PHOTOCATALYTIC DEGRADATION OF BATIK CUAL WASTEWATER USING $Fe_3O_4/SiO_2/TiO_2$ COMPOSITE DERIVED FROM TIN TAILING

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Received : October 24, 2023 Accepted : July 9, 2024 Published : December 31, 2024

Abstract: Batik cual dye wastewater is produced from the batik cual industry in Bangka, which contains Remazol dye, which is carcinogenic and a non-biodegradable organic compound that can be a pollutant for the environment. The elevated levels of remazol dyes in Batik Cual wastewater necessitate effective and environmentally safe waste management methods. One such method is photodegradation, utilizing a $Fe_3O_4/SiO_2/TiO_2$ composite photocatalyst derived from tin tailings sand. The $Fe_3O_4/SiO_2/TiO_2$ composites were synthesized via the hetero-agglomeration method. XRF characterization revealed that the SiO_2 content, following purification with HNO_3 acid leaching, reached 78.63%. XRD characterization of the tin tailings indicated the presence of peaks corresponding to quartz silica. Phase analysis using XRD diffractograms for the $Fe_3O_4/SiO_2/TiO_2$ composites showed peaks of magnetite, quartz silica, and a predominant anatase TiO_2 phase with strong diffraction patterns at 2θ angles of 27.42°; 39.19°; 44.00°, and 54.29°. UV-DRS analysis determined the band-gap energy of the $Fe_3O_4/SiO_2/TiO_2$ photocatalyst to be 1.9 eV. Morphological SEM analysis indicated that the synthesized $Fe_3O_4/SiO_2/TiO_2$ composite had a spherical, uneven, rough, and porous surface. The photodegradation of the $Fe_3O_4/SiO_2/TiO_2$ composite on cual batik dye waste showed that the optimal contact time for photodegradation was 60 minutes, and the catalyst dose was 500 mg at 98.1%. Consequently, these findings confirm that the $Fe_3O_4/SiO_2/TiO_2$ composite is a viable photocatalyst for photodegradation in handling cual batik dye waste.

Keywords: Photodegradation; Photocatalyst; Composite; Batik cual waste

Abstrak: Limbah zat warna batik cual dihasilkan dari industri batik cual di Bangka yang mengandung zat warna *Remazol*, bersifat karsinogenik dan merupakan senyawa organik *non-biodegradable* yang dapat menjadi pencemar bagi lingkungan Tingginya kadar zat warna *remazol* pada limbah batik cual membutuhkan penanganan limbah yang tepat dan aman bagi lingkungan. Salah satu metode yang dinilai efektif yaitu metode fotodegradasi menggunakan fotokatalis komposit $Fe_3O_4/SiO_2/TiO_2$ berbahan pasir *tailing* timah. Karakterisasi XRF menunjukkan kadar SiO_2 setelah dilakukan pemurnian *acid leaching* HNO_3 mencapai 78,63%. Hasil karakterisasi XRD *tailing* timah menunjukkan terdapatnya puncak yang menandakan silika kuarsa. Komposit $Fe_3O_4/SiO_2/TiO_2$ berdasarkan analisis fasa menggunakan XRD difraktogram menunjukkan puncak magnetit, silika kuarsa dan didominasi fasa TiO_2 *anatase* pada sudut 2θ 27,42°; 39,19°; 44,00°; dan 54,29. Berdasarkan

DOI: 10.22373/ekw.v10i2.20506

analisis UV-DRS didapatkan energi *ban-gap* untuk fotokatalis $Fe_3O_4/SiO_2/TiO_2$ sebesar 1,9 eV. Analisis morfologi pada komposit $Fe_3O_4/SiO_2/TiO_2$ menunjukkan bentuk sperik, permukaan yang tidak rata, kasar dan berpori. Uji fotodegradasi komposit $Fe_3O_4/SiO_2/TiO_2$ terhadap limbah zat warna batik cual didapatkan waktu kontak optimal fotodegradasi pada waktu 60 menit dan massa komposit 500 mg sebesar 98.1%. Sehingga dengan hasil tersebut dapat disimpulkan bahwa komposit dapat dijadikan fotokatalis dalam proses fotodegradasi dalam menangani limbah zat warna batik cual.

Kata Kunci : Fotodegradasi; Fotokatalis; Komposit; Limbah batik cual

Recommended APA Citation :

Fabiani, V. A., Putri, R. E., Davina, L. & Viriliana, Z. (2024). Photocatalytic Degradation of Batik Cual Wastewater Using $Fe_3O_4/SiO_2/TiO_2$ Composite Derived From Tin Tailing. *Elkawnie*, *10*(2), 221-231. https://doi.org/10.22373/ekw.v10i2.20506

Introduction

The dye wastewater resulting from the production of Batik Cual in Bangka contains carcinogenic remazol dyes and non-biodegradable organic compounds, which pose significant environmental contamination risks (Istiqomah et al., 2021). The high remazol dyes in batik cual waste require proper and environmentally safe waste handling (Fadillah Ramadhani et al., 2023; Lata et al., 2024). One practical and easily applicable method in Indonesia is photodegradation.

Extensive research on semiconductor photocatalysts for photodegradation has been conducted in the environmental sector. TiO_2 is recognized as a highly effective semiconductor photocatalyst due to its stability and high photocatalytic efficiency in addressing environmental issues (Sagadevan et al., 2022). However, using TiO_2 becomes less efficient because TiO_2, which functions as a photocatalyst, is challenging to retrieve from the dye. To overcome this, a Fe_3O_4/TiO_2 magnetic composite material was developed. Thus, using catalyst materials becomes more efficient and reduces the possibility of pollution by the catalyst material itself (Lee et al., 2022). Additionally, magnetic materials can be combined with adsorbent materials such as SiO_2 (Bilgic, 2022)

Interestingly, SiO_2 (silica) can be sourced from tin mining by-products (tailings), which are abundant on Bangka Island but remain underutilized. Evi et al. (2019) reported that tin tailings contain 80-90% SiO_2, making them an innovative source of SiO_2 for $Fe_3O_4/SiO_2/TiO_2$ photocatalysts. Combining TiO_2 with magnetic materials like Fe_3O_4 enhances the effectiveness of TiO_2 in degrading dyes, as Fe_3O_4 facilitates the retrieval of catalyst materials dispersed in dyes and provides magnetic absorption properties. Adding SiO_2 to these materials can maintain the stability of the TiO_2 catalyst and the function of Fe_3O_4 (Wardiyati et al., 2016). Wang et al. (2012) reported the preparation of $Fe_3O_4/SiO_2/TiO_2$ composites through the sol-gel method with a core-shell structure, so Li et al. (2020) modified the Stober method by replacing alkoxy groups to control the silica layer protecting the magnetite core, resulting in a sol-gel with a SiO_2 inner layer and a TiO_2 outer layer. Both studies demonstrated superior photodegradation capabilities with a core-shell structure for treating organic wastewater.

Based on this study, the present research aims to synthesize $Fe_3O_4/SiO_2/TiO_2$ composites from tin tailings and apply them as photocatalysts to degrade Batik Cual waste. Given that research utilizing SiO_2 from tin tailings has not been conducted previously, this study seeks to optimize local mineral resources on Bangka Island while addressing environmental problems caused by dye waste.

Method

Materials

The materials utilized in this research include tin tailings, iron (II) sulfate heptahydrate $FeSO_4.7H_2O$ (Merck, Germany), iron (III) chloride hexahydrate $FeCl_3.6H_2O$ (Merck, Germany) ammonium hydroxide 25% (Merck), distilled water, nitric acid HNO_3 68% p.a, titanium dioxide TiO_2 (Merck, Germany), ammonium sulfate $(NH_4)_2SO_4$), Batik cual waste taken from the Maslina Batik cual UMKM in Pangkalpinang, sodium hydroxide NaOH (Merck, Germany), and hydrochloric acid HCl (Merck, Germany).

Instrumentation

The equipment used in this research is a digital balance, magnetic stirrer, hot plate, UV-Vis spectrophotometer Shimadzu 1800, Scanning Electron Microscopy (SEM) Thermo Fisher Scientific, X-ray diffraction (XRD) PANalitycal MPD PW3040/60, X-ray fluorescence (XRF) PANalitycal, UV-Vis Diffuse Reflectance (UV-DRS) Shimadzu UV 2600 and standard laboratory glassware.

Preparation of SiO_2 from tin tailing

The tin tailings were washed with distilled water to remove macro impurities, then oven-dried at 110°C. After drying, the tailings were ground using a mortar and sieved through a 200-mesh sieve to obtain finer particles. The purification process involved acid leaching using 15% HNO_3 with a 1:10 ratio. Specifically, 50 grams of finely ground tin tailings were leached with 500 mL of 15% HNO_3 for 3 hours. The leachate was filtered, the residue was washed with distilled water, and then oven-dried at 110°C to yield SiO_2 powder. This powder was then characterized using XRD and XRF. Subsequently, 20 grams of SiO_2 obtained from the tin tailings was added to 250 mL of 3M NaOH solution. This mixture was heated on a hotplate stirrer at 80°C until boiling and thickening. The resulting sodium silicate filtrate was filtered using filter paper (Utari et al., 2020).

Synthesis of Fe_3O_4

Fe_3O_4 was synthesized by dissolving 4.17 grams of $FeSO_4 \cdot 7H_2O$ and 8.109 grams of $FeCl_3 \cdot 6H_2O$ in 30 mL of distilled water. Then, 60 mL of 10% NH_4OH solution was gradually added while stirring with a magnetic stirrer at 60°C and 450 rpm for 90 minutes. The resulting Fe_3O_4 solution was rinsed with distilled water, and the Fe_3O_4 precipitate was collected using an external magnet, washed seven times with distilled water, dried in an oven at 100°C for two hours, and then ground into a powder (Wardhani et al., 2023).

Synthesis of Composite $Fe_3O_4/SiO_2/TiO_2$

The synthesis involved mixing 2.3 mL of sodium silicate solution with the previously synthesized Fe_3O_4 precipitate. The solution's pH was adjusted to 8 using 2M HCl. In another beaker, 4.64 grams of TiO_2 was dispersed in 100 mL of 0.02M $(NH_4)2SO_4$ and sonicated for 30 minutes at room temperature. The dispersed TiO_2 was then mixed into the Fe_3O_4/SiO_2 suspension, and the pH was adjusted to 5 using 1M HCl. This mixture was sonicated for 30 minutes, centrifuged at 4000 rpm to separate solids and liquids, dried overnight in an oven at 60°C, and dried at 100°C for 3 hours. The final product, $Fe_3O_4/SiO_2/TiO_2$ composite, was characterized using XRD, SEM, and DR UV.

Photocatalytic Test

For the photocatalytic test, 20 mL of Batik Cual dye wastewater was treated with varying masses (400, 450, and 500 mg) of $Fe_3O_4/SiO_2/TiO_2$ composites, placed under UV-C light, and stirred for 60 minutes using a magnetic stirrer. The $Fe_3O_4/SiO_2/TiO_2$ composite was then filtered from the solution, and its absorbance was measured using UV-Vis spectrophotometry at a wavelength of 385.35 nm. Additionally, experiments were conducted with varying stirring times. For this, 20 mL of Batik Cual waste was treated with 450 mg of $Fe_3O_4/SiO_2/TiO_2$ composite and irradiated with a UV-C lamp for 30, 60, and 90 minutes (Istiqomah et al., 2021). The percentage degradation was calculated using the following formula:

$$\% \text{ degradation} = \frac{Co - Ct}{Co} \times 100\ \% \text{ ....(1)}$$

Co = initial concentration of dye
Ct = final concentration of dye

Result and Discussion

Preparation of SiO_2 from Tin Tailing

1. *Composition of Tin Tailing*

DOI: 10.22373/ekw.v10i2.20506

According to XRF analysis, SiO_2 constitutes the highest percentage in tin tailings, precisely 78.63%. The increase in SiO_2 content is accompanied by a reduction in impurity levels after purification.

Table 1. Composition of tin tailing sands

Sample	SiO_2 (%)	Al_2O_3 (%)	TiO_2 (%)	ZrO_2 (%)	P_2O_5 (%)
Tin Tailing (Before Leaching)	71,92	2,32	4,62	14,5	2,86
Tin Tailing (After Leaching)	78,63	1,12	3,50	11.3	2,63

Table 1. shows that the SiO_2 content in tin tailings increases after the leaching process with nitric acid, accompanied by a decrease in impurities such as Al_2O_3, TiO_2, and ZrO_2. Post-leaching, the relative weight percentage of SiO_2 is higher than other constituents. However, the increase in SiO_2 is not substantial, which may be attributed to the initially high impurity content in the tin tailings sand (Pusporini et al., 2020). The composition of the tin tailings aligns with the findings of Evi et al. (2019), which include SiO2, Al_2O_3, K_2O, TiO_2, and CaO.

2. *The phase of SiO_2 Tin Tailings*

The SiO_2 (silica) phase was also analyzed using X-ray diffraction (XRD). The XRD analysis results are presented in the diffractogram shown in Figure 1 below,

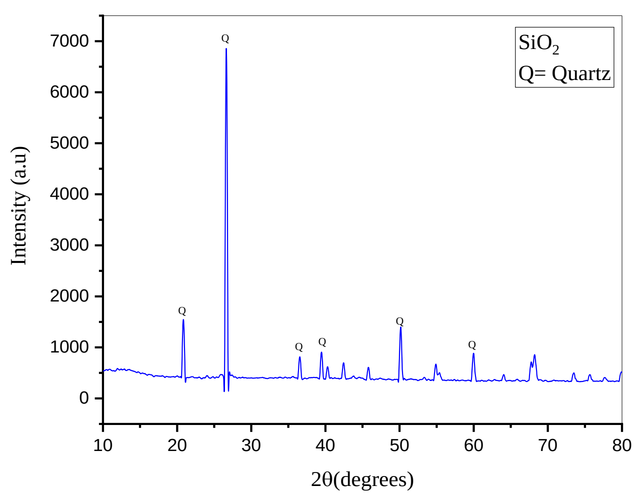

Figure 1. Diffractogram SiO_2

Figure 1 shows the diffraction pattern with sharp peaks, indicating that the formed silica structure is in a crystalline phase. Phase identification reveals that the peaks correspond to the quartz silica phase, consistent with ICDD data No. 01-075-

DOI: 10.22373/ekw.v10i2.20506

8320 at 2θ = 20.84° (100), 26.62° (011), 40.26° (011), 50.58° (003), 59.92° (121), and 62.13° (121). XRD analysis using the Debye-Scherrer equation shows that the average size of the SiO_2 crystallites formed is 66.41 nm.

Synthesis $Fe_3O_4/SiO_2/TiO_2$ Composite

1. Phase of $Fe_3O_4/SiO_2/TiO_2$ Composite

XRD analysis was performed to determine the phase formed in the composite. The diffractogram of the synthesized composite is shown in Figure 2.

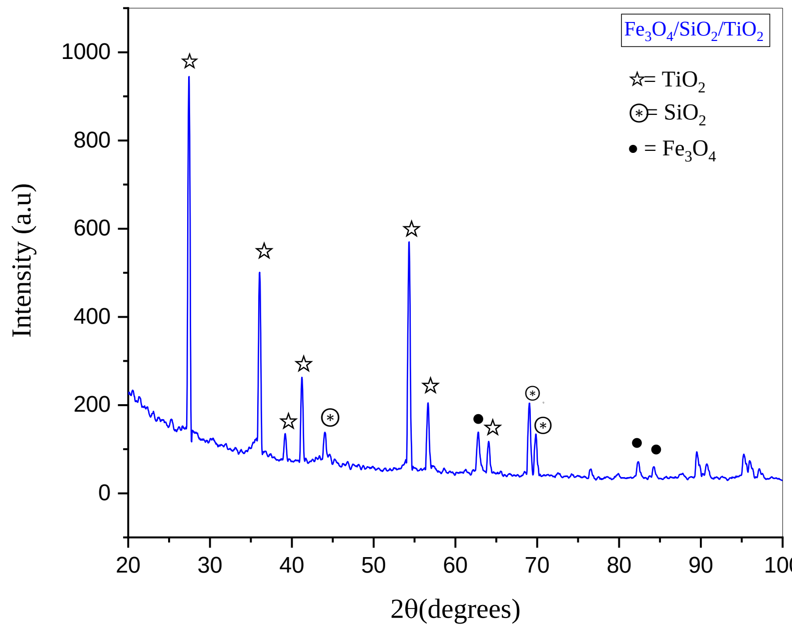

Figure 2. Diffractogram of $Fe_3O_4/SiO_2/TiO_2$ Composite

The diffractogram shows peaks at angles 62.73° and 82.32°, which correspond to the XRD patterns of magnetite, matching the JCPDS No. 85-1436 standard. In addition to the identified Fe_3O_4 phase, several peaks indicate typical TiO_2, aligning with JCPDS No. 21-1712. These are characterized by sharp diffraction patterns at 2θ angles of 27.42°, 39.19°, 44.00°, and 54.29°, indicating the anatase TiO2 phase (Theivasanthi & Alagar, 2013). The diffraction peaks in the $Fe_3O_4/SiO_2/TiO_2$ composite are predominantly from the anatase TiO_2 phase. Research by Esfandiari et al. (2020) indicates that the $Fe_3O_4/SiO_2/TiO_2$ composite's diffraction pattern, dominated by anatase TiO_2, suggests a successful coating process of TiO_2 onto the SiO_2 surface.

2. Morphology Analysis of $Fe_3O_4/SiO_2/TiO_2$ composites

SEM characterization was performed to determine the morphology of the $Fe_3O_4/SiO_2/TiO_2$ composite. SEM analysis provides information on the particle surface's texture and crystallographic details. The SEM analysis results of $Fe_3O_4/SiO_2/TiO_2$ composites are shown in Figure 3 below,

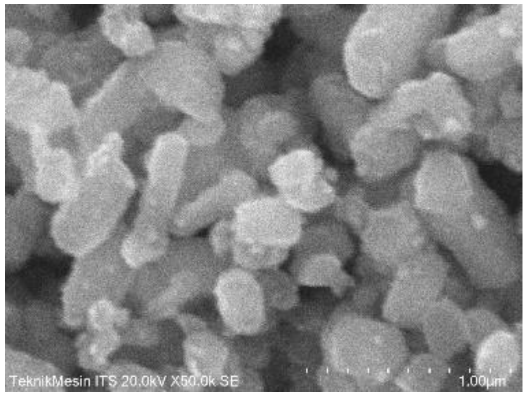

Figure 3. Morphology of $Fe_3O_4/SiO_2/TiO_2$

Figure 3 reveals that the synthesized $Fe_3O_4/SiO_2/TiO_2$ composite has a spherical structure with a rough and porous surface. According to Cimen et al. (2024), this morphology enhances the photocatalytic activity of the $Fe_3O_4/SiO_2/TiO_2$ composite against dyes. Additionally, the slightly rounded structure observed characterizes the core-shell structure of the composite, facilitating better contact with dyes and achieving higher degradation (Brossault et al., 2021).

3. Band Gap Energy Analysis

The band gap energy can be calculated using the Kubelka-Munk equation. The calculated band gap energy values for the $Fe_3O_4/SiO_2/TiO_2$ composite are presented in Table 2.

Table 2. Band gap energy of Fe_3O_4/SiO_2 and $Fe_3O_4/SiO_2/TiO_2$

Sample	Band gap
Fe_3O_4/SiO_2	3,24 eV
$Fe_3O_4/SiO_2/TiO_2$	1,94 eV

Band gap energy serves as a crucial parameter for photocatalytic materials. A smaller band gap energy value indicates greater effectiveness, as it requires less energy for electron excitation from the valence band to the conduction band (Wardiyati et al., 2016). This facilitates increased production of hydroxyl radicals, thereby enhancing the oxidation capability against batik cual dyes. Based on the characterization results, the band gap energy value of the synthesized $Fe_3O_4/SiO_2/TiO_2$ is 1.94 eV. This finding aligns closely with previous research by Wardiyati *et al*. (2016), who reported a band gap energy of 1.9125 eV for $Fe_3O_4/SiO_2/TiO_2$ composites. Table 2 illustrates that incorporating TiO_2 into the Fe_3O_4/SiO_2 composite reduces the band gap energy, facilitating rapid excitation of electrons from the valence band.

Photocatalytic activity

To evaluate the photocatalytic activity of $Fe_3O_4/SiO_2/TiO_2$, degradation experiments were conducted on batik cual dye waste under UV-C light. Various experimental parameters, including irradiation time and catalyst dosage, were varied. The experimental results of batik cual degradation by $Fe_3O_4/SiO_2/TiO_2$ are depicted in Figure 4 are shown in Figures 4.

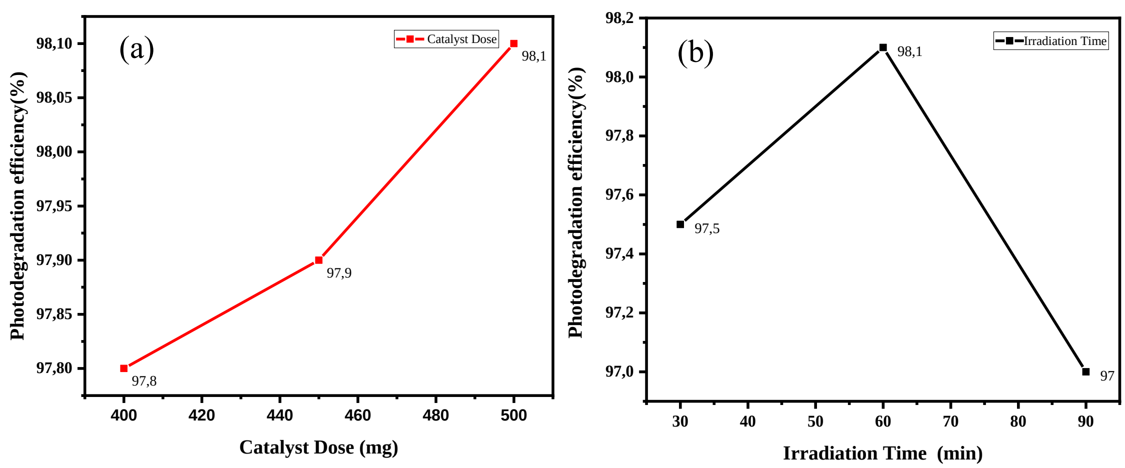

Figure 4. Photodegradation activity (a) effect of catalyst dose and (b) effect of irradiation time

Figure 4 illustrates the impact of catalyst dosage (a) and irradiation time (b) on the photodegradation of batik cual waste. The results indicate that irradiation time significantly influences the degradation efficiency of batik cual waste. Longer irradiation times correspond to higher percentages of batik cual degradation, with the peak efficiency observed at 98.1% after 60 minutes. However, the degradation efficiency slightly decreased to 97% after 90 minutes, likely due to prolonged exposure beyond optimal conditions. Additionally, Figure 4 demonstrates a positive correlation between catalyst dosage and degradation efficiency, achieving the highest degradation percentage of 98.1% at a composite dosage of 500 mg. This performance surpasses previous studies, such as Huang et al. (2011), which reported approximately 66% degradation efficiency.

The photodegradation process involves the release of electrons from the valence band to the conduction band, leaving behind positively charged holes (h^+). Hydroxyl radicals (-OH) are generated through the reaction of h^+ with OH- groups or water molecules on the TiO_2 surface. Simultaneously, electrons (e^-) in the conduction band react with dissolved O_2 to form superoxide ions (O_2^-). These reactive species, -OH and O_2^-, subsequently oxidize dye wastewater. Continuous exposure of the $Fe_3O_4/SiO_2/TiO_2$ composite to UV-C light promotes the formation of OH radicals, which actively degrade dye on the catalyst surface (Yang et al., 2024).

Conclusion

The acid leaching method effectively yields 78.6% SiO_2 from tin tailings, as confirmed by XRD analysis indicating a quartz silica phase. XRD data further reveals that the $Fe_3O_4/SiO_2/TiO_2$ composite comprises magnetite and quartz silica phases, predominantly featuring TiO_2 in its anatase phase. UV DRS analysis shows band-gap energies of 3.24 eV for Fe_3O_4/SiO_2 and 1.94 eV for $Fe_3O_4/SiO_2/TiO_2$.

Acknowledgement

The authors are grateful for the financial support from the Directorate General of Higher Education, Ministry of Education, Culture, Research, and Technology (Number 2283/E2/DT.01.00/2023). Gratitude is also extended to Universitas Bangka Belitung for providing essential laboratory facilities for this research.

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