV light and TiO_2-AC, diazinon with TiO_2-AC without UV, and diazinon without UV and TiO_2-AC. The concentration of diazinon was determined via ultraviolet-visible (UV-Vis) spectrophotometry and plotted as a function of time versus absorbance. Following irradiation, the material was washed, dried, and subjected to X-ray fluorescence (XRF) analysis to quantify the residual titanium content. This procedure was repeated three times to assess the material's reusability.

RESULTS AND DISCUSSION

Characterization of TiO_2-AC

The synthesis process of the TiO_2-AC composite from coffee grounds commences with a carbonization process, followed by activation. The activated carbon was then tested for moisture and ash content, which were found to be 1.59% and 2.2%, respectively. These data comply with the SNI 06-3730-1995 standard, which stipulates that the maximum moisture content for powder material is 15% and the maximum ash content is 10%. Deswardani et al. (2022) found that activated carbon from coffee grounds of 1173.8 mg/g meets the SNI standard of at least 750 mg/g. The next process is the synthesis of TiO_2-AC, which is carried out using the hydrothermal method. TTIP, ethanol, and distilled water as TiO_2 precursors are made constant. It is hypothesized that variations in the mass of activated carbon added will increase the adsorption power of the composite material. The synthesis process produced four samples of material

variants, namely TiO_2, TiO_2-AC/5, TiO_2-AC/10, and TiO_2-AC/15.

Characterization XRD

The crystal phase of a material can be identified using XRD characterization, with the anatase structure of TiO_2 being identified through the careful observation of the resulting diffraction pattern, particularly at the 2θ angle positions of 25.3°, 37.8°, 48.0°, 54.0°, and 55.1°. These peaks indicate an anatase structure and differ from those observed in other TiO_2 structures. The intensity of the diffraction peaks, particularly at 25.3°, which is typically the strongest, further corroborates the anatase structure. The anatase lattice parameters are a=b=3.784 Å and c=9.514 Å, suggesting a tetragonal crystal structure. The XRD characterization of the four synthesized TiO_2 materials exhibited a diffraction pattern consistent with the JCPDS No. 21-1272 data of anatase phase TiO_2 material. The integration of these data led to the confirmation of the anatase structure on TiO_2. The diffraction angle peaks of the TiO_2 material and the reference are depicted in **Figure 1**. Notably, the activated carbon possesses an amorphous structure, thereby precluding the observation of diffraction patterns characteristic of activated carbon in the composite material with TiO_2 (Riyanto et al., 2020).

Crystallite size is defined as the size of a small part of the crystal in the material that can affect its properties (Jones, 2019). The crystallite of the TiO_2 material and the TiO_2-AC composite material was calculated at an angle of $2\theta = 25°$. Due to its high intensity compared to other peaks, this peak is also often used to estimate crystallite using the Scherrer equation, which relates the width of the diffraction peak to crystallite (Smith et al., 2022). **Table 1** provides a comprehensive overview of the crystallite sizes for the individual TiO_2 materials and the synthesized TiO_2-AC composite material. The composite material crystallites are sorted from smallest to largest as follows: TiO_2-AC/10, TiO_2-AC/15, TiO_2, and TiO_2-AC/5, with sizes of 10.53 nm, 19.29 nm, 21.06 nm, and 23.18 nm, respectively. This observation indicates that alterations in the incorporation of activated carbon can influence the crystallite structure of the composite material. The irregular crystallite size can be attributed to the uneven distribution of TiO_2 on the surface of activated carbon, a finding corroborated by SEM EDX characterization.

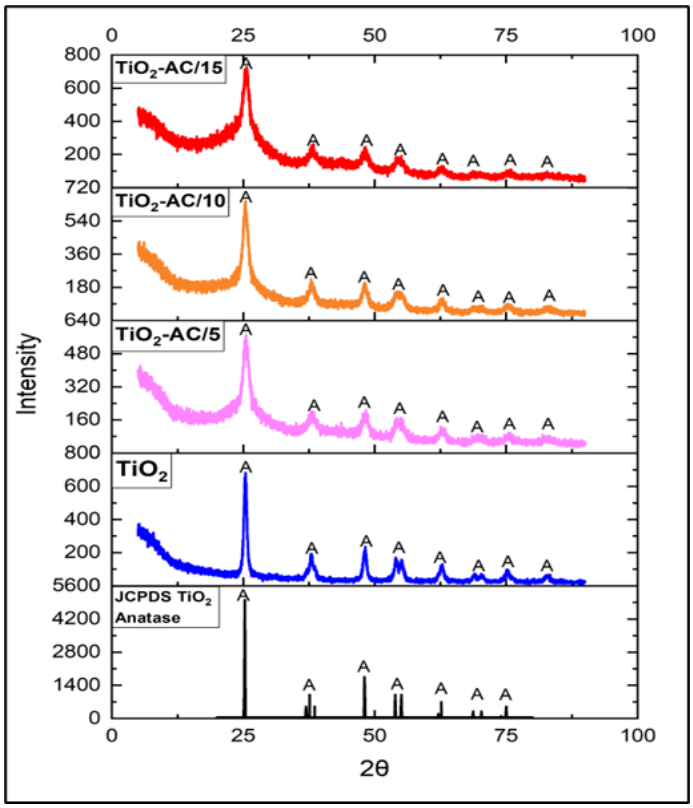

Figure 1. Composite material difractogram (A= Anatase)

Table 1. The crystal size of composite materials

Materials	Crystallite size (nm)
TiO_2	21.06
TiO_2-AC/5	23.18
TiO_2-AC/10	10.53
TiO_2-AC/15	19.29

Characterization SEM-EDX

The synthesized TiO_2 and TiO_2-AC were characterized by scanning electron microscopy (SEM) to obtain information regarding the morphology and particle size of the composite material. The ImageJ software was employed to identify the synthesized material's particle size and determine the particle size. **Figure 2** illustrates the SEM results of the synthesized material at a magnification of 20,000, which exhibits a non-uniform, round surface. The composite material demonstrates that the TiO_2 particles have attached to the surface of the activated carbon, displaying an uneven distribution. Consequently, the surface morphology of the material is predominantly characterized by activated carbon.

The mean particle size of TiO_2 was found to be 211.54 nm. The TiO_2 samples labeled TiO_2-AC/5, TiO_2-AC/10, and TiO_2-AC/15 exhibited mean particle sizes of 199.78 nm, 191.09 nm, and 164.74 nm, respectively. The measurements were conducted using the ImageJ software with 50 points. The particle size is larger than the crystallite size since particles typically comprise multiple smaller crystals that coalesce to form a larger entity. Each crystal is characterized by an ordered atomic structure and is separated by crystal boundaries that serve as a physical barrier between one crystal grain and another. The crystal grains are minute and numerous, collectively forming particles of a larger size. Consequently, particle size represents the aggregate of multiple crystals, whereas crystallite refers to a single grain within a particle. Figure 2 illustrates that TiO_2 has not adhered homogeneously to the surface of activated carbon, as evidenced by empty spaces on the activated carbon surface that have not been coated with TiO_2.

Following the synthesis of the material, an additional analysis was conducted utilizing EDX (Energy Dispersive X-Ray). **Table 2** demonstrates that the sample is composed of three elemental constituents: carbon (C), oxygen (O), and titanium (Ti). The elements are derived from the composition of the constituent materials and the synthesis process. The discrepancy in composition can be attributed to fluctuations in the quantity of activated carbon incorporated into the composite material. The synthesis results are deemed successful, as evidenced by the observed increase in the percentage of carbon, accompanied by variations in the addition of activated carbon.

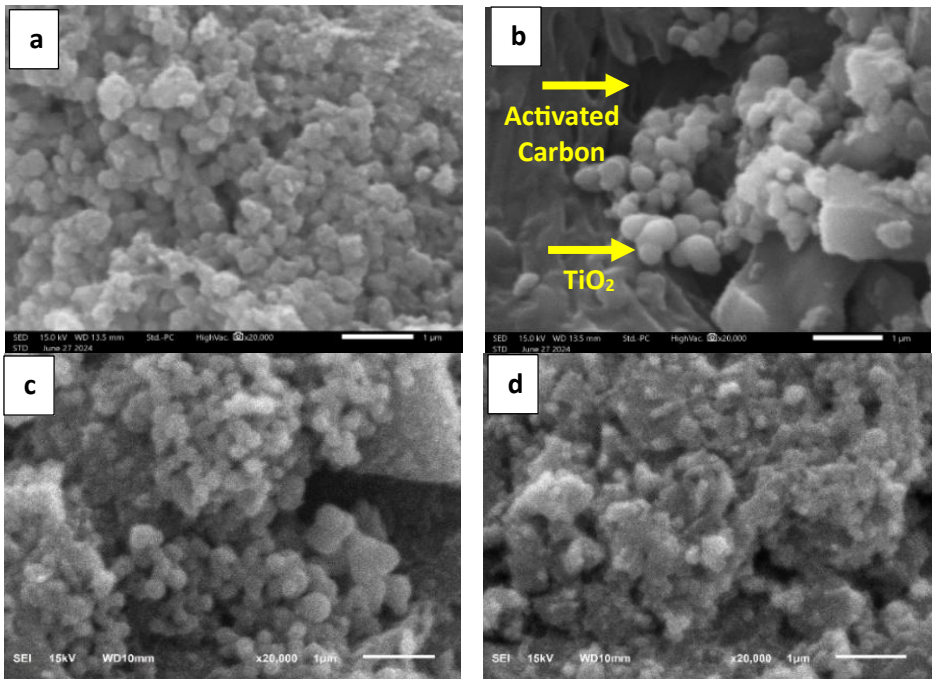

Figure 2. SEM characterisation results with 20,000 x magnification (**a**) TiO_2, (**b**) TiO_2-AC/5, (**c**) TiO_2-AC/10, (**d**) TiO_2-AC/15

Table 2. Presents the data on the constituents of the composite material

Sample	Elements (%)		
	C	O	Ti
TiO_2	0.69	26.04	73.27
TiO_2-AC (5)	43.91	28.70	27.40
TiO_2-AC (10)	71.31	8.57	20.12
TiO_2-AC (15)	79.27	7.39	13.34

Characterization Surface Area Analyzer

The synthesized material was characterized using a surface area analyzer and then analyzed using the BET method to determine the material's physical properties. TiO_2 exhibits a type IV isotherm (Figure 3) and forms a hysteresis curve at a pressure of 0.6 to 1.0, which is included in the mesoporous. Materials TiO_2-AC/5, TiO_2-AC/10, and TiO_2-AC/15 have a different graph shape than TiO_2 due to the addition of activated carbon. The three samples form a more significant type IVa isotherm graph for mesoporous particles due to capillary pore condensation. The TiO_2-AC sample shows a sharp increase in gas absorbed at relatively low-pressure values.

Figure 3 illustrates the existence of notable discrepancies in the adsorption outcomes, manifesting distinct hysteresis behavior because of the adsorbent milieu exhibiting open-loop characteristics. The addition of activated carbon may change the nature of the hysteresis loop for TiO_2-AC samples, which may then exhibit a hysteresis type of H4. This finding aligns with the conclusions of Shivakumar and Maitra (2020), who conducted a characterization of activated carbon derived from coconut husk and bagasse. The open-loop hysteresis observed in activated carbon can be attributed to the formation of bottleneck pores. These bottleneck pores facilitate particle condensation during the desorption stage, which occurs slower than the adsorption curve. This results in an open-loop hysteresis.

Furthermore, analysis utilizing the BJH (Barrett-Joyner-Halenda) method was conducted to provide insights into the pore size distribution. **Figure 4** illustrates that the TiO_2 sample exhibits a pore distribution with a range of 3-28 nm, whereas the TiO_2-AC/5, TiO_2-AC/10, and TiO_2-AC/15 samples display a range of 3-29 nm, classified as mesoporous. The four samples exhibit a macroporous structure, as evidenced by the 53-194 nm pore size range. These findings align with those of Sutisna et al. (2024), who synthesized TiO_2-coffee grounds with a pore size distribution encompassing a range of mesoporous and macroporous sizes

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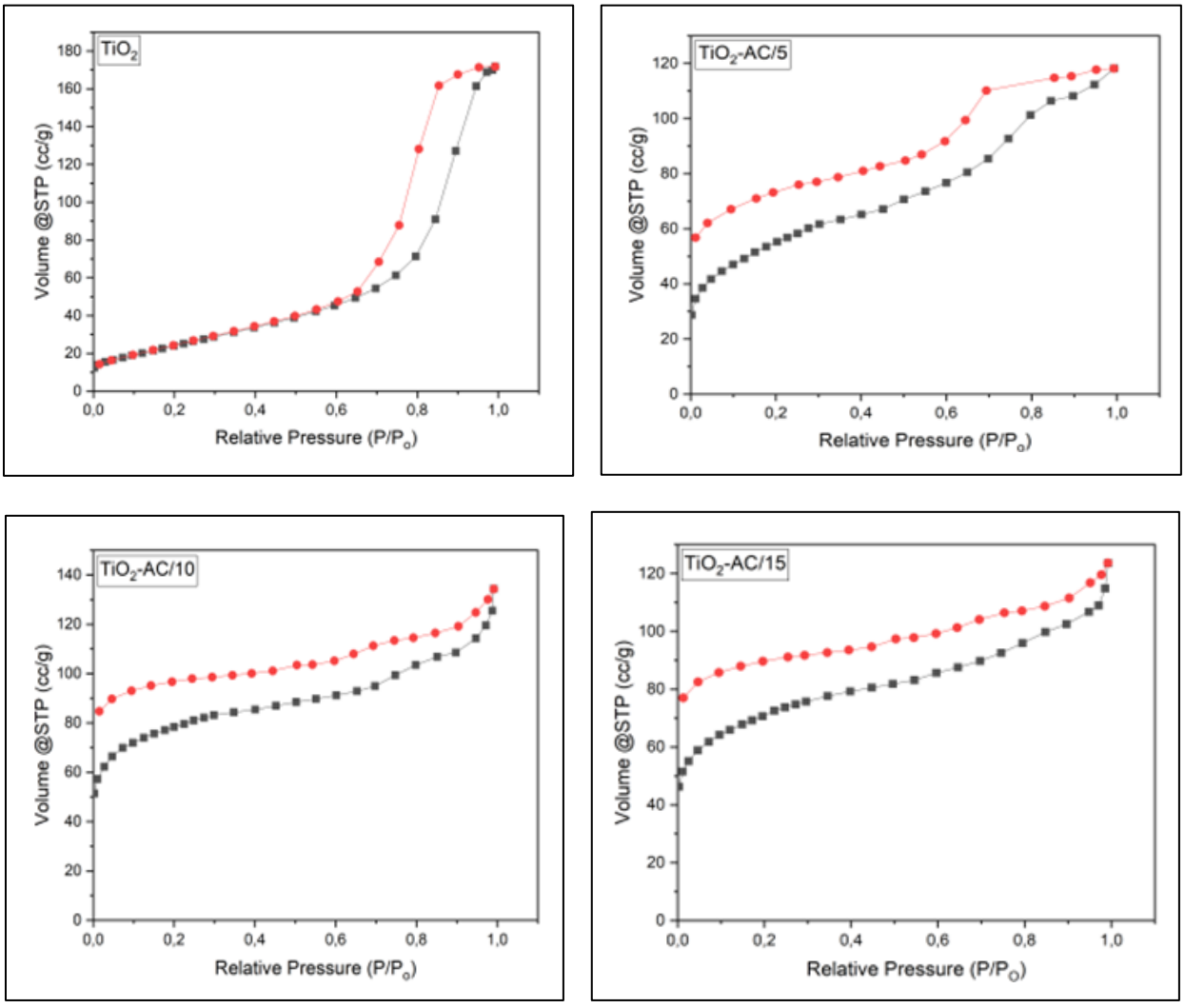

Figure 3. Adsorption-desorption isotherm curve

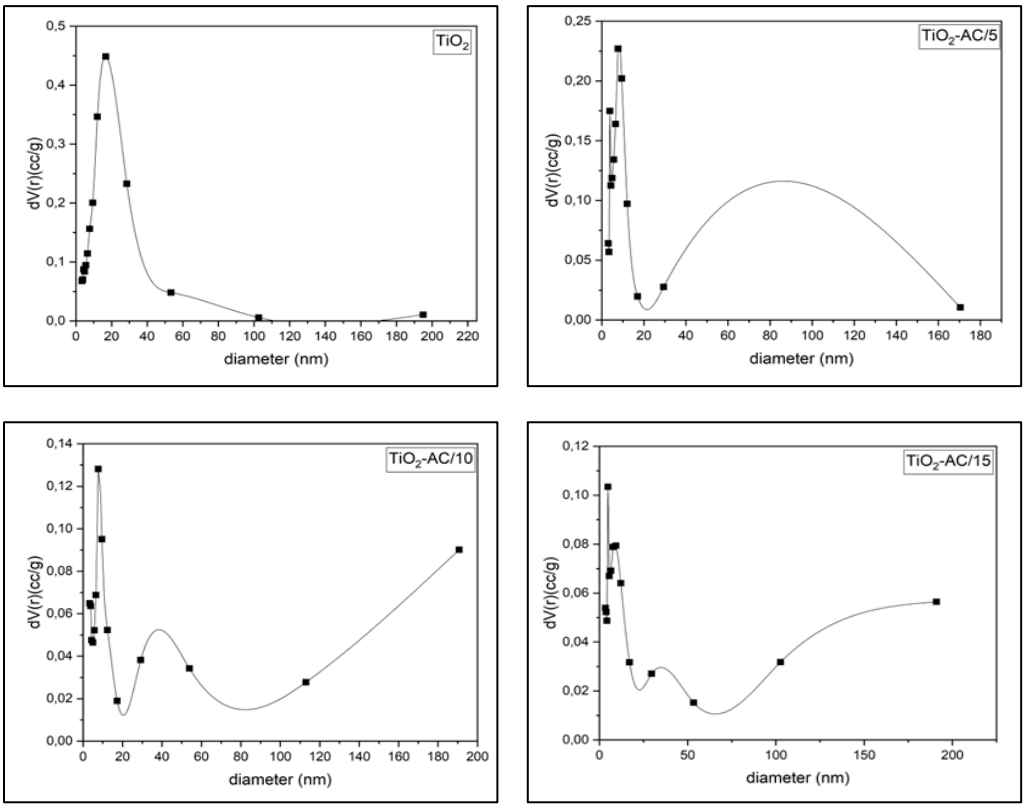

Figure 4. Pore size distribution of composite materials

Table 3. Surface area of composite materials

Samples	The Surface Area (m^2/g)	Mean Pore Diameter (nm)
TiO_2	95.02	11.180
TiO_2-AC/5	198.00	3.831
TiO_2-AC/10	286.10	2.905
TiO_2-AC/15	255.50	2.995

As demonstrated in **Table 3**, the surface area of TiO_2 and TiO_2-AC, as determined through BET characterization, is 95.02 m^2/g and 286.10 m^2/g, respectively. The surface area of TiO_2-AC/5, TiO_2-AC/10 and TiO_2-AC/15 is 198.00 m^2/g, 286.10 m^2/g and 255.50 m^2/g, respectively. It can be concluded that the larger surface area generally provides higher adsorption power in photocatalytic reactions. The surface area data has been demonstrated to be directly proportional to the average pore diameter produced, with a positive correlation between the two variables (Sutisna et al., 2023). A larger surface area is associated with a smaller pore diameter.

Characterization UV-Vis DRS

The TiO_2, TiO_2-AC/5, TiO_2-AC/10, and TiO_2-AC/15 samples were subjected to UV-Vis DRS characterization. The Tauc plot equation is employed for the determination of the band gap energy of semiconductor materials from data derived from UV-Vis absorption spectroscopy. The outcomes of the UV-Vis DRS characterization are illustrated in Figure 5. The band gap energy of TiO_2 is 3.26 eV, while in the composite materials TiO_2-AC/5, TiO_2-AC/10, and TiO_2-AC/15, the respective values are 3.23 eV, 3.21 eV, and 3.21 eV. The band gap energy value of the entire TiO_2 material is almost identical, with only a slight difference in the value observed. This is because the entire material is comprised of the anatase phase. As indicated in the referenced literature, the anatase phase is known to possess a band gap energy value of 3.2 eV (Sutisna et al., 2023). The findings regarding the band gap energy are consistent with the conclusions of Zeng et al. (2021), who determined that the band gap energy of pure TiO_2 is 3.07 eV and that of TiO_2-AC is 2,32. These results demonstrate that the addition of activated carbon impacts the band gap energy, although the effect is not pronounced. It has been demonstrated that the presence of activated carbon significantly affects the dispersion and size of TiO_2 particles. This observation is supported by morphological analysis conducted using SEM, which reveals that the activation of carbon leads to the dispersion of TiO_2 particles, resulting in smaller sizes. (Zeng et al., 2021)

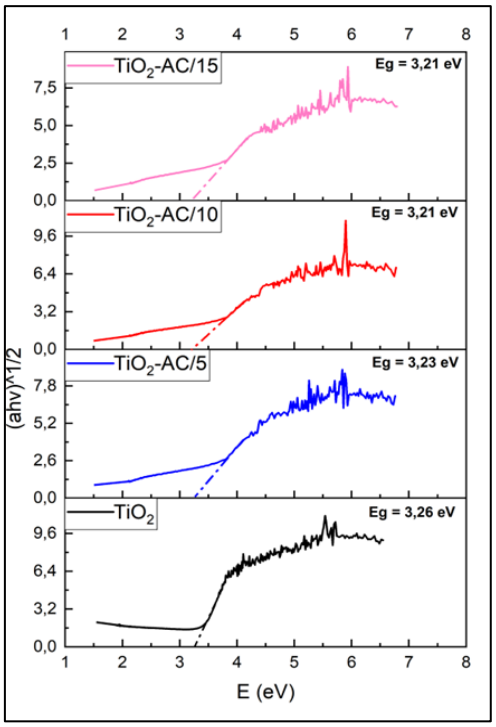

Figure 5. Band gap energy of composite materials

Photocatalytic Test

The photocatalytic activity of the synthesized TiO_2-AC samples was evaluated through the degradation of diazinon pesticides. The maximum absorbance value for the diazinon compound was observed at a wavelength of 248 nm, with a linear regression equation of $y = 0.0163x + 0.0169$ and an R^2 value of 0.9887. The enhanced photocatalytic activity can be attributed to combining TiO_2 with activated carbon, forming a composite material (Sutisna et al., 2023). The reduction of the diazinon solution is attributed to adsorption and photodegradation. The adsorption capacity of the synthesized material was evaluated by subjecting it to a stirred diazinon solution for one hour. The material combined with activated carbon is believed to exhibit high adsorption efficiency and a large surface area. This assertion can be substantiated by the findings of the Surface Area Analyser characterization presented in Table 3, which indicates that the TiO_2 material possesses the lowest surface area compared to the TiO_2-AC composite. The enhanced surface area will facilitate the creation of more active sites within the material, thereby optimizing the photocatalytic activity.

The photocatalytic activity of the synthesized material can be induced through irradiation with ultraviolet light. In the case of all variants of the TiO_2-AC composite material, a higher photocatalysis ability in reducing the percentage of diazinon pesticide solution was observed after 7 hours of UV radiation when compared to the TiO_2 material. However, over the subsequent 8 hours of irradiation, a negligible increase in the percent degradation was observed. This finding suggests that the adsorption mechanism of activated carbon is associated with the photocatalysis mechanism of TiO_2, thereby enhancing the photocatalysis ability of the TiO_2-AC composite material. As illustrated in **Figure 6**, the TiO_2-AC/10 composite material exhibits the highest photocatalytic activity in deleting the diazinon pesticide solution. During the photodegradation test, the TiO_2-AC/10 composite material demonstrated the capacity to reduce the absorbance of the diazinon pesticide solution by 65.18%. The other composite variants, namely TiO_2-AC/5 and TiO_2-AC/15, demonstrated a reduction in the absorbance of the diazinon pesticide solution by 56.32% and 60.14%, respectively, over the same time interval.

Furthermore, the SEM results indicate that the material exhibits increased photocatalytic activity, displaying a spherical surface morphology with a non-uniform size and TiO_2 attached to the surface of activated carbon. The XRD characterization of the four synthesized TiO_2 materials revealed peaks characteristic of anatase and distinct from those observed in other TiO_2 structures. The intensity of these peaks, particularly at 25.3°, which is the highest, also corroborates the anatase structure. The crystallite in the composite material demonstrated that alterations in the addition of activated carbon can influence the crystallite of the composite material.

The results of the UV-Vis DRS characterization also indicate that the synthesized material is in the anatase phase. This is evidenced by the band gap energy, which falls within the range of 3.2-3.3. It is established that the anatase phase exhibits greater photocatalytic activity than the brookite or rutile phase. The advantages of anatase TiO_2 include high thermal

stability and the capacity to absorb significant quantities of UV light, thereby enhancing photocatalytic activity (Sutisna et al., 2023). The surface area of the sample TiO_2-AC/10 (**Table 3**) is greater than that of the other materials. The larger surface area will result in increased degradation because a larger surface area has more active sites (Thambiliyagodage et al., 2022). Salsabillah (2021) reported that the highest percentage of degradation of diazinon was 67.13% at an irradiation time of nine hours with a material mass of 1.6 grams.

Reusability of Synthetic Materials

Reusability is the ability of a material to be reused without loss of performance. Good reusability is important as it can reduce operating costs and environmental impact. Materials that can be used multiple times are more efficient, energy-saving, and environmentally friendly, making them a more sustainable choice for various industrial applications. This test aims to determine the composite material's ability to be used repeatedly so as not to create new waste problems for the environment. The reusability of the composite material was tested in two ways. The first step is to repeat the degradation cycle of the composite against diazinon, as shown in **Figure 7**.

Secondly, the reusability of the TiO_2–AC composite was evaluated through three cycles of 30 ppm diazinon photodegradation for seven hours each cycle. The degradation efficiency exhibited a decline of 12.58% (TiO_2-AC/5), 11.63% (TiO_2-AC/10), and 9.80% (TiO_2-AC/15), respectively, which serves to illustrate the stability of TiO_2 on activated carbon. The substantial decline in degradation efficiency is attributable to the presence of TiO_2, which is isolated from the activated carbon. X-ray fluorescence (XRF) analysis confirmed the presence of titanium (Ti) particles on the surface of the activated carbon, thereby supporting the material's reusability (Table 4). The reusability of the TiO_2–AC composite was evaluated by XRF to ascertain the stability of the elemental Ti following three cycles of diazinon degradation. The findings indicated that Ti remained firmly attached to the activated carbon's surface, indicating the composite's excellent durability. This stability is of great importance for large-scale applications in wastewater treatment. The phenomenon of non-uniform repetition in the TiO_2-AC/10 and TiO_2/15 composite materials is particularly interesting. The hypothesis that has been formulated is that there may be a release of TiO_2 material from the surface and, concomitantly, that there is TiO_2 material that emerges from the pores to the surface of the catalyst. While elemental analysis using XRF focuses on the material on the surface, it does not reach the catalyst's more profound, more complex pores.

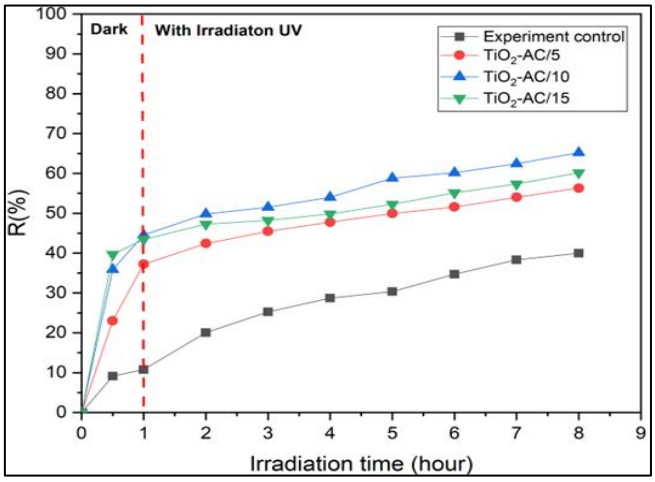

Figure 6. Diazinon Photodegradation Process

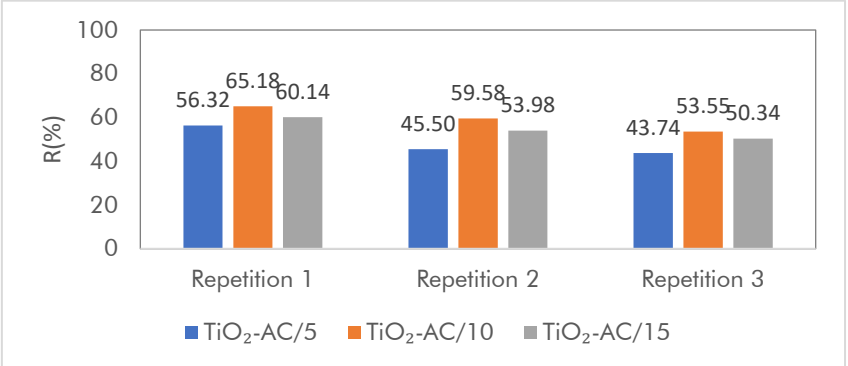

Figure 7. Illustrates the results of the percent degradation of diazinon.

Table 4. Elemental value of Ti in composite materials

Composite Materials	The Number of Ti (%)		
	Repetition 1	Repetition 2	Repetition 3
TiO_2-AC/5	24.152	20.246	19.894
TiO_2-AC/10	17.844	14.150	17.841
TiO_2-AC/15	12.265	11.203	14.437

CONCLUSIONS

The TiO_2 and TiO_2-AC samples exhibit a spherical morphology with a non-uniform particle size distribution. X-ray diffraction (XRD) and ultraviolet-visible diffuse reflectance spectroscopy (UV-Vis DRS) characterization revealed diffraction peaks corresponding to JCPDS No. 21-1272 and an energy band gap of 3.2-3.3 eV in the anatase phase. The surface areas of the TiO_2, TiO_2-AC/5, TiO_2-AC/10, and TiO_2-AC/15 samples were found to be 95.02 m^2/g, 198.00 m^2/g, 286.10 m^2/g, and 255.50 m^2/g, respectively. These values indicate the presence of mesoporous dominance. The TiO_2-AC/10 sample exhibited the highest photocatalytic activity, degrading 65.18% of diazinon in seven hours, despite a 9-13% decrease in degradation from the first to the third cycle. XRF characterization demonstrated the stability of Ti particles on the activated carbon surface.

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