

## Green Synthesis of TiO<sub>2</sub> Using Aloe Vera for Photodegradation in Sasirangan Waste

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### Abstract

Green synthesis of TiO<sub>2</sub> has been successfully carried out using aloe vera. Aloe vera extract was mixed with TiCl<sub>4</sub> while heated to obtain TiO<sub>2</sub> powder. The FTIR spectrum showed a peak at 487.67 cm<sup>-1</sup> corresponding to the Ti-O-Ti functional group, which indicates the formation of TiO<sub>2</sub> particles. The XRD results showed that the TiO<sub>2</sub> crystals were in the anatase phase. SEM images show that the particles tend to be spherical in shape as in the anatase phase. With the Scherer equation, the size of the TiO<sub>2</sub> crystal yielded was around 2.3 nm in diameter. The UV-vis spectrophotometer results showed the blue shift of absorption peak at 368 nm or band gap energy of 3.37 eV. A thin layer of TiO<sub>2</sub> was made by using the slip casting method. These thin films were applied to methylene blue and sasirangan waste samples for photodegradation tests. The exposure times used were 15, 30, 45, 60, and 75 minutes. The results showed photodegradation of 38.9 % and 4.5 %, respectively, for methylene blue and sasirangan waste.



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### Introduction

Sasirangan is the traditional fabric from South Kalimantan which has special colours and patterns [1]. The sasirangan colours are influenced by the type of the dye used. Sasirangan's natural dyes are now being replaced by synthetic dyes that are easier to obtain and produce attractive colours. Synthetic dyes produce dangerous waste which is accountable for water pollution [2]. Sasirangan waste has a concentrated colour and contains hazardous chemicals that are often discharged into the river without any prior waste treatment [3]. In addition, this waste is difficult to decompose, has a pungent odor, and pollutes the environment if disposed of carelessly, so waste treatment is needed to overcome this problem.

There are many methods to treat sasirangan waste, such as membranes [3], coagulation [4], adsorption [5], and a combination of filtration and phytoremediation methods [6]. However, these methods are expensive, difficult to implement, produce sludge, and are less effective against some color wastes. Photocatalyst is one of the simple and economical methods, that is effective for treating waste and parsing organic compounds such as methylene blue [7]. This method is economical and environmentally friendly because it can use UV light or sun in the reaction [8]. Photocatalysts can convert light energy into chemical energy that will produce hydroxyl radicals. Then it interacts redox with pollutants in liquid waste [9]. One of the materials that has a photocatalytic effect is  $\text{TiO}_2$  [10][7][11].  $\text{TiO}_2$  is a white dioxide compound that is a stable and effective catalyst for water and air purification, destroying microorganisms, degrading dyes and toxic chemicals [12].

Titanium dioxide ( $\text{TiO}_2$ ) could be greenly synthesized from natural extracts [13]. Green synthesis utilizes plants to synthesize particles [14] and is superior because it is environmentally friendly, cheap, and energy efficient. Several researchers used aloe vera to synthesize  $\text{TiO}_2$  [15][16][17]. According to Okto [8], aloe vera is a good reducer in the synthesis of  $\text{TiO}_2$  because aloe vera gave smaller  $\text{TiO}_2$  particles size compared to other plants such as lemon (*Citrus limon*), lime (*Citrus limett*), Indian gooseberry (*Phyllanthus Emblica*), pala indigo (*Wrightia tinctoria*), and purging nut (*Jatropha curcas L*). The previous research which used aloe vera in  $\text{TiO}_2$  synthesis were used in several applications such as: drug delivery [18] or catalyst [19].

Thus, in this study,  $\text{TiO}_2$  would be synthesized by using aloe vera and used as a photocatalyst in an attempt to degrade sasirangan waste in Banjarmasin. In Rao's research [15], the neutral pH was used, while in this research, several pH variations (acid, neutral, and base) in synthesizing  $\text{TiO}_2$  would be investigated. Moreover, the produced  $\text{TiO}_2$  will be made into thin films by using the slip-casting method, because the thin films are more suitable for use as catalysts [20]. Therefore, the purpose of this study is to synthesize  $\text{TiO}_2$  by using aloe vera extract, identify the characteristics of  $\text{TiO}_2$  produced, produce  $\text{TiO}_2$  thin film, and the investigate photodegradation of methylene blue and sasirangan waste.

## **Experimental Method**

### **Synthesis of $\text{TiO}_2$**

The aloe vera leaves were cleaned using water. Then the leaves were cut, and the gel was taken out. 100 gram aloe vera gel was mixed with 100 ml distilled water, then heated for 2 hours at  $90^\circ\text{C}$ . The extract was cooled and filtered using whatman filter paper with a pore diameter of 15-20  $\mu\text{m}$ . Next, The aloe vera extract was mixed with 100 mL of 1 N  $\text{TiCl}_4$  solution [15]. Then the 17.5% NaOH was added slowly until it reached acid, neutral, and base conditions while stirring at 600 rpm for 4 hours. Then, the mixed solution was precipitated and filtered. The formed precipitate, which is  $\text{TiO}_2$  powder, was rinsed with distilled water and then dried in an oven for at least 12 hours. The  $\text{TiO}_2$  powder, then, was heated in a furnace at  $500^\circ\text{C}$  for 4 hours. The  $\text{TiO}_2$  powder was characterized using UV-Vis spectrophotometer (LGS 53), FTIR (Bruker), and X-Ray Diffraction (XRD Bruker Eco D8 Advance).

### **The deposition of $\text{TiO}_2$ thin film**

The  $\text{TiO}_2$  films were deposited by the slip casting method because the method is simple and possible in doing in Indonesia. The size of slide glass as substrate is presented in Figure 1.

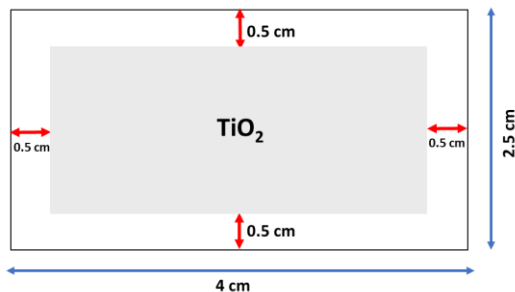


Figure 1. Size of substrate and TiO<sub>2</sub> thin film

The substrate was cleaned with detergent, followed by distilled water and acetone. The substrate's side was given a barrier by attaching a 0.5 cm wide tape. TiO<sub>2</sub> paste is produced through the mixture of TiO<sub>2</sub> powder and 96% ethanol, which was applied on the substrate. TiO<sub>2</sub> paste was allowed to dry. The tape had to be removed before the thin film was calcined for 2 hours at temperature of 650 °C [10]. After the calcination, the thin film was stored in desiccator to cooling down.

Thin films were evaluated by physical tests such as water resistance, moisture content, shape, color, and odor. Water resistance was investigated by immersing the thin film in methylene blue solution for 24 hours to evaluate its resistance to the solution [2]. Moisture content was done by placing the sample in a porcelain cup and heated in the oven at 105°C for 30 minutes. Then, cooling down in a desiccator before the cup was weighed. Moisture content was calculated using the equation [21] below:

$$\% \text{ Moisture content} = \frac{B-C}{B-A} \times 100\% \quad (1)$$

Where:

A: Weight of empty cup (g)

B: Weight of cup + thin film (g)

C: Weight of cup + thin film after drying (g)

### Photocatalyst activity of TiO<sub>2</sub>

The photocatalyst activity was observed by placing the TiO<sub>2</sub> thin film into 50 mL of methylene blue and sasirangan waste solution. The samples were placed in a box sized 35x34x35 cm equipped with a 25-watt Spotlight UV Anti-Bacterial LED lamp, as shown in Figure 2.

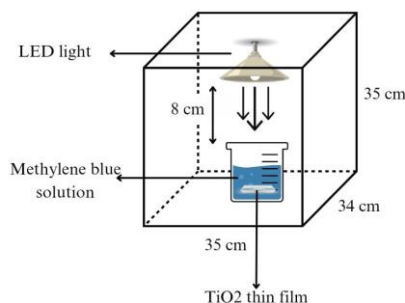


Figure 2. Photocatalyst Reactor in a Box

Then irradiated for 15, 30, 45, 60, and 75 minutes. For each irradiated time, the absorption spectra of methylene blue and sasirangan waste were measured by UV-Vis spectrophotometer to determine their photodegradation. The degradation results were calculated using the Equation (2).

$$\% \text{Degradation} = \frac{C_0 - C_t}{C_0} \times 100\% \quad (2)$$

Using the same photocatalytic activity method, the percentage of degradation by commercial  $\text{TiO}_2$  was observed and compared with the synthesized  $\text{TiO}_2$  to determine the difference in the level of degradation effectiveness. The size of  $\text{TiO}_2$  particles was calculated using the Debye and Scherrer equation as shown in Equation (3) [22].

$$D = \frac{k \lambda}{\cos(\theta) \text{WFHM}} \quad (3)$$

## Result and Discussion

### Synthesis and characterization of $\text{TiO}_2$ particles

Titanium dioxide ( $\text{TiO}_2$ ) was synthesized by using sol-gel method in 3 pH variations (acidic, neutral and base). The weight of  $\text{TiO}_2$  particles obtained is shown in Table 1.

**Table 1. pH variations of  $\text{TiO}_2$  synthesis**

No	pH	$\text{TiO}_2$ results (g)
1	3	0.4451
2	7	0.044
3	9	0.686
4	10	1.4264

From Table 1., it seems that pH of  $\text{TiO}_2$  synthesis affected the weight of  $\text{TiO}_2$  particles obtained. As can be seen, the base condition gave the most of  $\text{TiO}_2$  result in weight. In addition, based on the research of [16] green synthesis of aloe vera with a pH variation of 9 produces a smaller particle size and has the purest crystal phase among other variations.

FTIR spectra describes the relationship of transmittance (%) with wavenumber ( $\text{cm}^{-1}$ ), where the absorption peak indicates the location of the functional group in the infrared region. In this study, FTIR was used to measure the transmission of the aloe vera extract, the mixed solutions and the resulting powders in the range of  $500\text{-}3500 \text{ cm}^{-1}$  to determine the present of  $\text{TiO}_2$ . Figure 3 shows the FTIR spectra of the aloe vera extract, the mixed solutions and the resulting  $\text{TiO}_2$  powders.

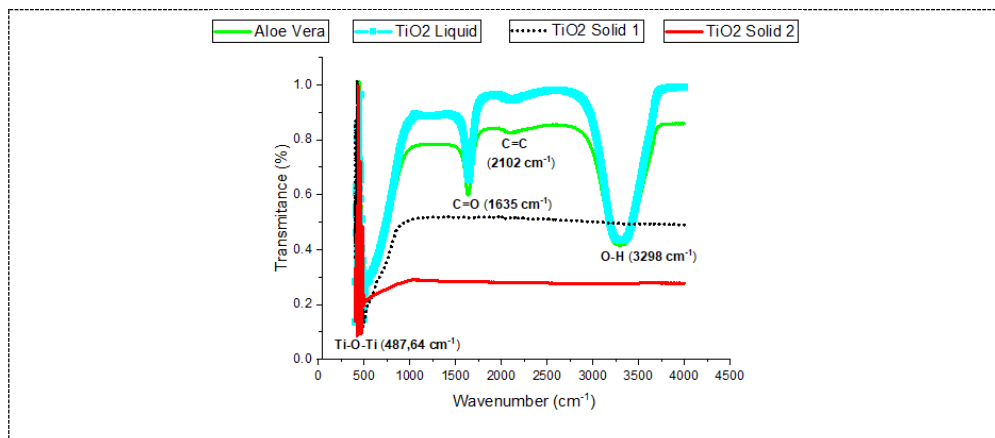


Figure 3. TiO<sub>2</sub> Particle and Aloe Vera FTIR Spectrum

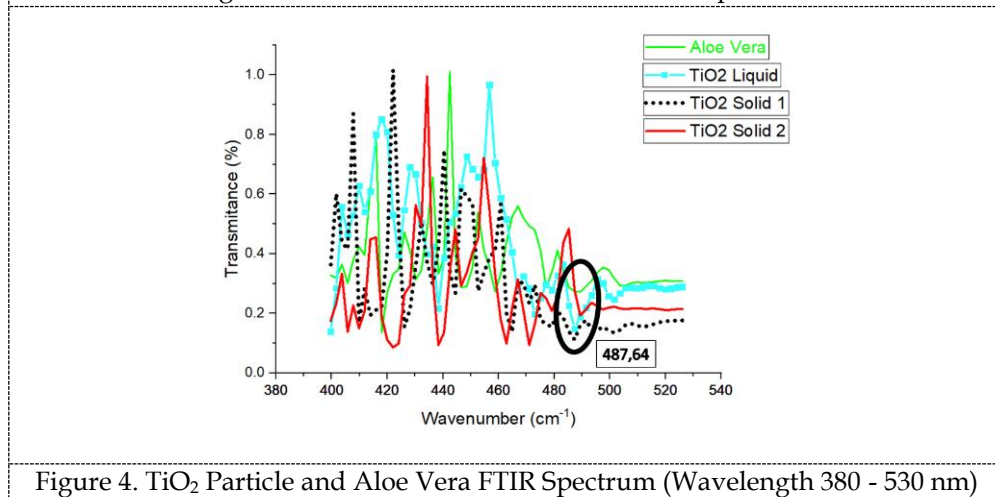


Figure 4. TiO<sub>2</sub> Particle and Aloe Vera FTIR Spectrum (Wavelength 380 - 530 nm)

There is a peak at 3298 cm<sup>-1</sup> which indicating hydroxyl (O-H) functional group and/or phenolic hydroxyl (O-H) functional group vibrations in aloe vera extract [23]. This shows that there is absorption of water molecules as a result of the use of distilled water in the synthesis of aloe vera extract and TiO<sub>2</sub> [24]. At the absorption peak of 1636.45 cm<sup>-1</sup>, there is a C = O group which is a bond of the carbonyl group. This indicates that organic compounds such as phenolics, anthraquinones, flavonoids, and terpenoids found in aloe vera play an important role in stabilizing the process of forming TiO<sub>2</sub> particles [25]. The peak of 2100 cm<sup>-1</sup> indicates the C=C vibration of the aromatic ring by aloe vera [17]. When heated, peaks of organic compounds have been cleaned [26]. Then, the absorption region below 950 cm<sup>-1</sup> reflects the typical vibrational bonding of TiO<sub>2</sub>. The peak at 487.64 cm<sup>-1</sup> shows absorption for Ti-O-Ti bonds, as shown in Figure 4.

According to [27], the band gap energy value plays a key role in the performance of semiconductors, because it determines the energy required to move electrons in the process of excitation and hole. The band gap energy can also be determined based on the particle size, whether nanoparticle or bulk-sized, and is determined based on the shift in the absorption peak [28]. Therefore, the absorption peak of TiO<sub>2</sub> solution was observed by UV-Vis spectrophotometer. The resulting spectra of UV-Vis spectrophotometer was converted to

energy and plotted using Originlab 2018 software (see Figure 5.). The band gap energy of TiO<sub>2</sub> was determined by Gaussian fitting method.

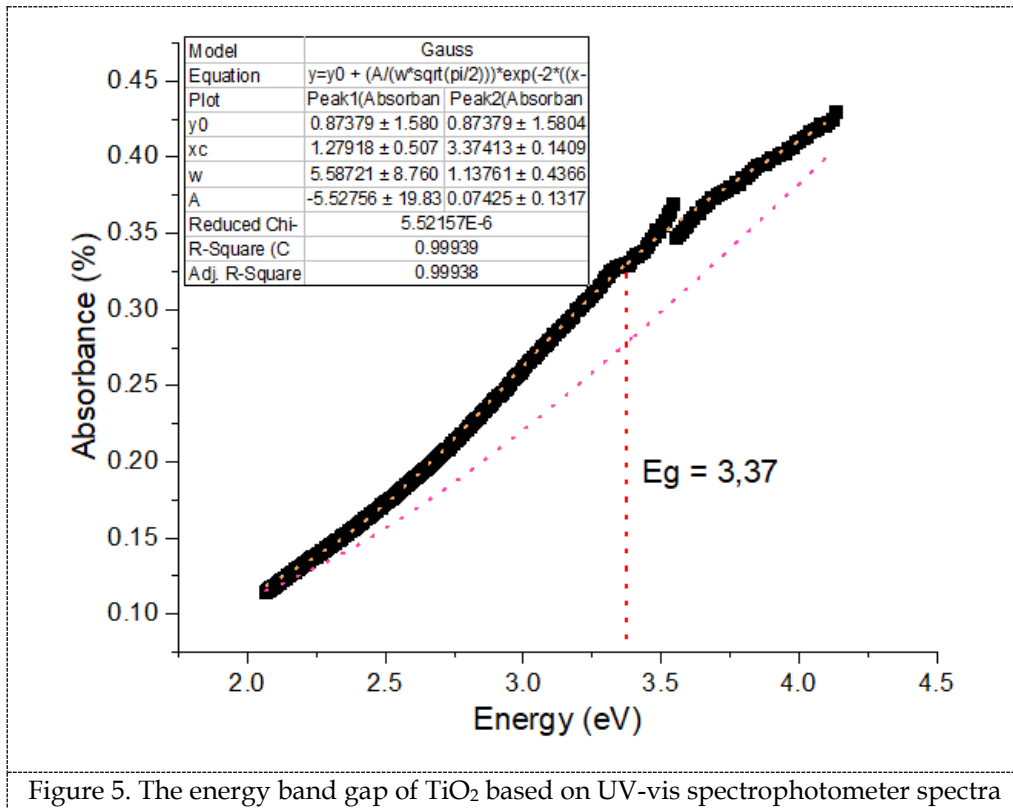


Figure 5. shows that the band gap energy for the variation of TiO<sub>2</sub> solution with pH 10 is 3.37 eV. This is in line with research by [29]. TiO<sub>2</sub> has a band gap energy ranging from 3.2-3.9 eV. After calculating using the Planck equation, the absorption peak of TiO<sub>2</sub> was found to be at 368 nm. This TiO<sub>2</sub> energy gap indicates the particles are in nano-sized. Research by [2] states that the energy gap of TiO<sub>2</sub> would increase with decreasing particle size. The bulk TiO<sub>2</sub> energy gap is about 3.2 eV. This increase is also related to the phase, namely the anatase phase has a band gap greater than the rutile, which has an impact on the effectiveness of photocatalytic greater [30].

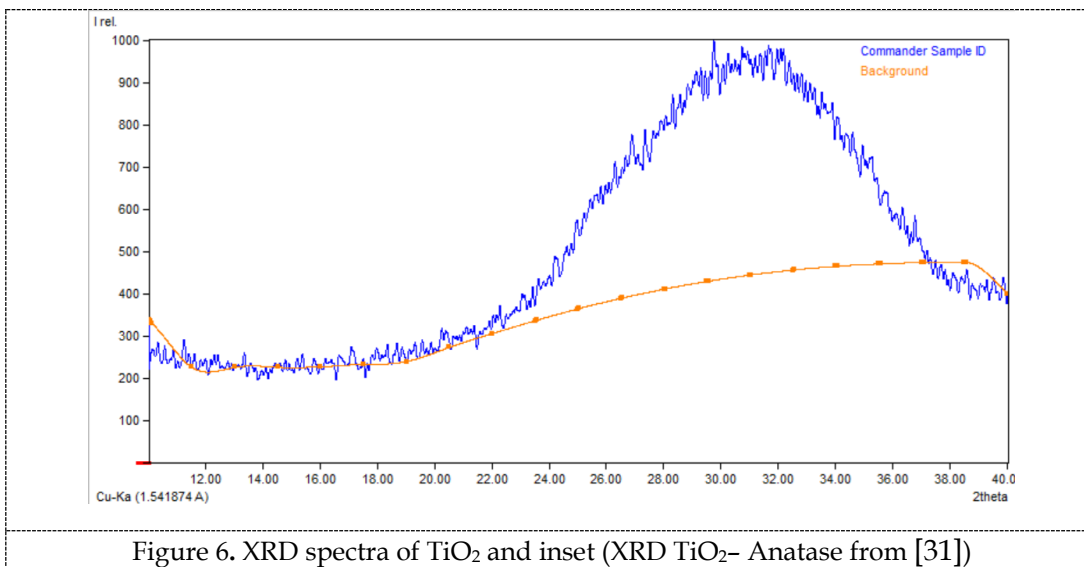
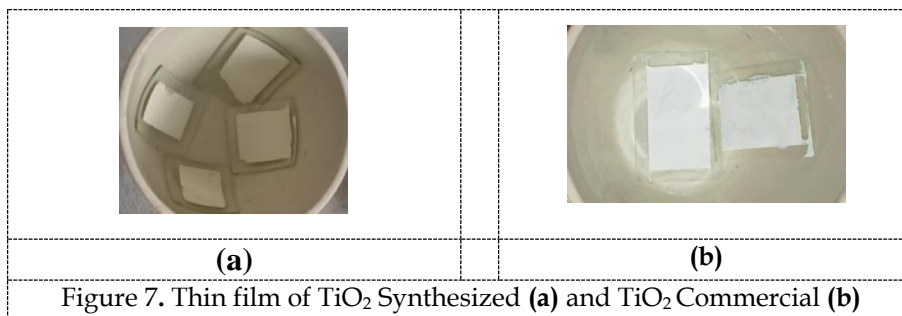


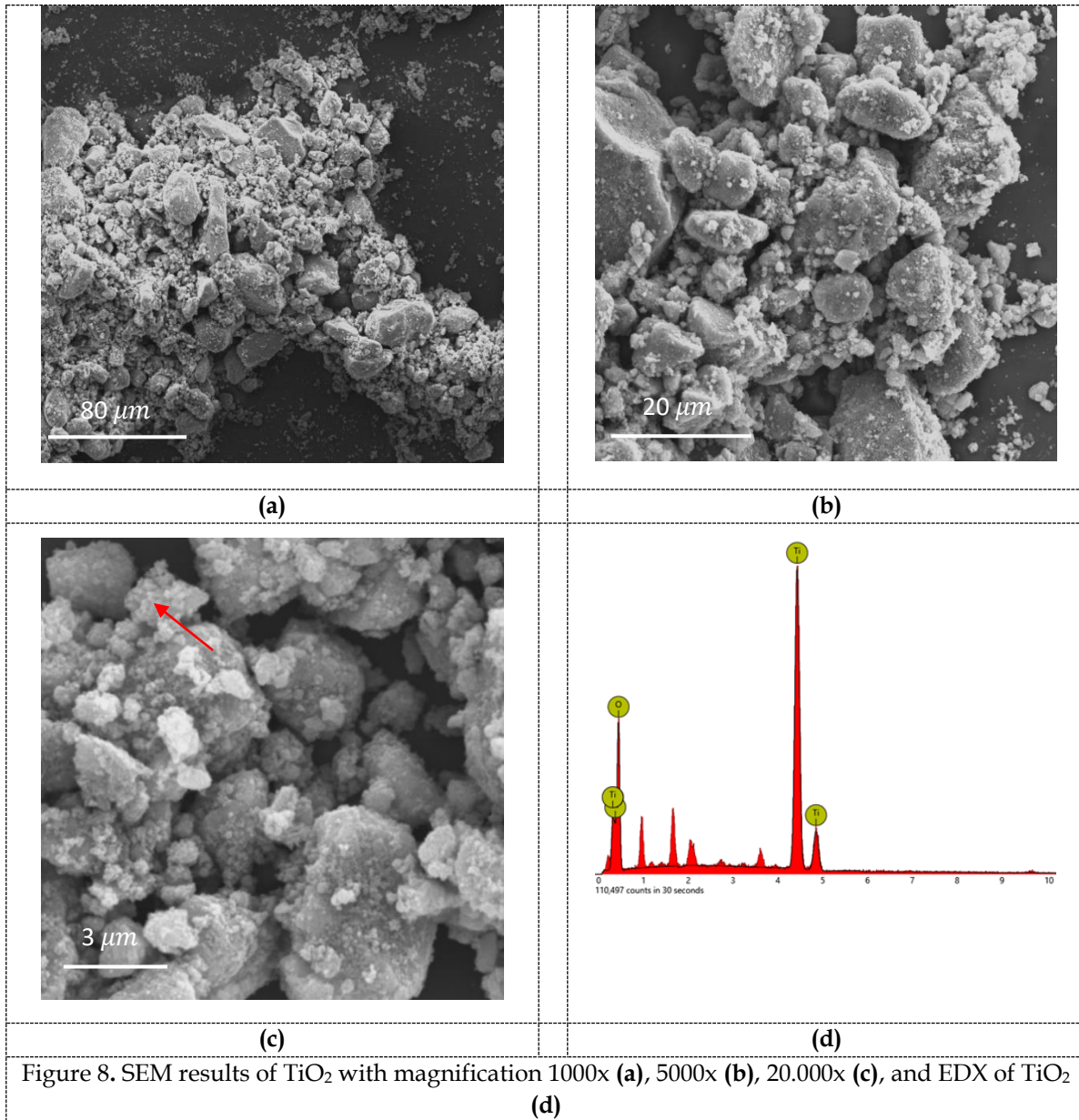
Figure 6. shows that the XRD spectra has a peak around  $2\theta = 30^\circ$ . The inset shows that the anatase phase of TiO<sub>2</sub> appears at  $2\theta = 30^\circ$  as found in research by [31]. This means the TiO<sub>2</sub> obtained in this study was also in the anatase phase (in base condition). This is in line with some research that pH solution would affect the phase that would be formed. Base pH forms the anatase phase, and acidic pH tends to form the rutile phase [32]. The anatase phase on TiO<sub>2</sub> is the most appropriate phase in its application as a photocatalyst [33]. TiO<sub>2</sub> with an anatase phase has advantages such as having a large surface area and smaller particle size. This can support the increase in photocatalyst activity in degrading pollutants and dyes [11]. However, the graph displayed is not sharp, indicating that amorphous particles dominate. This may be due to the addition of aloe vera as an organic material containing complex compounds. The Scherrer equation (Equation (3)) can be used to calculate the particle size of TiO<sub>2</sub> [34], which yielded 2.3 nm in size.

### Deposition of TiO<sub>2</sub> thin films and its characterizations

The thin films of TiO<sub>2</sub> were rectangular in shape, white in color, and had no odor (see Figure 7.). Water resistance and moisture content would affect the photocatalyst process. High water resistance and low moisture content would maintain its structure as an effective, stable and long-lasting photocatalyst. Each thin film of TiO<sub>2</sub> has 100% water resistance (not soluble in water) with moisture content between 0.43% to 0.48%.







Scanning Electron Microscope (SEM Phenom ProX) measurement was conducted to examine the morphology, size, and composition of the TiO<sub>2</sub> samples. According to research by [26], TiO<sub>2</sub> can have a spherical or irregular shape depending on the plant used during the synthesis process. As can be seen in Figure 8., the particles of the synthesized TiO<sub>2</sub> have large irregular chunks (Figure 8. (a) and (b)) and a small portion of particles in spherical shape (Figure 8. (c)). It is assumed that the large irregular chunks are caused by the addition of aloe vera extract to the sample, so that the particle size becomes large due to clumping and aggregate formation. As a stabilizer, the aloe vera extract would experience a decrease in performance due to the presence of particles whose size increases and is not uniform [17]. The large chunks may also be due to particle distribution in the form of a single particle or a group of non-uniform particles. However, after further examination, very small round particles could be seen (Figure 8. (c)), which were identified as TiO<sub>2</sub> nanoparticles with anatase phases that form sponge-like



aggregates [35]. This finding is in line with previous research, which shows that TiO<sub>2</sub> particles in the anatase phase tend to be round. This is in line with the previous result of XRD measurement, which yielded that the TiO<sub>2</sub> sample has a particle size of about 2.3 nm and is in anatase phase.

EDX results indicate the presence of Ti and O elements in TiO<sub>2</sub> thin film samples (Figure 8. (d)). The percentage and atomic weight of Ti and O elements in TiO<sub>2</sub> thin film samples were presented in Table 2.

Table 2. Sample Spectrum TiO<sub>2</sub>

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
22	Ti	Titanium	25.94	51.17
8	O	Oxygen	74.06	48.83

The EDX result shows that the TiO<sub>2</sub> was successfully synthesized. This is confirmed by the results of FTIR and UV-vis spectrophotometer which show the peak formation of TiO<sub>2</sub> and absorption blueshift.

Photocatalyst activity TiO<sub>2</sub>

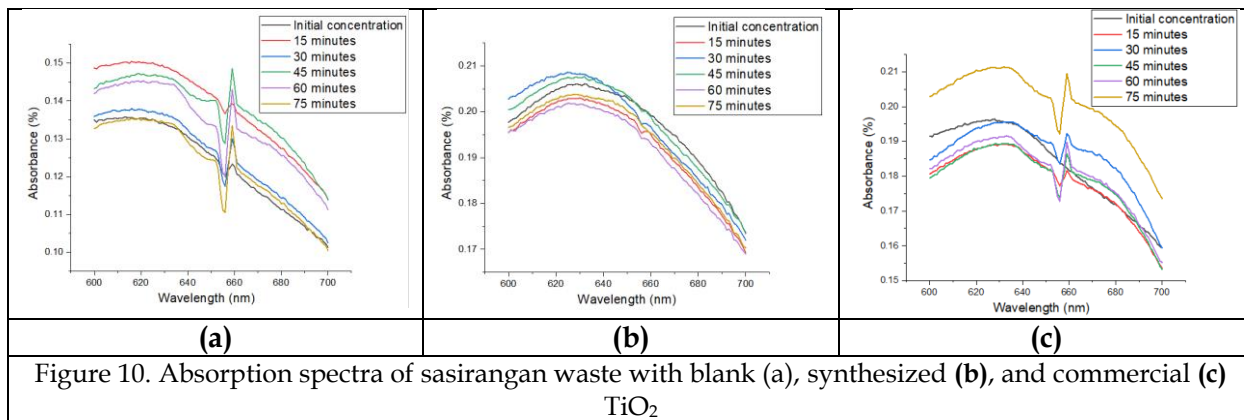
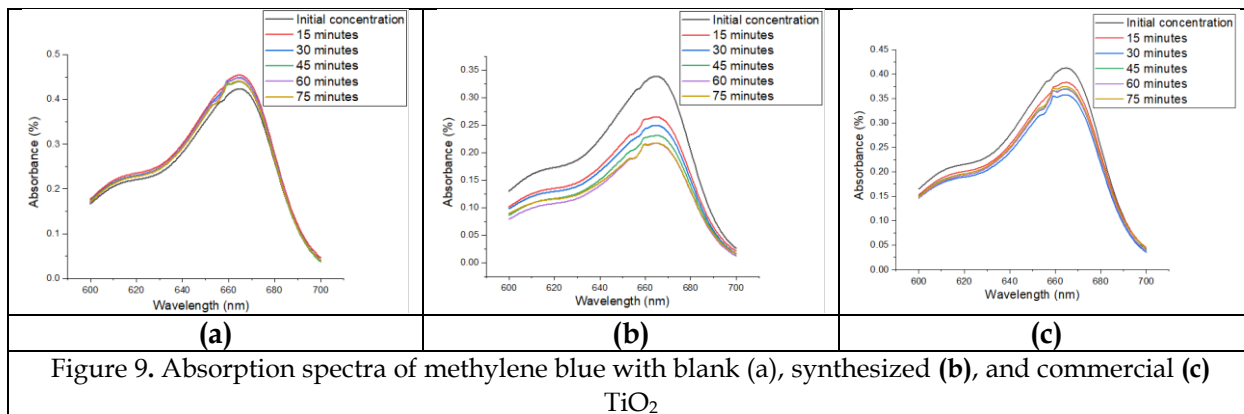


Figure 9. and 10. shows the absorbance peaks at 665 nm (methylene blue) and 656 nm (sasirangan waste). The irradiation of UV light on methylene blue and sasirangan waste

without TiO<sub>2</sub> thin films was shown in Figure 9. (a) and Figure 10 (a). Both of them show an irregular pattern. The absorbance peaks for both methylene blue and sasirangan waste were up and down unpredicted in accordance with the irradiation times. In the presence of TiO<sub>2</sub> films, the absorbance decreased with the length of the irradiation times, indicating the photocatalyst activity of TiO<sub>2</sub>, respectively, for methylene blue (Figure 9. (b)) and sasirangan waste (Figure 10. (b)). The irregular absorbance peaks of methylene blue are also likely due to the concentration of methylene blue (4 ppm) being too low for UV-Vis spectrophotometry device. This is by [36] that UV-Vis spectrophotometry uses lower energy, so it cannot be too accurate for too dilute concentrations. This also shows the superiority of aloe vera in TiO<sub>2</sub> synthesis (stable curve Figure 9. (b)). In sasirangan waste, the irregular adsorption graph is likely caused by the complex waste content. Similar research by [37] with ZnO as a photocatalyst also showed a decrease in absorbance over time. TiO<sub>2</sub> thin films have good and stable photocatalyst activity, allowing electron excitation that causes holes to react with H<sub>2</sub>O to form hydroxyl radicals that degrade organic compounds [38]. We suspect that this is the cause of the pH of the sasirangan waste also changing from 9 to 7 after 24 hours. In order to see much clearer differences in photocatalyst activity, the photodegradation of methylene blue and sasirangan waste were calculated by using Equation (2) based on the decrease in the absorbance curve of UV-Vis spectrophotometer. Furthermore, the photodegradation of the synthesized TiO<sub>2</sub> tended to increase linearly until 75 minutes (see Figure 11.).

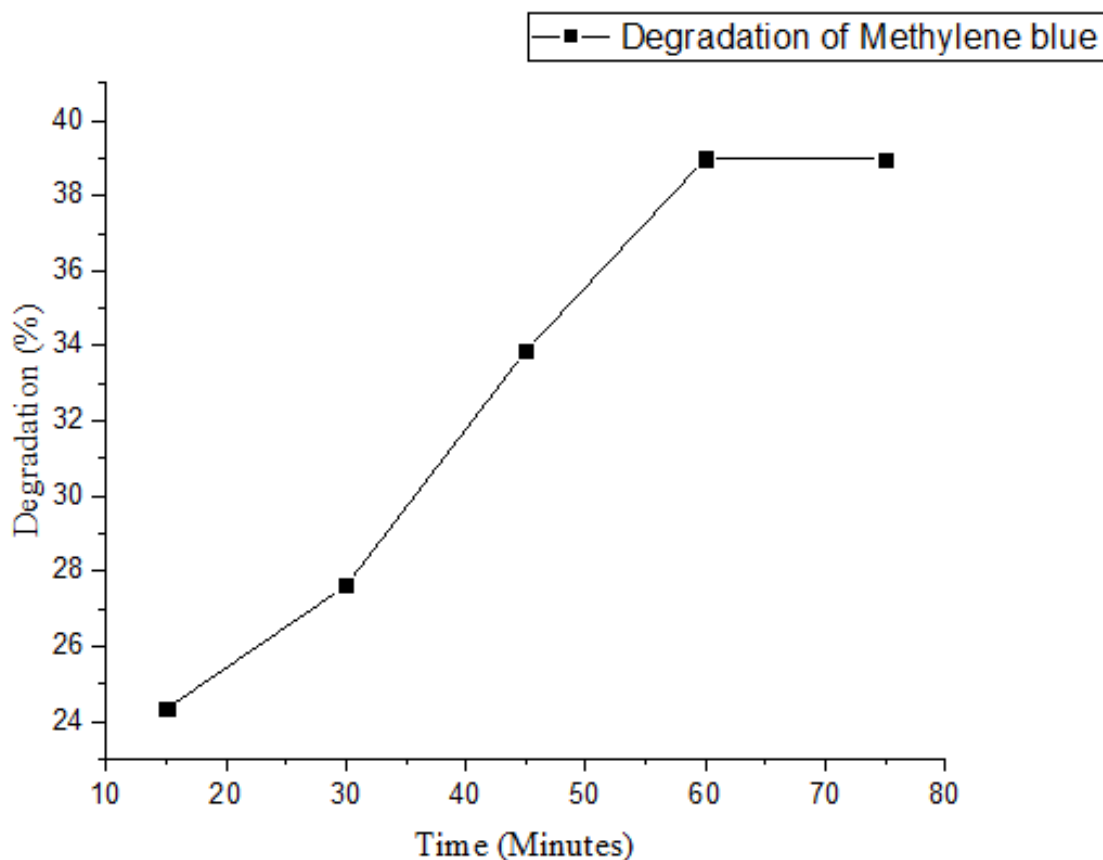


Figure 11. Percentage Degradation of TiO<sub>2</sub>

Therefore, photocatalyst activity of green synthesis TiO<sub>2</sub> using TiCl<sub>4</sub> and aloe vera extract were more effective in degrading methylene blue compared to commercial TiO<sub>2</sub>. This difference may be related to the unique characteristics of green synthesis photocatalysts, such as particle size [39] and the presence of bioactive compounds from aloe vera that can stabilize the TiO<sub>2</sub> particle formation process [40][25].

### Conclusion

TiO<sub>2</sub> has been successfully greenly synthesized using aloe vera. The formation of TiO<sub>2</sub> was confirmed by FTIR measurement, while the results of UV-Vis spectrophotometer showed that the resulting TiO<sub>2</sub> has optical properties suitable for photocatalyst applications. XRD and SEM characterizations show that the use of aloe vera extract did not affect the formation of the desired TiO<sub>2</sub> phase, namely anatase. In base conditions, more TiO<sub>2</sub> was produced than under acidic or neutral conditions. The TiO<sub>2</sub> thin film showed significant photodegradation of 38.9% and 4.5%, respectively, for methylene blue and sasirangan waste. Unfortunately, the photodegradation of sasirangan waste is not directly proportional to the duration of exposure. Further investigation into the components of sasirangan waste is needed to explain the irregular pattern.

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