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Sunlight Assisted Degradation of Linear Alkylbenzene Sulfonate by Floating Catalyst TiO_2-Coconut Fiber

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Abstract

The increasing number of laundry businesses in Pontianak causes increased laundry waste, which is dangerous for health and the environment because anionic surfactants such as Linear Alkylbenzene Sulfonate (LAS) are hard degradable. Photocatalyst is a method that can be used to degrade the LAS structure. TiO_2 carried in coconut fiber can optimize sunlight irradiation in degrading LAS content when light reaches the water's surface. This study aims to determine the characteristics and optimum activity time of photocatalyst TiO_2-coconut fiber in degrading LAS. Photocatalyst characterization was carried out using XRD, XRF, and DR-UV, while the optimum activity test of photocatalysts in degrading LAS was carried out using a UV-Vis spectrophotometer. XRD diffractogram analysis showed the peaks of coconut fiber at 2θ = 22.2°, 34.8° and TiO_2 at 2θ = 25.3°, 37.8°, 48.1°, 55.1°, and 62.1°. The TiO_2 attached to the fiber after being synthesized was 21.12%. The band gap of TiO_2 and TiO_2-coconut fiber is 3.21 and 3.18 eV, with light absorption at 386.5 and 390.3 nm. Photocatalyst was carried out in LAS with a mass ratio of TiO_2 and coconut fiber of 20:80; 30:70; 40:60, and 50:50 w/w with a time range of 0, 30, 60, 90, and 120 minutes. The results of photocatalysis of TiO_2-coconut fiber in a ratio of 20:80 w/w showed the optimum photocatalytic activity at 120 minutes with the highest degradation rate of 80.43%. This research is expected to be applied as an alternative to handling LAS in laundry industry waste.

Keywords: laundry waste, linear alkylbenzene sulfonate, photocatalyst, TiO_2

1. Background

The laundry industry is an industry whose activities use water and detergent for washing [1]. The increasing number of laundry businesses in Pontianak City causes an increase in the amount of waste produced [2]. The waste is often discharged into the environment without prior treatments, polluting the environment due to anionic surfactants such as Linear Alkylbenzene Sulfonate (LAS) (Figure 1), which may harm health and the environment [3].

LAS compound is an anionic surfactant that is used as an essential ingredient in the manufacture of detergents. The surfactants in these detergents are difficult to decompose biologically, and they will impact the environment [4]. The presence of LAS waste in water degrades aerobically but takes a long time, which is about nine days, and only degrades 50% [5]. Therefore, an effective alternative method is needed to reduce LAS content in laundry waste, one of which is the photocatalyst method.

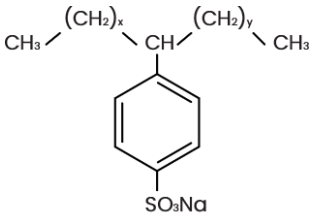

Figure 1. General Chemical Structure of LAS [6]

The photocatalyst method uses light and a semiconductor catalyst to accelerate chemical transformations. The light source may come from visible light and UV light. The basic mechanism of the photocatalyst is when TiO_2 absorbs sunlight with the same or greater energy; it causes a charge

separation or photoexcitation, which produces hydroxyl radicals (•OH) which are very reactive in attacking organic molecules [7]. The photocatalyst method is widely used because it has several advantages. The process is not complicated, the processing time is relatively fast, and the materials used are cheap and easy to obtain [4].

TiO_2 is a semiconductor catalyst with an energy gap of 3.2 eV, which is non-toxic and affordable [8]. Several previous studies have used TiO_2 powder as a photodegradation of surfactant compounds carried out by sprinkling TiO_2 powder directly into laundry waste [9]. This technique has drawbacks, namely that pure TiO_2 powder is used less optimally because of its relatively low surface area. It becomes a new pollutant in the waste if it cannot separate TiO2 powder and wastewater after the clarification process [10]. In addition, TiO_2 also has a downside for its low effectiveness when deposited into the bottom of the water. Only 20% of visible light and 1% of Ultraviolet (UV) light can penetrate water with a depth of 0.5 meters [11]. One of the carriers that can be used is coconut fiber (Figure 2 (b)). Coconut fiber was chosen because it has several advantages compared to other photocatalyst materials, including lightweight and high cellulose content [12]. fibers that underwent a delignification process would reduce the lignin and hemicellulose content, thus increasing the number of cavities in the fiber [13].

(a)  (b) 

Figure 2. *(a)* Coconut, and *(b)* Coconut Fiber

Based on the points mentioned above, a TiO_2-coconut fiber photocatalyst was synthesized for the photodegradation of Linear Alkylbenzene Sulfonate by optimizing the aid of direct sunlight in the UV range. This study will examine the ratio of mass (TiO_2: coconut fiber), determine the density, and determine the character of TiO_2-coconut fiber. Photocatalyst characterization used X-Ray Diffraction (XRD) to determine the type of TiO_2 crystal, X-Ray Fluorescence (XRF) to determine the chemical composition of the photocatalyst, and Diffuse Reflectance-Ultraviolet (DR-UV) to determine the band gap energy. The degradation efficiency of Linear Alkylbenzene Sulfonate was determined using a UV-Vis spectrophotometer.

2. Methodology

2.1 Tools and Materials

The tools used in this research are a stir bar, spray bottle, hot plate SCILOGEX, Lux Meter KRISBOW, analytical balance BEL, oven ESCO, sonicator BRANSON, spatula, quartz tube Iwaki, X-Ray Diffraction (XRD) PANalytical, X-Ray Fluorescence (XRF) PANalytical Epsilon 3, Diffuse Reflectance–Ultraviolet (DR-UV), and UV-Vis Spectrophotometer Shimadzu UV-2600.

The materials used are distilled water (H_2O), laundry waste, sulfuric acid (H_2SO_4) Merck, ethanol (C_2H_5OH) SMART-LAB, chloroform ($CHCl_3$) Merck, Linear Alkylbenzene Sulfonate (LAS), methylene blue Merck, sodium hydroxide (NaOH) Merck, sodium dihydrogen phosphate monohydrate ($NaH_2PO_4.H_2O$) Merck, titanium oxide (TiO_2) Merck and coconut fiber.

2.2 Sample Preparation

Samples of coconut fiber were washed using clean water and dried to a constant weight at 105˚C for 24 hours in a laboratory drying oven [14]. After that, the coconut fiber was cut into small sizes and then filtered using a shiver shaker of 3 mm and 1 mm. Then, the samples retained in the 1 mm sieve were retrieved for the next step.

2.3 Coconut Fiber Activation

Coconut fiber was subjected to a delignification process using organosolv treatment. Organosolv treatment is an effective delignification method to remove lignin and hemicellulose content from natural fibers [15]. Coconut fiber was soaked using a 2:1 of water:ethanol solvent with ultrasonic treatment for 2 hours to clean impurities, increased the number of pores, and reduced the lignin and hemicellulose components in the lignocellulosic structure of coconut fiber [13]. The coconut fiber was filtered to get activated coconut fiber.

2.4 Synthesis of TiO_2-Coconut Fiber

Synthesis of TiO_2-coconut fiber was carried out by adding 5 g of coconut fiber and 5 g of TiO_2 into distilled water:ethanol with a ratio of 2:1. The suspension was stirred using a magnetic stirrer for 2 hours to get a homogeneous suspension [16]. The residue obtained was then filtered and dried at

80°C for 7 hours to produce TiO_2-Coconut Fiber. TiO_2-coconut fiber obtained was then separated using a sieve shaker with a 100 mesh sieve (0.15 mm) to separate unattached TiO_2 from coconut fiber. The TiO_2-coconut fiber photocatalyst was prepared using a mass ratio (coconut fiber: TiO_2) of 80:20; 70:30; 60:40, and 50:50 w/w.

2.5 Density Measurement

The measurement of material density ρ_f was carried out using the Archimedes method as in equation (1)

$$\rho_f = \frac{(M_3 - M_1)\rho_l}{((M_3 - M_1) - (M_4 - M_2))} \quad (1)$$

with M_1 is the weight of the empty pycnometer, M_2 is the weight of the pycnometer filled with distilled water, M_3 is the weight of the pycnometer filled with material, M_4 is the weight of the pycnometer filled with material in liquid, and ρ_l is the density of aquades (0.997 g/cm^3). The density of materials calculated includes TiO_2, coconut fiber, and TiO_2-coconut fiber mass ratio variations of 50:50, 40:60, 30:70, and 20:80 w/w.

2.6 Linear Alkylbenzene Sulfonate Degradation Activity Test

Photocatalyst activity test of TiO_2-coconut fiber was carried out by adding 200 mL of LAS with a concentration of 100 ppm and 100 mg of TiO_2-coconut fiber. The suspension was then irradiated in the sun for 0, 30, 60, 90, and 120 minutes using a hot plate stirrer with three repetitions in a quartz tube, and the light intensity was measured using a Lux Meter. The concentration of LAS was determined using the MBAS (Methylene Blue Active Substance) method. 10 mL of LAS sample was put into a separating funnel and then added with 2.5 mL of methylene blue solution (0.0125 g methylene blue: 12.5 g $NaH_2PO_4.H_2O$: 1.7 mL H_2SO_4: distilled water up to 250 ml in a volumetric flask), and 5 mL of chloroform, then was shaken. The gas was removed for 2×1.5 minutes and then set aside until the water and the chloroform phase formed on the flask's top and bottom. The chloroform phase was collected in a different separating funnel, while the aqueous phase was discarded. The chloroform phase was added with 5 mL of washing solution (12.5 g $NaH_2PO_4.H_2O$: 1.7 mL H_2SO_4: distilled water to 250 mL in a volumetric flask) was shaken again while removing the gas for 1.5 minutes and allowed to remain still. The lower layer was collected in a 10 mL volumetric flask and diluted to the mark with chloroform, while the upper phase was discarded [17]. The diluted solution was ready to be tested with a UV-Vis spectrophotometer with a maximum wavelength of 653.6 nm [18].

$$\text{Degradation Efficiency} = \frac{A_0 - A_t}{A_0} \text{x } 100\% \quad (2)$$

Where A_0 (mg L^{-1}) is the initial concentration of LAS, and A_t is the concentration of LAS degraded at a certain time [16].

2.7 Characterization and Identification of TiO_2-Coconut Fiber Photocatalyst

TiO_2 and photocatalyst TiO_2-coconut fiber with a mass ratio (TiO_2: coconut fiber) of 50:50 w/w were characterized using XRD at Padang University, XRF at Padang University, and DR-UV at Indonesia University.

3. Result and Discussion

3.1 X-Ray Diffraction (XRD)

The result of XRD characterization is 2θ angle spectrum data describing the character of the analyzed crystal [19]. This study used XRD to determine and ascertain the desired character of TiO_2, coconut fiber, and TiO_2-coconut fiber. The results of the XRD analysis are shown in Figure 3. Based on the analyzed XRD diffractogram, the spectrum of TiO_2 has peak characteristics of 2θ = 25.3°, 37.8°, 48.1°, 55.1°, and 62,1° with lattice spacings of 0.353 nm, 0.237 nm, 0.189 nm 0.166 nm and 0.148 nm which is the peak of anatase type TiO_2 compound in the form of crystals. The diffractogram of TiO_2 is in line with the literature, which shows peaks of 2θ = 25.1°, 37.6°, 47.9°, and 54.9° with a lattice spacing of 0.35 nm, 0.23 nm, 0.18 nm, and 0.16 nm identifying TiO_2 anatase compound [20]. Characteristics of coconut fiber showed peaks of 2θ = 22.2° and 34.8°, indicating the presence of SiO_2 mineral compounds and carbon elements [21]. Diffractogram of TiO_2–coconut fiber showed peak characteristics of 2θ = 22.1°, 25.3°, 34.8°, 81.4°, and 98.1°. These five peaks showed TiO_2 anatase and coconut fiber compounds with carbon peaks at 2θ = 34.8° and 98.1°.

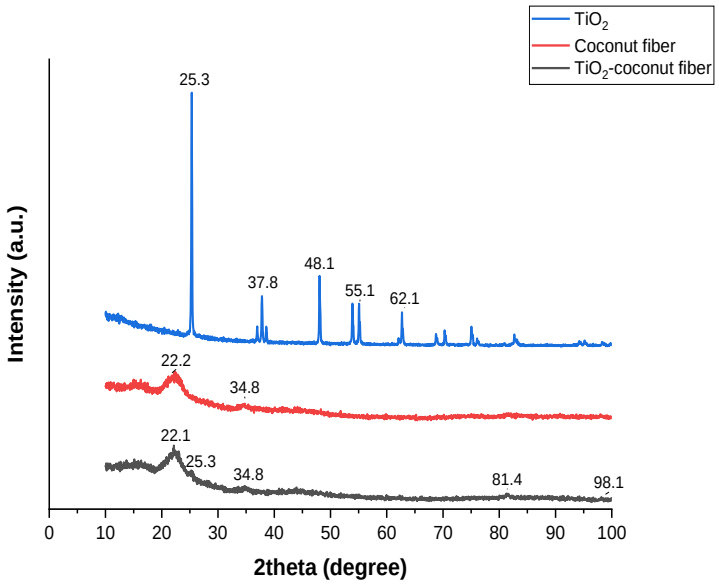

Figure 3. Diffractogram of TiO_2, Coconut Fiber, and TiO_2-Coconut Fiber

TiO_2 filled with coconut fiber has a smaller crystal size of 13.367 nm than Merck's TiO_2, which is 26.733 nm. The difference in the size of TiO_2 crystals before and after being carried with coconut fiber can be seen from the diffraction peaks, which are measured based on the width at half peak height or Full Width at Maximum High (FWHM), where a broad peak will produce a smaller crystal size [22]. The diffraction patterns on TiO_2 and TiO_2-coconut fiber did not change in the anatase crystal phase. The absent of some peaks in TiO_2–coconut fiber is caused by a more dominant substrate covering part of the matrix in TiO_2 crystals [23].

3.2 X-Ray Fluorescence (XRF)

XRF analysis provides information about a sample's percentage of elements and oxides. The results of the XRF analysis are shown by spectral peaks representing the type of element according to its characteristic X-ray energy [24]. This analysis aims to determine the percentage of TiO_2 contained in the synthesized TiO_2-coconut fiber photocatalyst.

Table 1. XRF Analysis of TiO_2-Coconut Fiber

Compounds (oxides)	Content (%)
P_2O_5	24.70
SiO_2	24.69
TiO_2	21.12
CaO	11.48
Al_2O_3	11.47
Fe_2O_3	2.58

Based on the measured data (Table 1), TiO_2-coconut fiber contains essential elements such as Al, Si, P, Ca, and Ti. This analysis's results align with the previous study, which states that the composition of coconut fiber is Si, Al, and Fe [25]. The SiO_2 compound seen in XRF analysis comes from the active groups of silanols and siloxanes in coconut fiber, forming SiO_2 when heated [26]. The content of the anatase TiO_2 compound in its coconut fiber composite was 21.12%. Therefore, it can be concluded that the TiO_2-coconut fiber photocatalyst has been well synthesized due to the significant TiO_2 content in the TiO_2-coconut fiber photocatalyst.

3.3 Diffuse Reflectance-Ultraviolet (DR-UV)

The analysis using DR-UV aims to see the band gap value of the resulting material. Based on the absorbance value to the wavelength of the analysis (Figure 4), the band gap value can be obtained, which is calculated using the Tauc equation:

$$(hva) \cdot \frac{1}{n} = A(hv - E_g) \qquad (3)$$

where h is Planck's constant, v is the frequency with $v = c/\lambda$, a is the absorption coefficient, E_g is the band gap, and A is the proportional constant [27].

The linear equation for TiO_2 used in this study is $y = 6.104x - 19.61$ with $R^2 = 0.994$, and TiO_2-coconut fiber is $y = 3.914x - 12.45$ with $R^2 = 0.975$. The resulting straight line intersection (trendline) gives a band gap value (Figure 4) of Merck's TiO_2 of 3.21 eV with an absorption wavelength of 386.5 nm and TiO_2-coconut fiber of

3.18 eV with an absorption wavelength of 390.3 nm. The resulting TiO_2 is of anatase type because it has a band gap energy range of 3.20–3.30 eV, and its structure has a large photocatalytic activity compared to rutile structure [28]. This result is also supported by a theory that states that the anatase TiO_2 structure that has a band gap value of 3.2 eV, having better photocatalytic activity than the rutile phase [29]. The shift in the energy gap in TiO_2 when it is carried with coconut fiber is caused by a heating process that changes the quantity/native defect slightly so that it gives a new energy level to the TiO_2 structure. This is consistent with the theory which states that temperature affects the level of damage to crystals so that it affects the absorption shift and the gap value of TiO_2 [30].

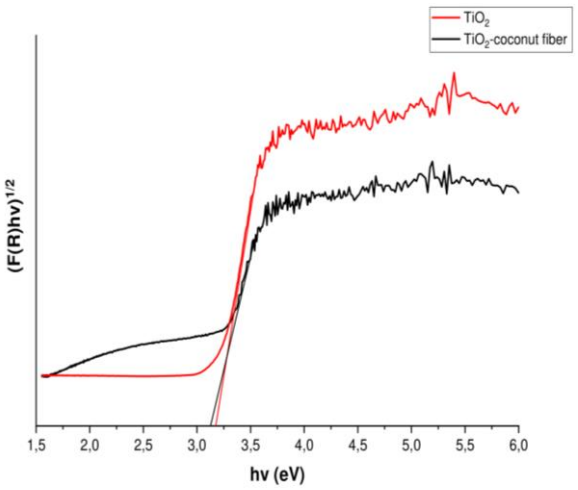

Figure 4. Band Gap of TiO_2 and TiO_2-Coconut Fiber

3.4 Linear Alkylbenzene Sulfonate Degradation Activity Test

The photocatalytic activity of TiO_2-coconut fiber against LAS was carried out in a greenhouse laboratory for 120 minutes with a photocatalyst mass ratio (TiO_2:coconut fiber) of 20:80; 30:70; 40:60 and 50:50 w/w. Absorbance measurements were carried out at the optimum wavelength of LAS, which had previously been measured at 664 nm. The degradation percentage is then plotted in a curve against the photocatalysis time (Figure 6).

Floating photocatalyst TiO_2-coconut fiber with various mass ratios shows decreased LAS content. The mass ratio of TiO_2:coconut fiber 50:50 w/w experienced the lowest decrease in LAS content, namely 76.89% ±1.86% of initial LAS content with 30 minutes of irradiation time. The mass ratio of TiO_2:coconut fiber 20:80 w/w experienced the highest decrease in LAS content, about 80.43%±1.61% of the initial LAS content, with an irradiation time of 120 minutes. The graph illustrates the relationship between irradiation time and variations in a mass ratio of TiO_2: coconut fiber to the decrease in LAS content (Figure 6).

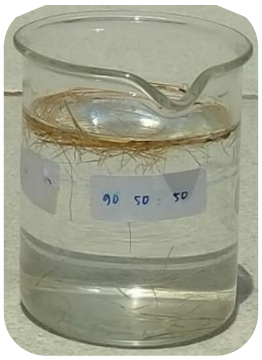

Figure 5. Floating Catalyst TiO_2-Coconut Fiber

The difference in the results of the degradation of the floating photocatalyst with variations in mass is influenced by the density of the catalyst. The greater the density of the catalyst, the faster the floating catalyst will sink to the bottom of the solution, thereby reducing the degradation efficiency at a certain time. The results of density calculations are shown in Table 2.

Table 2. Density of TiO_2, Coconut Fiber, and TiO_2-Coconut Fiber

Sample	Density (g/cm³)
TiO_2	3.890 ± 0.025
Coconut fiber	0.840 ± 0.007
TiO_2-coconut fiber (20:80)	0.789 ± 0.008
TiO_2-coconut fiber (30:70)	0.808 ± 0.009
TiO_2-coconut fiber (40:60)	0.869 ± 0.008
TiO_2-coconut fiber (50:50)	0.873 ± 0.009

TiO_2-coconut fiber with 50:50 w/w variation has the highest degradation activity in the first 30 minutes compared to other variations due to its greater density. However, at 60 to 120 minutes, the degradation activity decreases because the greater the density, the faster the floating catalyst is absorbed by water so that it sinks faster to the bottom of the solution. The greater the density of TiO_2-coconut fiber, the more TiO_2 is attached to the fiber. This result is in accordance with the theory, which states that the greater the volume of the constituent material with the greater the density, the formed catalyst composite has high density and vice versa [31].

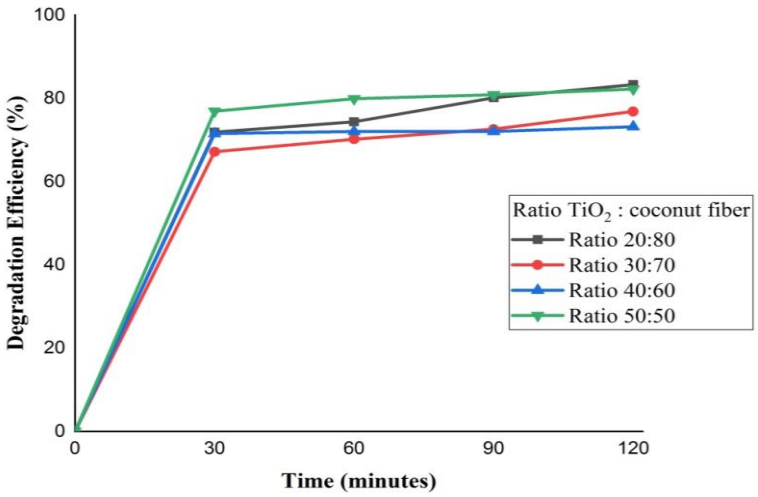

Figure 6. Degradation LAS at Sunlight Irradiation Time

The percentage of LAS content degradation increased in the irradiation time range of 30 to 120 minutes, where the percent degradation of the LAS compound increased along with longer irradiation time. The length of irradiation will increase the amount of OH•, formed as a degrader of the LAS compound. The percentage of LAS compound degradation increased significantly at the initial irradiation time of 30 minutes. In contrast, at 60 to 120 minutes, it tended to be stable, and the percent degradation of LAS compounds did not change much because the amount of OH• produced by the photocatalyst process has been maximized so that the length of time of irradiation will not have a significant effect on the formation of OH•.

The maximum percentage of LAS compound degradation occurred at 120 minutes of irradiation with TiO_2: coconut fiber of 20:80 w/w. Based on the ANOVA test, each variation in mass ratio had a significant difference in the average percentage of degradation. The linearity regression test showed that the length of time of irradiation affected the LAS degradation percentage by 57.4%. In comparison, 42.6% was influenced by other factors, such as the difference in the amount of TiO_2 carried in coconut fiber, showing a difference in the average degradation percentage compared to each variation.

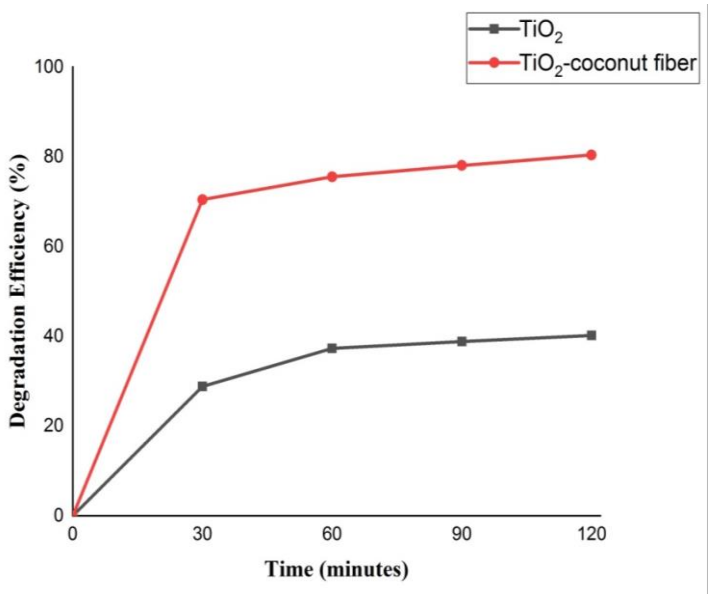

Figure 7. Comparison Curve of Floating Photocatalyst to Control

TiO_2 photocatalyst at the optimum ratio, namely TiO_2-coconut fiber (20:80), compared the activity of the photocatalyst with that of the TiO_2 photocatalyst control (without carrier) which was allowed to sink to the bottom of the solution in degrading LAS. The results obtained (Figure 7) show that there are differences in degradation activity. The TiO_2 catalyst immersed in the bottom of the solution has a degradation percentage of 40.20%, while TiO_2 composited on coconut fiber resulted in a degradation activity of 81.43% within 120 minutes. This difference is due to the different light intensities obtained. TiO_2, which is submerged, will absorb less light than the catalyst carried by coconut fiber. This is due to the differences in the intensity of light penetrating the water, which generally decreases with the water's depth [32].

The mechanism of the TiO_2 floating photocatalyst, namely TiO_2, absorbs photons from sunlight that reach the water surface and result in the excitation of electrons from the valence band to the conduction band if the photon energy is equal to or greater than the band gap value. Simultaneously, electron vacancies (positive charge/H+) also form in the valence band. The positive charge then interact with water and produce •OH. The OH radical reacts with the carbon atom in the LAS benzene ring. LAS undergoes a thermochemical reaction, producing CO_2 and H_2O [33]. In addition, LAS has a sulfonate group (SO) which makes the LAS compound quickly adsorb on the catalyst's surface. So that it can increase the reaction between •OH and C atoms in the LAS benzene ring [34].

4. Conclusion

The band gap of TiO_2 and TiO_2-coconut fiber is 3.21 and 3.18 eV, with light absorption at 386.5 and 390.3 nm. TiO_2 that may stick to coconut fiber when carried is 21.12%. TiO_2-coconut fiber in photocatalyst with a ratio of 20:80 w/w showed optimum photocatalytic activity at 120 minutes with the highest degradation of 80.43%. The less TiO_2 composition allows the catalyst to float longer so light optimization can occur more optimally.

5. Acknowledgments

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