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Activated Carbon of Coconut Shell Modified TiO_2 as a Batik Waste Treatment

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ABSTRACT

Research about the modification of activated carbon of coconut shell with TiO_2 as a waste treatment Batik has been done. The purpose of this study was to determine the effect of modified TiO_2 on activated carbon characteristics and the effect of TiO_2 concentration on the adsorption power of activated carbon in batik waste. The method utilized was activated carbon soaked in TiO_2 with 5% and 10% concentrations in a ratio of 1: 5, then stirred with a magnetic stirrer for 2 hours. Next, the mixture was placed in an autoclave bottle and an oven (200°C for 30 minutes). The results were then washed with distilled water and dried (100°C for 5 hours), then the material was characterized by FTIR, XRD, SEM-EDX, and application to batik waste. FTIR results indicated the presence of Ti-O-Ti groups after modification at wave number 682 cm^{-1}, XRD indicated the presence of a combination of amorphous KA and crystalline TiO_2 at 25,2°; 37,7°; 48,1°; 53,8°; and 55°, and SEM results of TiO_2 agglomeration on the surface of the railroad. Adsorption of batik waste showed $KATiO_2$-10 (0,052) lower than KA (0,059) and $KATiO_2$-5 (0,057), as well as the presence of COD KA results = 705,6 mg / L (pH = 8), $KATiO_2$-5 = 504,0 mg / L (pH pH = 7) and $KATiO_2$-10= 403,2 mg / L (pH = 7). Based on this research, the activated carbon modified TiO_2 can be used as a material for processing batik waste with the most significant concentration of TiO_2 represent 10%.

1. INTRODUCTION

The development of the batik industry in recent years has been extraordinarily rapid. This is appropriate to an increase in the number of batik enthusiasts both in terms of producers and consumers. However, the problem that arises from this development is the waste generated from batik production process . Batik liquid waste contains dyes, color enhancers, and collections that can cause damage to the aquatic environment (Mukimin et al. 2018). One way to overcome pollution due to batik liquid waste is to use activated carbon as an adsorbent.

Currently, commercial activated carbon has a relatively high selling price According to Global and China Activated Carbon Industry Report 2017-2021 coconut shell-based activated carbon commands the highest price, above USD 1427,98/t. This causes, it is necessary to synthesize activated carbon from materials containing high carbon sources but at a low price like an agricultural waste (Pujiono, et al. 2017; and Mulyati, 2018). One of the agricultural waste that is used as raw material for making activated carbon is coconut shell. The results of Hidayu, et. al. (2016) research shows that with the same activator, the surface area of activated carbon of coconut shell is greater (1.011 m^2 / g) than that of oil palm shells (584 m^2 / g).

The potential of activated carbon from a coconut shell as water treatment has been reported in several studies. Omo-Okoro, et al. (2018) shows that activated carbon from coconut shells can be used as a potent adsorbent for organic wastes and polyfluoroalkyl in aqueous waste. Aljabore, et al. (2017) also reported that activated carbon from coconut shells was effectively used as an adsorbent for toxic textile

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dyes. A similar thing was reported by Cazetta et al. (2011) which shows that activated carbon has an adsorption power of 916 mg/g for dyes. On the other hand, activated carbon can still be increased its adsorption power through modification of the carbon structure. One compound that is usually used as a modifier for activated carbon is TiO_2.

Modification of activated carbon using TiO_2 has several advantages, namely relatively inexpensive, excellent potential economic value to remove pollutants, good availability, inert, excellent photocatalytic activity, relatively excellent chemical stability, effective form of remediation and can prevent the formation of undesirable by-products (Schneider et al. 2014 and Skocaj et al. 2011). Research by Simonetti et al. (2018) showed that activated carbon from coconut shells modified with TiO_2 can be used to increase the adsorbent power of activated carbon to 60.53 m^2/g dye waste using the sol-gel method. MiarAlipour, et al. (2018) determined a combination of TiO_2 photocatalysts which were immobilized in activated carbon to increase the adsorption and photocatalytic efficiency using the in situ method. Research by Pujiono, et al. (2019) showed that activated carbon modified with TiO_2 was able to reduce COD levels up to 93,28%.

Based on the background, a study was conducted on the modification of activated carbon from coconut shells with TiO_2 to adsorb batik liquid waste. Activated carbon synthesized from coconut shell agricultural waste was further modified with TiO_2 at various concentrations and applied to batik waste adsorption.

2. METHODS

2.1. Materials

Materials needed in this study are coconut shell from Bandar Traditional Market, Kediri, East Java; Batik Waste Derived from the batik home industry in Sampang, Madura; Sodium Hydroxide (NaOH) MERCK 99,9%; Titanium Dioxide (TiO_2)Sigma Aldrich 99%; Ferro Ammonium Sulfate ($(NH_4)_2Fe(SO_4)_2{\cdot}6H_2O$) Sigma Aldrich 99%; Ferroin indicator solution 0,1 wt.% in H_2O Sigma Aldrich; Potassium dichromate ($K_2Cr_2O_7$) 99% Sigma Aldrich and distilled water.

2.2. Instrumentation

The instrumentation in this study are glassware, oven for dry sample, furnace, for carbonized sample, magnetic stirrer, pH meter, UV-Vis spectrophotometer (Shimadzu), X-Ray Diffraction (JOEL diffractometer), Brucker FT-IR Spectrometer, Scanning Electron Microscopy (Zeiss EVO MA 10). Digestion vessel, Reflux, and Micro Burette

2.3. Procedure

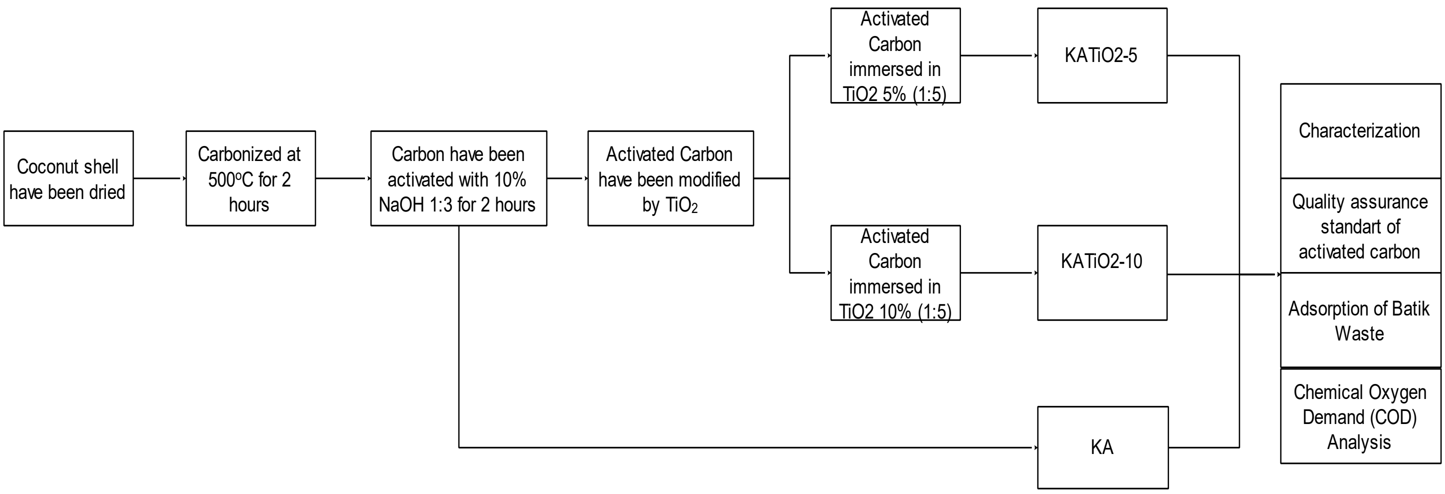

Figure 1. Procedure Synthesis and Characterization of Activated Carbon of Coconut Shell Modified TiO_2 as a Batik Waste Treatment

2.3.1 Sample Preparation

The coconut shell was washed to remove the dirt then dried. The dry coconut shell was then carbonated.

2.3.2 Synthesis of Activated Carbon

The coconut shell was carbonized in the furnace at 500°C for 2 hours. The carbon that has been produced was then ground to fine and sieved to homogenize at 100 mesh. The carbon from the coconut shell that has been produced was then soaked in 10% NaOH in a ratio of 1: 3 for 2 hours. After the stirring process, it was filtered and neutralized with hot distilled water to pH 6.5-7. The precipitate was dried in an oven at 130°C for 4 hours. The result is namely activated carbon (KA).

2.3.3 Modification of Activated Carbon with TiO_2

The activated carbon (KA) was then immersed in TiO_2 (1: 5) with 5% and 10% concentrations, respectively. Then it was added with 45 ml of distilled water and stirred with a magnetic stirrer for 2 hours. Next, the mixture was put in an autoclave bottle and heated at 200°C for 30 minutes. it was then washed with distilled water and dried at 100°C for 5 hours. Activated carbon modified with 5% TiO_2 is called $KATiO_2$-5 while those modified with 10% TiO_2 are called $KATiO_2$-10.

2.3.4 Characterization of Materials

The crystallinity of the material (activated carbon and activated carbon modified with TiO_2) was determined by XRD (X-Ray Diffraction) with CuKα radiation (λ = 1.54056 Å) at 40 kV and 30 mA. The diffractogram pattern is measured at 2θ between 5–50 °. Material functional groups are determined by FTIR (Fourier Transform Infrared) at wavelengths between 400–4000 cm^{-1}. The surface structure of the material is determined by SEM (Scanning Electron Microscope). In addition, the quality assurance standard of activated carbon material before and after the modification of TiO_2 was carried out, such as moisture content, ash content, iodine numbers, and methylene blue numbers.

2.3.5 Application in Batik Waste

Modified activated carbon TiO_2 ($KATiO_2$) is applied as batik wastewater material. it is mixed with batik wastewater (1:5) and stirred with a magnetic stirrer for 2 hours. Then the filtrate was measured using a UV-Vis spectrophotometer (at a wavelength of 400 - 800 nm).

2.3.6 Chemical Oxygen Demand (COD) Analysis

The sample is inserted digestion vessel with a digestion solution ($K_2Cr_2O_7$ 0,1 N) and sulfuric acid reactant. Next, the mixture is shaken until homogeneous. After that, the mixture is refluxed for 2 hours and cooled at room temperature. Then the test solution is titrated with Ferro Ammonium Sulfate (FAS) standard solution and Ferroin indicator until the color changes from bluish-green to reddish-brown. COD results are calculated by the equation :

$$\text{COD (mg/l)} = \frac{((Vb-Vc)\times NFAS\times 8000}{Vs}$$

With :

Vb = volume of FAS solution needed for blank (ml)

Vc = volume of FAS solution needed for the test sample (ml)

NFAS = normality of FAS solution (N)

Vs = sample volume (ml)

3. RESULT AND DISCUSSION

3.1 Activated Carbon Synthesis

In this research, activated carbon is synthesized from the coconut shell with NaOH activator. At first, the carbon is synthesized by heating it in a furnace at 500 ºC. This process aims to release volatile molecules like CO_2 and H_2O (Asrijal, et al. 2014) and describe the components contained in coconut shells, namely cellulose, hemicellulose, and lignin (Hartanto and Ratnawati, 2010). The carbon produced is then activated with a 10% NaOH solution. NaOH activator acts as a dehydrating agent that can break C-O-C and C-O bonds from raw materials, causing a decrease in mass after activation (Bachrun, et al. 2016). The yield of activated carbon produced in this study reached 87.38% (Table 1).

Table 1. Yield of Activated Carbon

Carbon mass (g)	Activated carbon mass (g)	Yield (%)
10,006	8,743	87,38

3.2 Activated Carbon Modified with TiO_2

Activated carbon (KA) was modified with 5% and 10% TiO_2. The variation of TiO_2 concentration was chosen based on the research of Orha, et al (2017) and Pujiono, et al (2019). It aims to determine the effect of TiO_2 concentration on activated carbon characteristics. Modification of activated carbon is produced by stirring activated carbon with a TiO_2 solution for 2 hours to distribute TiO_2 evenly on the entire surface of activated carbon (Yuliusman, 2014). The precipitate was dried at 100 °C to remove water vapor. Modified activated carbon at 5% TiO_2 hereinafter referred to as KATiO_2-5 while modification of 10% TiO_2 as KATiO_2-10.

3.3 Characterization

In this study, the quality assurance standard of activated carbon before and after TiO_2 modification was carried out by comparing data on moisture content, ash content, iodine numbers, and methylene blue numbers with SNI 06-37-30-1995 requirements. Table 2 shows that activated carbon and TiO_2 modified activated carbon have met the permitted activated carbon quality standard (SNI 06-37-30-1995).

Table 2. The quality assurance standard of activated carbon before and after TiO2 modification

Type of Test	Materials			SNI 06-37-30-1995
	KA	KATiO_2-5	KATiO_2-10	
Moisture content (%)	15,5	15,3	15,2	Max 25%
Ash content (%)	5,2	13,2	13,9	Max 15%
Iodine numbers (mg/g)	856,78	951,97	983,71	Min 750 mg/g
Methylene blue numbers (mg/g)	120,82	123,14	124,03	Min 120 mg/g

Table 2 shows that the modification of TiO_2 on activated carbon did not significantly change the moisture content. This is following the research of Anisuzzaman, et al (2015) which shows that there wasn't a significant correlation between moisture content and the ratio of impregnation to activated carbon modification. On the other hand, ash content increases after modification of TiO_2. This is due to TiO_2 is a metal oxide that has a bond between titanium tetravalent ions and oxygen ions so that it is stable at high temperatures (Nasution and Fitri, 2018). Table 2 also shows that activated carbon modified with TiO_2 has greater absorption of iodine and methylene blue when compared to activated carbon without modification of TiO_2. These results indicate the existence of modification of activated carbon with TiO_2 able to increase the ability of adsorption ability of activated carbon in adsorbing adsorbate. This is consistent with the research of Dey, et al., (2011) who reported that modification of carbon fiber with TiO_2 was able to increase the ability of adsorption of methylene blue.

Sample characterization was carried out to show that the activated carbon modified TiO_2 was successfully synthesized. Modification of activated carbon with TiO_2 based on Orha et al. (2017) showed that activated carbon modified with TiO_2 has two adsorption properties, that is physically by activated carbon and chemically by TiO_2 through photocatalytic reactions. It is expected that the activated carbon modification process can increase its adsorption capability. Characterization of the samples conducted in this study was FTIR, XRD, and SEM-EDX. The results of the characterization by FTIR are shown in Figure 2. The peak at wave number 3390 cm^{-1} indicates the hydroxyl group (-OH). The peak is increased in intensity on activated carbon. Because of the reaction of water vapor with free compounds on the activated carbon surface (Wibowo, et al. 2011; Pujiono, et al. 2017). However, the peak decrease occurs in carbon which has been modified with TiO_2. This is appropriate to the role of TiO_2 modifiers that can reduce hydrogen bonds between the surface of activated carbon and water vapor (Orha, et al. 2016).

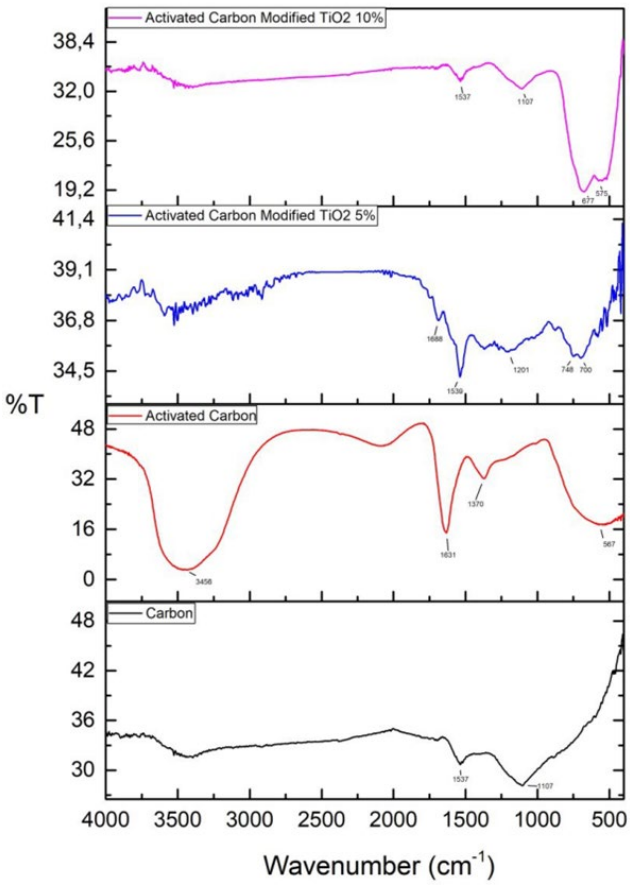

Figure 2. Spectra FTIR of the Materials

The difference between activated carbon before and after activation represents a peak in the wavenumber 1695 cm^{-1}. This peak indicates the stretching of titanium carboxylate (Ti-O-C). Besides that, it shows a peak at wavenumber 1572 cm^{-1} which indicates the presence of Ti-O-C groups. The wavenumbers 1695 cm^{-1} and 1572 cm^{-1} on modified carbon with the between activated carbon and TiO_2 (Irwan et al. 2016). $KATiO_2$-10 peaks at these wavelengths express a more extraordinary intensity than $KATiO_2$-5 which assumed there are more TiO_2 particles attached to activated carbon (Pujiono et. al., 2019). The peak at wave number 682 cm^{-1} indicates the presence of Ti-O-Ti stretching bonds indicating that TiO_2 has covered the activated carbon site.

The difference in a chemical structure on the surface of activated carbon modified TiO_2 equally affects its crystallinity as shown in Figure 3. The diffractogram pattern of activated carbon indicates the presence of widening peaks at 5-10° and 20-30°. This is the characteristic peak of activated carbon which indicates an amorphous form (Le, et al. 2012; Pujiono, et al., 2017; Mulyati, et al. 2017). Besides, the XRD patterns between $KATiO_2$-5 and $KATiO_2$-10 indicate a combination of widening peaks in the 5-10° and 20-30° indicate the presence of activated carbon characteristics and a sharp peak at 25,2°; 37,7°; 48,1°; 53,8°; and 55° which represent the characteristic peak of TiO_2 anatase (Theivasanthi and Alagar, 2013, Pujiono, et al. 2019). The decrease in the intensity of activated carbon modified TiO_2 compared to Anatase because there is an interaction between carbon which is amorphous, thereby reducing the crystallinity of TiO_2. The predominant ananthase in this material provides the advantage of being able to increase its photocatalytic ability when compared to rutile (He et al. 2013). This indicates that there is a combination of amorphous activated carbon forms and TiO_2 crystals which the modification of activated carbon with TiO_2 has been successfully synthesized.

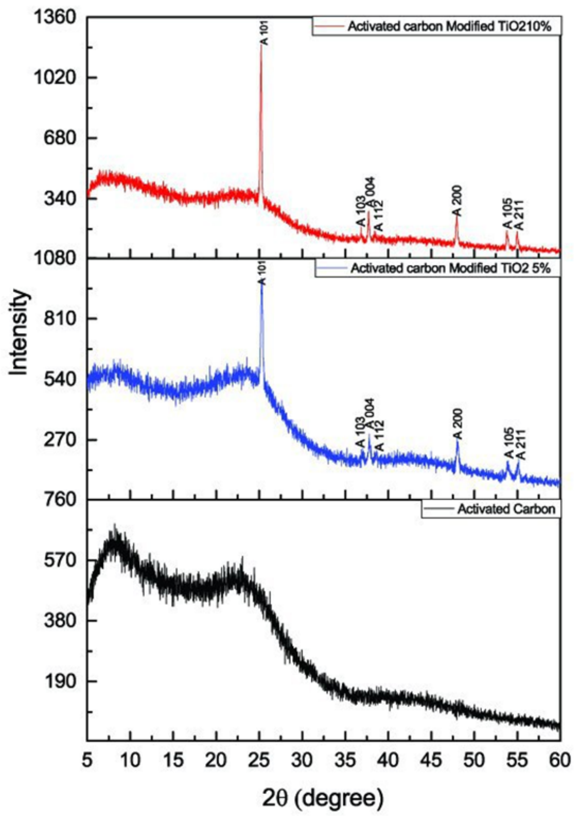

Figure 3. Diffractogram Patterns of The Materials

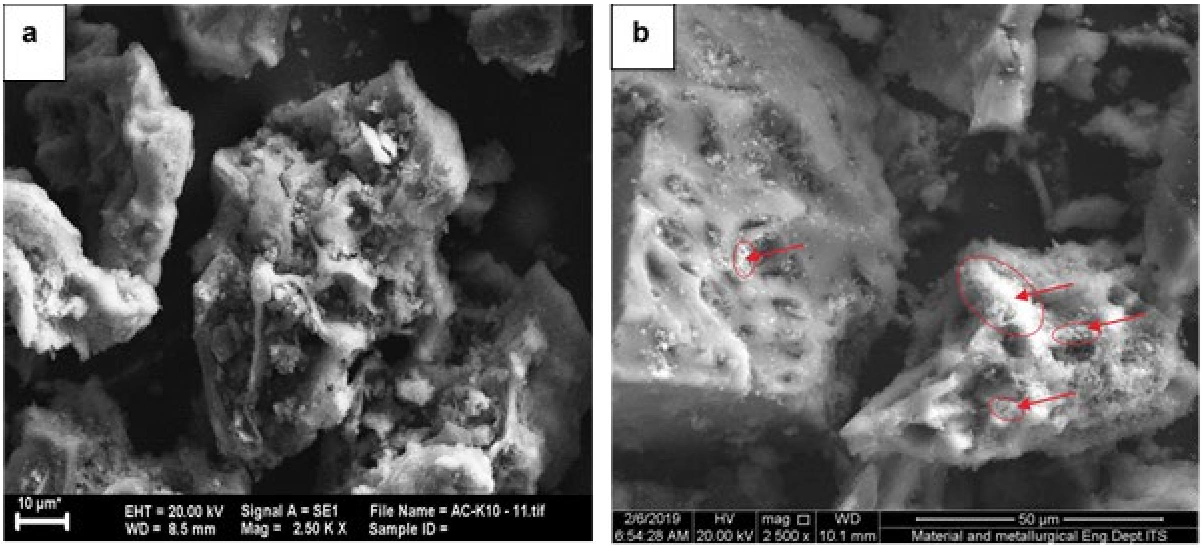

Figure 4. SEM Images of The Materials : (a) Activited Carbon (KA) (b) Activated Carbon Modified by TiO_2 10% (KATiO_2-10)

The morphology of activated carbon and activated carbon modified with TiO_2observed using SEM are shown in Figure 4. Figure 4 (a) shows the surface morphology of the activated carbon, while Figure 4 (b) shows the surface morphology of KATiO_2-10. Based on Figure 4 (a) it can be seen that pores have formed on the surface of activated carbon, this shows that activated carbon has been successfully synthesized. Figure 4 (b) shows that the modified carbon obtains a structure like activated carbon. However, the agglomeration of TiO_2 on the surface of activated carbon indicates the presence of TiO_2 on the surface of activated carbon (Pujiono, et al., 2019). This further proves it has been successfully synthesized.

3.4 Adsorption Batik Waste use Modified Activated Carbon

This modified TiO_2 activated carbon was applied to adsorb batik waste. Batik waste used in this study is Madura batik waste which is yellow and turbid. The waste has a pH = 8 and COD = 1500 mg / L. The results of batik waste adsorption are shown in Table 3. The measurement of batik liquid waste showed a peak at a wavelength of 400 nm with adsorption of 0,536. This is due to the color of the compounds in the batik liquid waste (Rahmawati, et al. 2009). The maximum wavelength of the batik waste solution obtained will be used to measure the absorbance of the waste solution using KA, KATiO_2-5, and KATiO_2-10 materials.

Table 3. Batik Waste Adsorption on Activated Carbon and Activated Carbon Modified with TiO_2

Materials	Absorbance
Batik Waste	0,536
KA	0,059
KATiO_2-5	0,057
KATiO_2-10	0,052

Table 3 shows that there are differences between KA, KATiO_2-5, and KATiO_2-10. KATiO_2-10 obtained lower adsorption that is equal to 0,052 when compared with KA and KATiO_2-5 respectively of 0,059 and 0,057. These results indicate that the adsorption ability obtained. This is because the amount of absorbance indicates the concentration of a dye, if the dye has been adsorbed then the concentration will decrease. This means that the smaller the absorbance, the smaller the concentration of the dye due to adsorbent which shows high adsorption ability (Nsami, et al., 2013). The absorbances obtained prove that TiO_2 not only causes photocatalytic reactions but also increases the adsorption power of activated carbon (Lubis et al. 2016).
This modified TiO_2 activated carbon is then applied to adsorb batik Increasing the number of dyes molecules adsorbed will increase the number of dyes undergoing an oxidation reaction with the formation of OH radicals on the catalyst surface of KATiO_2, then • OH will react with the

dye waste by attacking aromatic rings and the formation of open rings which contribute to the formation of intermediate compounds, which subsequently oxidized to CO_2 and H_2O (Lubis, et al. 2016). The reaction mechanism can be written as follows (Subramani, et al. 2007):

$TiO_2/KA + hv \rightarrow {}^{*}TiO_2 - KA$

$$H_2O + h^+ \xrightarrow{TiO_2^{\bullet} - KA} {}^{*}OH + H$$

${}^{*}OH$ + dyes → intermediates → $CO_2 + H_2O$ + mineral salt

Table 3 also shows that the adsorption of batik waste with activated carbon before and after the modification of TiO2 did not show a significant difference. These results lead to the need for batik COD tests before and after adsorption to determine the ability of the most optimum activated carbon adsorption. COD test aims to determine the number of pollutants that still exist in batik waste. The result of the COD test on batik waste before and after adsorption was shown in Table 4.

Table 4. Batik Waste pH and COD after adsorbed with activated carbon and Activated Carbon Modified with TiO_2

Materials	pH	COD (mg/L)
Batik Waste		1500
KA	8	705,6
$KATiO_2$-5	7	504,0
$KATiO_2$-10	7	403,2

These results are supported by COD (chemical oxygen demand) analysis of batik waste after treatment with activated carbon. Table 4 shows that batik waste treated by KA was 705,6 mg / L (pH = 8). After being processed with $KATiO_2$-5 COD levels were obtained 28,57% and $KATiO_2$-10 increased COD levels by 42,86%. This shows that the presence of TiO_2 is effective in removing dyes from water and TiO_2 is efficient for degrading organic pollutants in liquid waste (Pujiono, et al. 2019). The results of the process of adsorption on batik waste showed that activated carbon modified with TiO_2 can be used as a material for processing batik waste.

4. CONCLUSION

Based on the results of the study showed that the influence of TiO_2concentration on the adsorption power of activated carbon in batik waste, where $KATiO_2$-10 had the lowest adsorption i.e 0,052 compared to KA (0,059) and $KATiO_2$-5 (0,057). The activated carbon modified TiO_2 can be used as a material for processing batik waste with the most significant concentration of TiO_2 represent 10%.

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