



## RESEARCH ARTICLE

# Filtration of Kutawaru Cilacap batik waste using fly ash activated by sulfuric acid

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**OBJECTIVES** The batik industry is one of the largest contributors to liquid waste. Batik liquid waste if not treated properly has the potential to increase disease and pollute the environment. Pollutant levels contained in the waste can be degraded by using fly ash as an adsorbent. Fly ash is obtained from Steam Power Plant waste. The purpose of this study was to determine the best concentration of sulfuric acid between 1M and 3M added to activate fly ash to reduce Chemical Oxygen Demand (COD), Biological Oxygen Demand (BOD), Total Suspended Solid (TSS), color change and pH of Kutawaru batik waste. **METHODS** The research consisted of three stages: the first stage was the activation of fly ash by immersing it in a solution of 1M and 3M sulfuric acid with a ratio of 1:5 for 3 hours. Then, wash with water until the pH is neutral. Furthermore, the fly ash was dried using an oven at 105 °C for 4 hours to a constant weight, resulting in sulfuric acid-activated fly ash. The second stage of the adsorption process, where batik waste was mixed with sulfuric acid-activated fly ash in a ratio of 5:1 for 3 hours, resulted in the waste after adsorption. In the last stage, testing of the waste before and after adsorption was carried out at the Cilacap Environmental Laboratory. **RESULTS** The results showed that the best concentration of sulfuric acid for the activation of fly ash was 1M because it reduced COD, BOD and TSS by up to 90%. **CONCLUSIONS** Changes in COD, BOD, TSS, color and pH of batik waste before and after adsorption using 1 M sulfuric acid-activated fly ash, namely COD 13678 mg/L to 1302 mg/L, BOD 8480 mg/L to 870 mg/L, TSS 460 mg/L becomes 47 mg/L, the color of the batik waste changes from black to yellow, and pH 9 becomes 7.

**KEYWORDS** fly ash; batik liquid waste; activation; adsorption; sulfuric acid

## 1. INTRODUCTION

Industrial activities inevitably produce waste. One of the most produced liquid wastes is from the batik industry. In the batik industry, the process of coloring and procedure of removing the wax in between dyeing colors is an important part of the process of making batik cloth and requires quite a lot of water. The Kutawaru batik industry uses synthetic dyes and natural dyes from mangrove seed coats. The coloring and bleaching processes involve synthetic materials which produce liquid waste and can pollute the environment. The batik industry wastewater produced is large in quantity and produces waste containing dyes, reactive dye residues and chemicals (Niu 2013; Apriyani 2018). Based on the regulation of the Ministry of Environment Number 5 of 2014 concerning wastewater quality standards, it does not specifically regulate quality standard limits for the batik industry, but regulations regarding limits on wastewater that can be discharged into the environment can refer to quality standard limits for the textile industry. Therefore, proper processing is required before being released into the environment. Parameters used to assess organic pollutant content in water bodies include Chemical Oxygen Demand (COD), Biological Oxygen Demand (BOD), Total Suspended Solid (TSS), color and pH. The COD value is a value that shows the total amount of total oxygen needed to chemically oxidize organic matter (Lumaela et al. 2013). BOD is the amount of oxygen needed to oxidize chemical compounds by microorganisms (Chadijah et al. 2013). And TSS is a solid material, both organic and non-organic, suspended in waters (Jiyah et al. 2017). One way to treat liquid waste is by using adsorption. Adsorption is an effective method for removing chemicals from wastewater (Han et al. 2012). Transfer of adsorbent with liquid waste indicates a good adsorption process.

Coal burning in a Steam Power Plant produces waste, one of which is fly ash. Fly ash is in the form of pulverized coal consisting of amorphous glass and several crystalline phases (Goodarzi 2006). Fly ash is fine particles that contain silica (SiO<sub>2</sub>), alumina (Al<sub>2</sub>O<sub>3</sub>), peroxide (Fe<sub>2</sub>O<sub>3</sub>), calcium oxide

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(CaO), as well as other additional elements such as magnesium oxide (MgO), titanium oxide (TiO), alkaline (Na<sub>2</sub>O), K<sub>2</sub>O, and carbon (Mufrodi et al. 2008). The biggest content in fly ash is silica which is equal to 58.75%. This proves that coal fly ash has a high absorption capacity (Blissett and Rowson 2012; Wijaya et al. 2021). One way to treat liquid waste is by using adsorption. Adsorption is an effective method for removing chemicals from wastewater (Han et al. 2012). Transfer of adsorbent with liquid waste indicates a good adsorption process. Therefore, coal fly ash can be used as an adsorbent in waste. Generally, natural materials containing silica such as coal fly ash can be directly used as adsorbents. However, the work of silica or the absorption capacity of fly ash as an adsorbent is not maximized due to the presence of other oxides as other impurities in almost the same amount as silica (Caroles 2019). One way to increase fly ash absorption is to activate it using chemicals. Chemical activation with acid solution on fly ash shows a larger specific surface area and higher pore volume with low energy consumption. Chemical activation is an activation process using chemicals, one of which is sulfuric acid. The surface area of fly ash increases significantly after being modified with sulfuric acid, causing increased adsorption of fine mud particles and improving mud dewatering performance. The strong corrosive capacity of sulfuric acid significantly changes fly ash microspheres (Chen et al. 2010). Chemical activation is an activation process using chemicals, one of which is sulfuric acid. Sulfuric

Activated carbon treated with sulfuric acid can adsorb heavy metals. Sulfuric acid will decompose the metals contained in batik liquid waste during the adsorption process (Lv et al. 2010; Yulianti et al. 2018).

In previous research by Arnesya Ramadhani et al. (2023), fly ash without activation could reduce the COD level of waste by up to 84%. In a previous study by Chen et al. (2010), the best results were obtained from a comparison of liquid waste with activated fly ash 1:5 as much as 4 mol. Based on this background, this study aims to find the optimal conditions for activated fly ash with sulfuric acid concentrations of 1 M and 3 M in reducing Chemical Oxygen Demand (COD), Biological Oxygen Demand (BOD), Total Suspended Solid (TSS) of Kutawaru Cilacap batik waste. It is hoped that this research can reduce water pollution, as well as provide new knowledge to batik industry business actors about how to properly process batik wastewater and together become an environmentally friendly industry.

## 2. RESEARCH METHODOLOGY

### 2.1 Tools and materials

#### 2.1.1 Tools

The tools used in this study consisted of an oven as part of the physical activation process, a 150 mesh sieve, a container, a stirrer, an analytical balance, a pH meter, and a filter cloth. The filter cloth is used after the waste adsorption process.

TABLE 1. Laboratory test results for batik waste samples at cilacap reGENCY environmental service laboratory.

No	Sample	Parameter				
		COD (g/L)	BOD (g/L)	TSS (g/L)	pH	Color
1	A	13678	8480	460	9	Black
2	B	1302	870	47	7	Yellow
3	C	1841	1225	30	7	Yellow

Information:

A = Batik waste without adsorbs (initial sample)

B = Batik waste adsorption of 1M sulfuric acid-activated fly ash

C = batik waste adsorption of fly ash activated by 3 M sulfuric acid

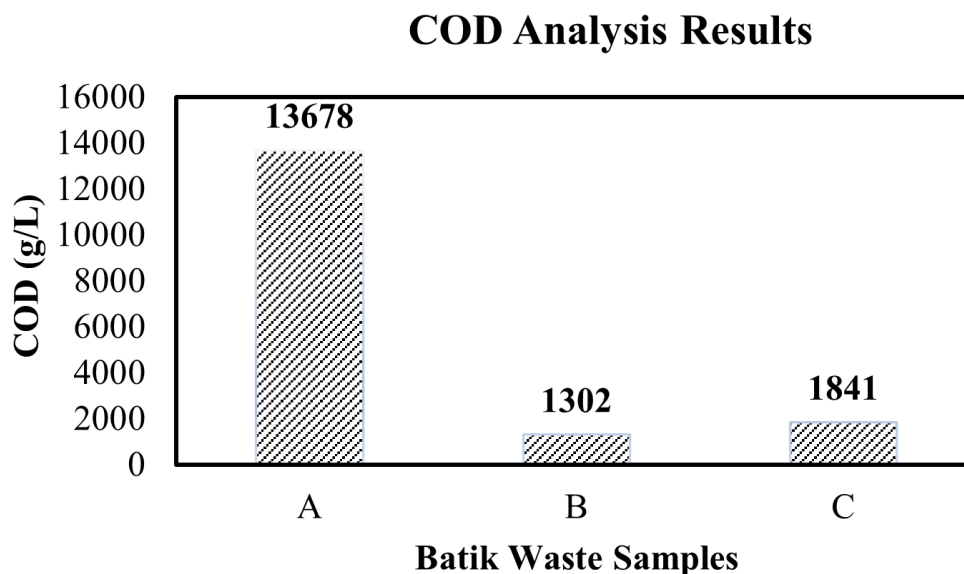


FIGURE 1. Graph COD parameter analysis results.

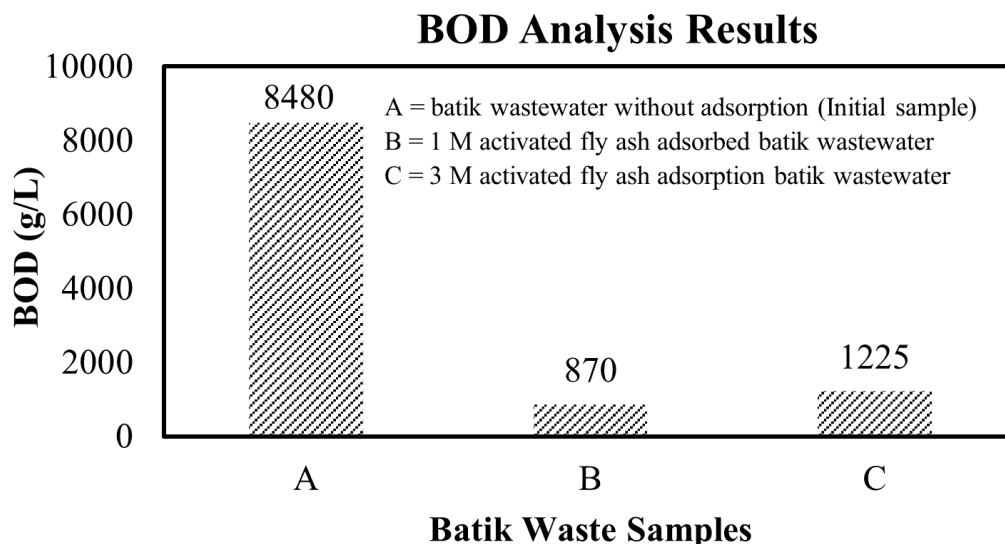


FIGURE 2. Graph BOD parameter analysis results.

### 2.1.2 Materials

The main material used is fly ash activated with sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) obtained from a chemical supply store with concentrations of 1 M and 3 M and other supporting materials, namely distilled water. Aquades is an ingredient for washing fly ash after it has been activated and litmus paper to measure pH.

### 2.2 Procedures

#### 2.2.1 Fly ash activation

Activation is a change in the surface area of carbon which becomes larger because the pores are released (Danarto and Samun 2008). Fly ash is activated with sulfuric acid solution. The fly ash activation process is carried out by soaking fly ash in sulfuric acid solution with a concentration of 1 M and 3 M with a ratio of 1:5 for 3 hours. Chen et al. (2010) research shows that the best ratio of fly ash to sulfuric acid solution is 1:5 compared to a ratio of 1:1 and 1:3 and the best soaking

time is 3 hours (Chen et al., 2010). Then, the washing process uses distilled water until the pH is neutral. Furthermore, the fly ash was dried using an oven at 105 °C for 4 hours to a constant weight, and sulfuric acid-activated fly ash was produced. Concentration solutions of 1 M and 3 M were prepared by mixing sulfuric acid with distilled water. Concentrations of 1 M and 3 M require sulfuric acid of 98 gr/L and 294 gr/L with 1 L of distilled water for a volume of 1 L. The formula for calculating followed the Equation (1) (Setiawati et al. 2022):

$$M = \frac{\text{Molecular mass}}{Mr} \times \frac{1000}{V} \tag{1}$$

#### 2.2.2 Adsorption of batik waste

The adsorption process of batik waste is batik wastewater mixed with sulfuric acid-activated fly ash with a ratio of 5:1 for 3 hours. Adsorption is a mass transfer process that involves the accumulation of substances at a two-phase inter-

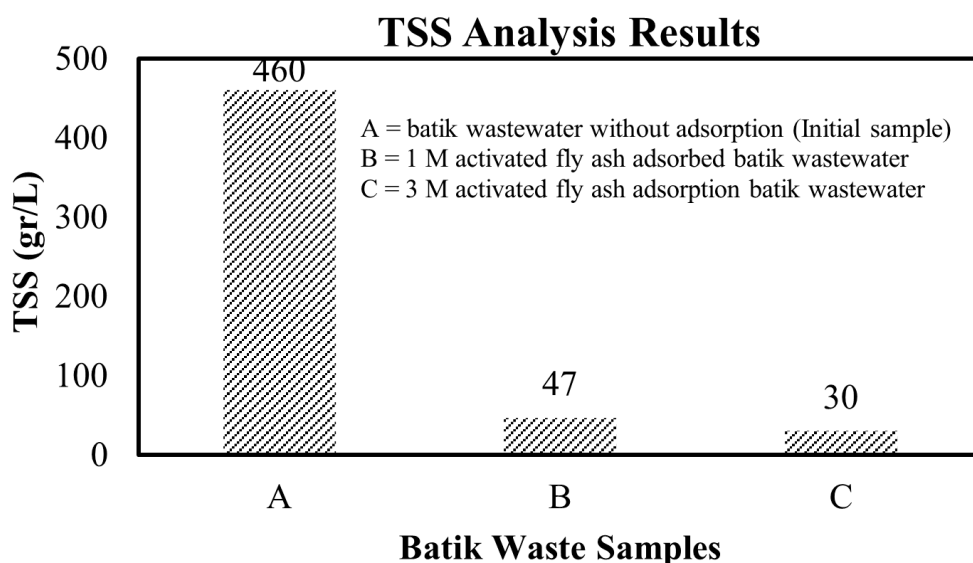
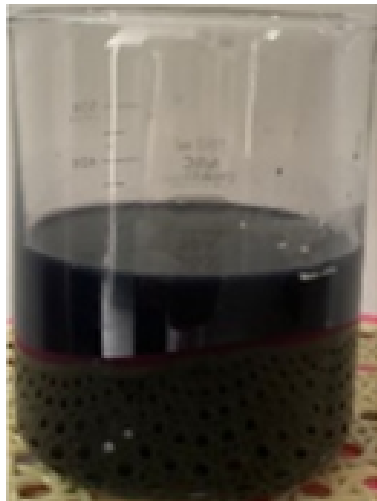


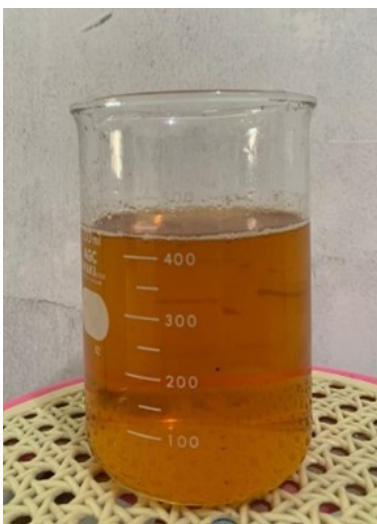
FIGURE 3. Graph TSS parameter analysis results.



(a)



(b)



(c)

FIGURE 4. (Results of pH and color analysis a) Sample A image, (b) Sample B image, and (c) Sample C image.

face, such as a liquid-liquid, gas-liquid, gas-solid, or liquid-solid interface (Konig-Péter et al. 2014). After 3 hours of immersion, the results of the adsorption process waste were obtained. The waste is then brought to the Cilacap Regency Environmental Service laboratory for the sample testing stage.

### 3. RESULTS AND DISCUSSION

Fly ash was activated with concentrations of 1M and 3M sulfuric acid. The activation process is carried out chemically, namely by mixing sulfuric acid into fly ash. After the activated fly ash is produced, the next step is the adsorb process where the batik liquid waste is mixed with activated fly ash in a ratio of 5:1 for 3 hours. The samples were tested in the Cilacap Regency Environment Service laboratory and the results are shown in Table 1 laboratory test results of batik waste samples.

The results of laboratory tests on sample A had a COD content of 13678 g/L, BOD 8480 g/L, TSS 460 g/L, pH 9 and black batik waste. Sample A is a sample of batik wastewater before the adsorption process is carried out. Sample B is batik wastewater adsorption of fly ash activated by 1M sulfuric acid. Sample B showed a COD content of 1302 g/L, BOD of 870 g/L, TSS of 47 g/L, had a pH of 7 and the resulting waste was yellow. While sample C, namely batik waste adsorbed fly ash activated by 3 M sulfuric acid, the results showed a COD content of 1841g/L, BOD of 1225 g/L, TSS of 30 g/L, pH 7 and the resulting waste was yellow.

#### 3.1 COD analysis

Figure 1 shows the test results based on the COD parameter. The smaller the COD test result, the better the fly ash adsorption result. The best results were obtained in sample B, namely at a concentration of 1 M sulfuric acid, and a COD of 1302 g/L was obtained. The result of a low COD content indicates low organic matter so that the quality of the Kutawari batik wastewater is getting better or cleaner so that it is safe to dispose of into the environment (Yuniarti et al. 2019). Fly ash activated by 1M sulfuric acid is very effective in reducing the COD content and the largest content in fly ash is silica of 58.75%. This proves that fly ash has a high absorption capacity so it can reduce the COD content (Blissett and Rowson 2012; Wijaya et al. 2021).

#### 3.2 BOD analysis

The smaller the BOD test result, the better the fly ash adsorption result. The results of the BOD analysis are shown in Figure 2 the best results of the BOD analysis were seen in sample B with a value of 870 g/L because sample B produced a small BOD content. The smaller the BOD value, the better the wastewater quality compared to sample C which produced a BOD content of 1225 gr/L. With low BOD results, this shows that not too much oxygen is needed to degrade waste and the quality of wastewater is getting better (Doraja et al. 2012). Fly ash activated by 1M sulfuric acid is very effective in reducing the BOD content and the largest content in fly ash is silica of 58.75%. This proves that fly ash has a high absorption capacity so it can reduce the BOD content (Blissett and Rowson 2012; Wijaya et al. 2021).

#### 3.3 TSS analysis

The smaller the TSS test result, the better the fly ash adsorption result. The results of the Total Suspended Solid (TSS) test are shown in Figure 3 the best results were obtained in sample C of 30 g/L. Low TSS can reduce barriers to light penetration into the water so that the microorganisms or biota in the waters are not polluted and are clearer (Ma'arif and Hidayah

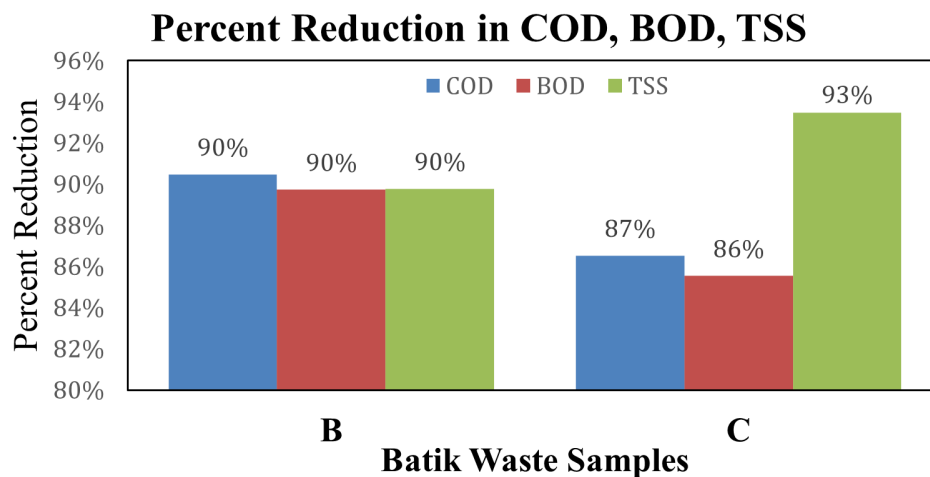


FIGURE 5. Graph percent reduction in COD, BOD, TSS.

2020). The lower the TSS level, the less the amount of impurity or pollutant load. The maximum TSS value was found in batik wastewater, namely 2693 mg/L (Erwindo 2019). TSS results in samples B and C, both fall into the small category.

### 3.4 pH and color analysis

One of the criteria markers to determine water quality is the degree of acidity (pH). Hydrogen potential was measured using litmus paper. Figure 4 shows the best pH results in samples B and C. The litmus paper changed color and was then compared to the color size in the pH measurement guide with litmus paper. Basically, good water is water with pH 7. According to the Decree of the Minister of Environment No. Kep-03/MNKLH/II/1991 1 February 1991 stipulated that factory wastewater may be discharged into rivers or the environment if the pH of the wastewater is in the range of 6 to 9 (Menteri Negara Kependudukan Dan Lingkungan Hidup 1991). The pH of the water should be neutral (7) (Sulistia and Septisya 2020). The results of the sample pH indicate that batik liquid waste with a pH of 7 is safe to be released into the environment.

Physical assessment of water quality can be seen through color. The clearer the color of the water, the better the quality. As can be seen in the picture above, the waste liquid that has the clearest color is in picture b, namely sample B. Sample B has a clear yellow color compared to sample A which is black. Even though sample C is also yellow, it is clearer in sample B. This indicates that sample B is of better quality than the other sample colors. However, this is inversely related to the results of the TSS parameters in Figure 3 where the TSS results in sample C are smaller than those in sample B. This is due to the human error factor where during the TSS testing there is a possibility that dirt will be included in sample B.

### 3.5 Best result analysis

Based on the description of the results and discussion above, the best adsorbent results were obtained, namely sample B. Sample B was a sample resulting from adsorption with sulfuric acid-activated fly ash with a concentration of 1M. Where the COD value is 1302 g/L, BOD is 870 g/L, TSS is 47 g/L, has a pH of 7 and is yellow.

It can be seen in Figure 5 the reduction in COD content in sample B from the initial sample was 90% greater than the reduction in COD content in sample C from the initial sample, which was 86%. The reduction in the BOD content in sample B from the initial sample was 90%, which was also greater than the reduction in the BOD content in sample C from the initial sample, which was 85%. The reduction in the TSS content in sample B from the initial sample was 90%. Even though the TSS content of sample C was better than sample B, among the 5 parameters tested, sample B had the best results and met environmental quality standards.

## 4. CONCLUSION

The batik industry produces batik waste in the form of liquid, solid and gas waste which is a by-product of the series of processes carried out. The processes in the batik industry that produce the most liquid waste are the coloring and procedure of removing the wax in between dyeing colors processes. The large amount of water used in the process also produces a large amount of liquid waste. Based on the results of laboratory tests for the organic content of Kutawaru batik liquid waste from adsorption results with activated fly ash, it meets environmental quality standards. The best adsorption was produced from sulfuric acid-activated fly ash with a concentration of 1M. Processing using fly ash adsorbent which has been activated with sulfuric acid can be an alternative for batik wastewater treatment.

## 5. ACKNOWLEDGEMENTS

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