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# OPTIMIZATION OF COFFEE GROUNDS AND FLY ASH BASED ADSORBENT COMPOSITION WITH PHOSPHORIC ACID ACTIVATOR FOR TOFU INDUSTRY LIQUID WASTE PURIFICATION

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## ABSTRACT

The direct disposal of tofu industrial wastewater without treatment can have negative impacts on the environment due to its organic content. One effort that can be done is to process tofu liquid waste using the adsorption method. Nowadays, coffee shops are growing rapidly in indonesia, resulting in a lot of coffee grounds waste which can be used as raw material for activated charcoal because of its carbon content. In addition to coffee grounds, other waste in the form of fly ash is also used as an adsorbent because of its silica content. This study aims to determine the optimization of the composition of coffee grounds and fly ash adsorbents in the purification of tofu industry liquid waste and to determine the optimum contact time so that liquid waste is obtained in accordance with quality standards. Observation parameters include acidity (ph) and chemical oxygen demand (cod). Coffee grounds charcoal is activated using 0.1 n $h_3po_4$ solution then mixed with fly ash in a ratio of 20:80; 30:70; 50:50; 70:30; and 80:20. The adsorption process is carried out in batches for 60, 90, and 120 minutes. The results of the study showed the optimum composition of the adsorbent 20:80 and contact time 120 minutes with a final cod of 2,761 mg/l, ph 6.21, and a cod reduction of 80.46%.

***Keyword:*** Adsorption, Coffee Grounds, COD, Fly Ash, Tofu wastewater.

## 1. INTRODUCTION

The tofu industry, which has developed rapidly in Indonesia, produces liquid and solid waste that triggers environmental pollution due to direct waste disposal into rivers. Liquid tofu waste is rich in organic materials such as protein and carbohydrates which are the main sources of pollution and cause a decrease in oxygen levels in the waters and increase the levels of dissolved solids. The organic content in liquid tofu waste ranges from 40–60%. Therefore, efforts are needed to reduce oxygen levels in liquid tofu industry waste.

One of the waste treatment efforts is the adsorption method. Adsorption is a process that occurs when a fluid, liquid or gas, is bound to a solid or liquid (absorbent, adsorbent) and eventually forms a thin layer or film (absorbed substance, adsorbate) on its surface [1]. Activated charcoal is one of the adsorbents that is widely used in the adsorption process because it has a large surface area and pores and contains 85-95% carbon produced from carbon-containing materials with a heating process at high temperatures [2]**.** One source of carbon can be obtained from coffee grounds which are waste from the beverage industry, especially from coffee shops which are currently growing rapidly in Indonesia.

In addition to coffee grounds, fly ash can also be used as an adsorbent. Relatively large fly ash waste can cause quite dangerous pollution impacts. The use of fly ash as an adsorbent is a good alternative because this adsorbent can be used in the removal of heavy metals and organic compounds in waste

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processing. Activation of activated charcoal can increase the adsorption capacity and increase the effectiveness of adsorption.

Based on the research results of [3] it was shown that both coffee grounds and fly ash used as adsorbents in the process of purifying nh3 concentration in urea liquid waste were able to provide good absorption efficiency, namely 84.64% and 75.16%, respectively. Thus, this study presents an innovation by combining coffee grounds and fly ash as absorbents to purify tofu liquid waste.

## 2. RESEARCH METHODOLOGY

### 2.1 Location

This research was conducted at the Industrial Waste Treatment Laboratory, Department of Chemical Engineering, Politeknik Negeri Bandung.

### 2.2 Tools and Materials

The equipment used in this study included an oven, furnace, crucible, desiccator, sieve shaker, analytical balance, chemical glassware, jar test, filter paper, funnel, Erlenmeyer flask, burette, stand, pH meter, measuring flask, measuring pipette, dropper pipette, suction bulb, Hach tube, and COD Digester. Meanwhile, the materials used were coffee grounds, fly ash, phosphoric acid, industrial tofu wastewater, potassium dichromate, concentrated sulfuric acid, mercury (II) sulfate, silver sulfate, ferrous ammonium sulfate, and ferroin indicator.

### 2.3 Research Stages

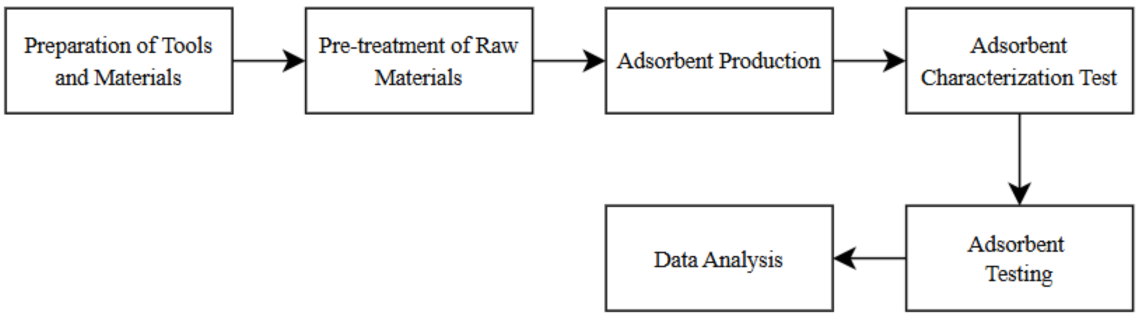


**Figure 1.** Outline of Research Stages

#### 2.3.1 Pre-treatment of Raw Materials

The coffee grounds were cleaned, then dried under sunlight for 24 hours. Subsequently, 500 grams were dried again using an oven at a temperature of 105–110°C for 30 minutes. The coffee grounds were then placed in a desiccator and weighed.

#### 2.3.2 Adsorbent Preparation

The dried coffee grounds were then carbonized using a furnace at a temperature of 600°C for 75 minutes, cooled to 25°C, and sieved using a sieve shaker at a size of 125 μm. The activated carbon from the coffee grounds is activated by soaking it in a 0.1 M $H_3PO_4$ solution for 48 hours, then filtered and washed with distilled water until a neutral pH is achieved.

#### 2.3.3 Adsorbent Characteristic Test

The characteristic test was carried out by analyzing the functional groups using a Fourier Transformed Infrared (FTIR) instrument and analyzing the surface image of the adsorbent using a Scanning Electron Microscope (SEM) instrument. In addition, moisture content and ash content tests were also carried out. The moisture content test was performed by placing 2 grams of activated carbon in a porcelain dish into an oven at 105°C for 3 hours, then cooling and weighing it. The ash content test was performed by placing 1 gram of activated carbon in a porcelain dish into a furnace at 600°C for 2 hours, then cooling and weighing it.

#### 2.3.4 Adsorpting Testing

Activated carbon and fly ash were mixed in varying compositions of 20:80; 30:70; 50:50; 70:30; and 80:20. The mixture was then added to a glass beaker containing 50 mL of soybean waste liquid and stirred at a speed of 150 rpm. The adsorption process was conducted in batches for 60 minutes, 90 minutes, and 120 minutes.

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## 3. ANALYSIS AND RESULST

### 3.1 Results of Moisture Content and Ash Content Tests of Activated Charcoal from Coffee Grounds

The determination of moisture content and ash content was carried out to determine the characteristics of activated carbon and non-activated carbon. The results of the activated carbon characteristics test of coffee grounds are shown in Table 1 below.

**Table 1.** Results of Activated Carbon Characteristics Testing from Coffee Grounds

| Characteristics | Activated Charcoal Quality Standard (SNI No. 06-3730-1995) | Activated Charcoal from Coffee Grounds |
|---|---|---|
| Moisture Content | Maximum 15% | 12.56% |
| Ash Content | Maximum 10% | 3.78% |

Moisture content determination was conducted to assess the hygroscopic properties of activated carbon; the lower the moisture content of activated carbon, the better its quality [4]. The moisture content of the activated carbon tested was found to be 12.56%. This indicates that the percentage of moisture content in this activated carbon meets the standards of SNI No. 06-3730-1995. The decrease in moisture content in coffee grounds activated carbon indicates that the free water and bound water in the coffee grounds activated carbon have evaporated during the carbonization process. According to [5] it stated that this decrease in moisture content is related to the hygroscopic nature of the phosphoric acid activator. Hygroscopicity means that phosphoric acid can absorb and bind water molecules from the surrounding environment. When water molecules are bound by phosphoric acid, the surface of the activated carbon becomes drier and more capable of absorbing other substances, thereby enhancing its adsorption capacity.

Meanwhile, ash content testing is conducted to determine the remaining metal oxide content in coffee grounds activated carbon. This ash content affects the quality of activated carbon as an adsorbent. The ash content of coffee grounds meets SNI No. 06-3730-1995 standard with a percentage of 3.78%. The reduction in ash content in activated carbon from coffee grounds occurs due to the presence of phosphoric acid as an activator that can dissolve metal oxides in the coffee grounds. According to [6] activated carbon consists of layers stacked on top of each other that form pores, where the carbon pores usually contain impurities in the form of inorganic minerals and metal oxides that clog the pores. During the activation process, these impurities dissolve in the activator, thereby increasing the surface area of the pores. This is what enables the activation process to enhance the adsorption capacity of an adsorbent.

### 3.2 FTIR Test Results of Activated Charcoal from Coffee Grounds and Fly Ash

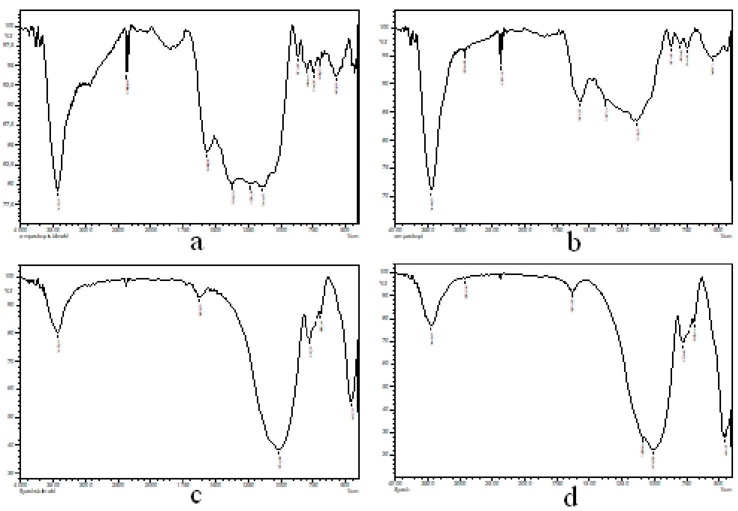


**Figure 2.** a) Spectrum of Coffee Grounds Before Activation, b) Spectrum of Coffee Grounds After Activation c) Spectrum of Fly Ash Before Activation, d) Spectrum of Fly Ash After Activation

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Figure 2 shows that there are differences in the absorption band lengths before and after activation in both activated carbon samples from coffee grounds and fly ash. For more details, see Table 2 and Table 3.

**Table 2.** FTIR Spectrum Results of Coffee Grounds Charcoal

| Peak | Coffee Grounds Charcoal Before Activation | Coffee Grounds Charcoal After Activation | Wavenumber ($cm^{-1}$) | Functional Group |
|---|---|---|---|---|
| A | 1566.20 | 1577.77 | 1450-1600 | C=C |
| B | 1145.72 | 1132.21 | 1050-1150 | C–O |

**Table 3.** FTIR Spectrum Results of Fly Ash

| Peak | Fly Ash Before Activation | Fly Ash After Activation | Wavenumber ($cm^{-1}$) | Functional Group |
|---|---|---|---|---|
| X | 1014.56 | 1093.64 | Around 1100 | Si-O-Si |
| Y | 773.31 | 773.46 | Around 800 | Si-O |

Based on the analysis results in Table 3.2.1 above, peak A at a wavenumber of 1566. 20 $cm^{-1}$ to 1577.77 $cm^{-1}$ indicates the presence of C=C stretching functional groups from aromatic rings, as indicated by the wavelength range of 1450–1600 $cm^{-1}$, which suggests the presence of carbon. The change in the spectrum of coffee grounds indicates an increase in the chemical bond structure of activated carbon in coffee grounds. However, the shift is only slight, which may be due to the use of phosphoric acid as an activator, which is classified as a weak acid, resulting in only a small increase in carbon content in the activated carbon from coffee grounds. Meanwhile, based on research by Khalaida and Sinna (2024), the shift in the activated carbon from coffee grounds sample was from 1566.20 $cm^{-1}$ to 1624.06 $cm^{-1}$. In this case, the increase is quite significant because the activator used is hydrochloric acid, which is classified as a strong acid, so the carbon produced is indicated to be stronger.

At peak B, the wavenumber shifted from 1132.21 $cm^{-1}$ to 1014.56 $cm^{-1}$, corresponding to the absorption band of C-O, indicating the presence of ether groups containing carbon. From the table, it can be concluded that coffee grounds, both before and after activation, contain carbon in aromatic C=C groups and C-O groups, making them suitable for use as absorbents.

The data in Table 3.2.2 regarding the FTIR spectrum of fly ash before and after activation strongly indicates that fly ash contains silica with absorption bands around 1014.56 $cm^{-1}$ and 1093.64 $cm^{-1}$ for Si-O -Si, and 777.31 $cm^{-1}$ and 773.46 $cm^{-1}$ for Si-O stretching, respectively, indicating the presence of characteristic bonds of silica. It can be concluded that the changes observed in the spectrum after activation support the presence of silica in fly ash.

### 3.3 SEM Test Results of Activated Charcoal from Coffee Grounds and Fly Ash

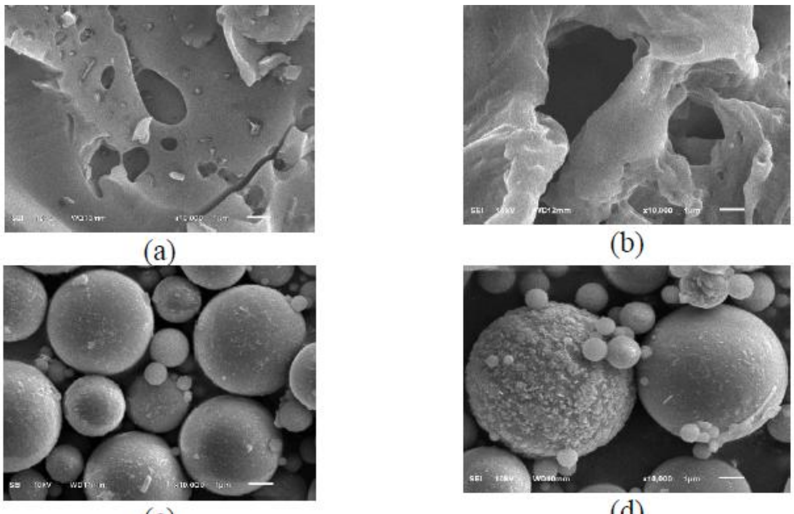


**Figure 4.** Monograph: a) Coffee grounds before activation, b) Coffee grounds after activation, c) Fly ash before activation, and d) Fly ash after activation

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In Scanning Electron Microscope (SEM) testing before activation with phosphoric acid, the SEM test results showed that the morphology of the coffee grounds-based adsorbent appeared asymmetrical, while the fly ash-based adsorbent that had not been physically activated had a symmetrical spherical shape.

After activation with phosphoric acid, the SEM test results showed that the morphology of both coffee grounds-based and fly ash-based adsorbents that were physically activated exhibited qualitative thickening visible in the micrograph at 10,000x magnification. Additionally, the activation process of activated carbon with phosphoric acid solution affected pore size. As seen in Figure (b), the pore size of activated carbon is larger compared to Figure (a) before activation. According to [7] phosphoric acid solution as an activator can influence surface area because it is a strong acid capable of removing hydrocarbon compounds or impurities, thereby causing pore formation on the carbon surface. This will affect the efficiency of the adsorption process because, according to [8], an increase in the number of pores on the surface of activated carbon will enhance its adsorption capacity.

### 3.4 EDS Test Results of Activated Charcoal from Coffee Grounds and Fly Ash

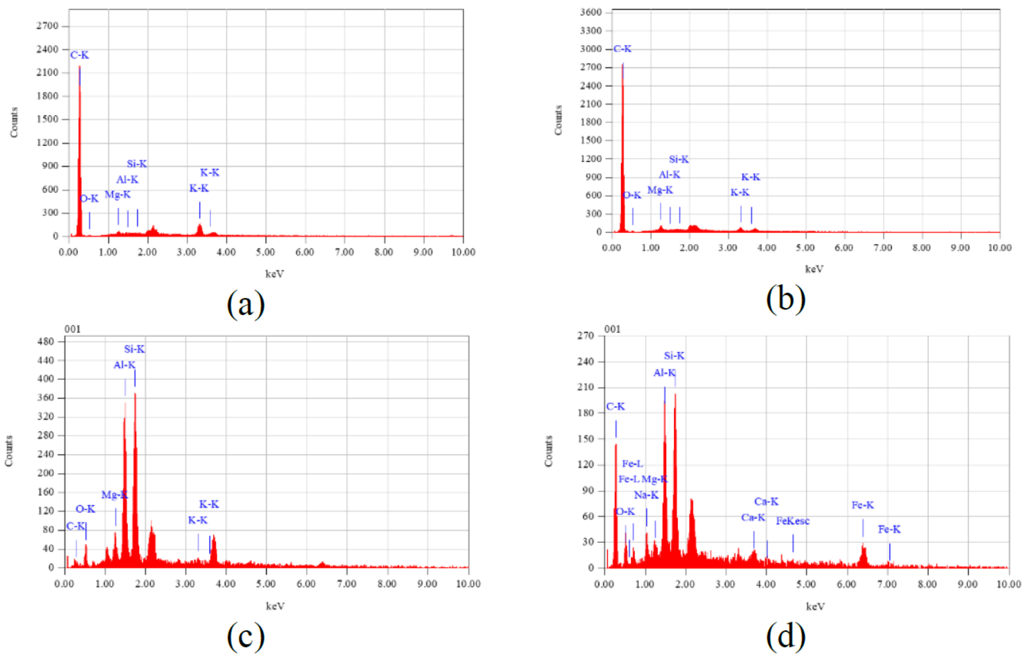


**Figure 5.** Graph: a) Coffee grounds before activation, b) Coffee grounds after activation, c) Fly ash before activation, and d) Fly ash after activation

Based on characterization testing using Energy Dispersive X-Ray Spectroscopy (EDS) with 3000x magnification, information was obtained on the chemical elements present in the coffee grounds-based adsorbent before activation with phosphoric acid, with each element identified by its percentage Mass: C (91.86%), O (2.20%), Mg (0.54%), Al (0.02%), Si (0.01%), and K (5.37%). Meanwhile, in the fly ash-based adsorbent before physical activation, there were several elemental components with the following mass percentages: C (10.48%), O (10.56%), Mg (4.00%), Al (30.44%), Si (43.19%), and K (1.32%).

There was a change in the elemental composition of the coffee grounds-based adsorbent after activation with phosphoric acid, with each element having 8% mass as follows: C (95.32%), O (2.06%), Mg (0.88%), Al (0.09%), and K (1.65%). Meanwhile, in the physically activated fly ash-based adsorbent, there are several elemental components with the following mass percentages: C (34.32%), O (3.81%), Na (1.60%), Mg (0.77%), Al (8.99%), Si (11.90%), Ca (1.97%), and Fe (36.64%).

### 3.5 COD Analysis Results

The liquid waste from tofu industries generally has physical characteristics such as pale-yellow color, turbidity, unpleasant odor, slightly viscous texture, and acidic nature. Before the adsorption process was carried out, the tofu wastewater was first measured for its COD content and pH value.

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**Table 4.** Initial Wastewater Analysis

| Parameter | Initial Concentration of Tofu Liquid Waste | Quality Standard |
|---|---|---|
| COD | 141,32 mg/L | 300 mg/L |
| pH | 4.44 | 6-9 |

It can be observed that the COD content in the tofu wastewater is very high, and the pH value is considerably acidic. Therefore, an adsorption process was carried out to reduce the COD level and increase the pH value. Table 5. shows the COD concentration after the adsorption process.

**Table 5.** COD Concentration of Tofu Liquid Waste After Adsorption Process

| Time | COD (mg/L) | | | | |
|---|---|---|---|---|---|
| (min) | 2 ak : 8 fa | 3 ak : 7 fa | 5 ak : 5 fa | 7 ak : 3 fa | 8 ak : 2 fa |
| 0 | 14132 | 14132 | 14132 | 14132 | 14132 |
| 60 | 6660 | 6985 | 8934 | 8284 | 12183 |
| 90 | 4711 | 5037 | 7959 | 7959 | 9584 |
| 120 | 2761 | 3411 | 4711 | 6497 | 8609 |

*Note: ak = coffee grounds; fa = fly ash*

The experimental results indicate that the COD content in the tofu liquid waste decreased after the adsorption process using a mixed adsorbent of coffee grounds and fly ash. Based on the compositions used, the lowest COD value was obtained from the adsorption process using an adsorbent composition of activated coffee grounds charcoal: fly ash in a 2:8 ratio. Meanwhile, based on contact time, the lowest COD value was obtained from the adsorption process carried out for 120 minutes. The trend of COD reduction is illustrated in Figure 6 below.

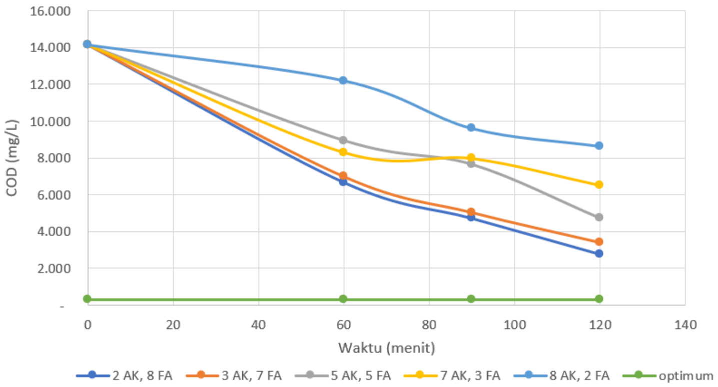


**Figure 6.** COD Concentration Curve vs. Contact Time

The graph shows that the higher the proportion of fly ash used in the adsorbent mixture, the lower the resulting COD concentration in the tofu wastewater. This may be due to the smaller particle size of fly ash compared to coffee grounds activated charcoal, which gives fly ash a larger surface area. The larger the surface area of the adsorbent, the more adsorbate can be absorbed, making the adsorption process more effective [9].

In addition, the graph also indicates that the longer the contact time between the adsorbent and the tofu liquid waste, the lower the resulting COD value. Contact time affects the adsorption process because the longer the contact between the adsorbent and the waste, the better the diffusion and attachment of impurities [10].

Based on the experimental results, although the COD concentration in the tofu liquid waste after adsorption is still significantly higher than the quality standard of 300 mg/L, our research has demonstrated a notable reduction in COD levels. Figure 7. presents the percentage reduction in COD concentration of tofu liquid waste after the adsorption process.

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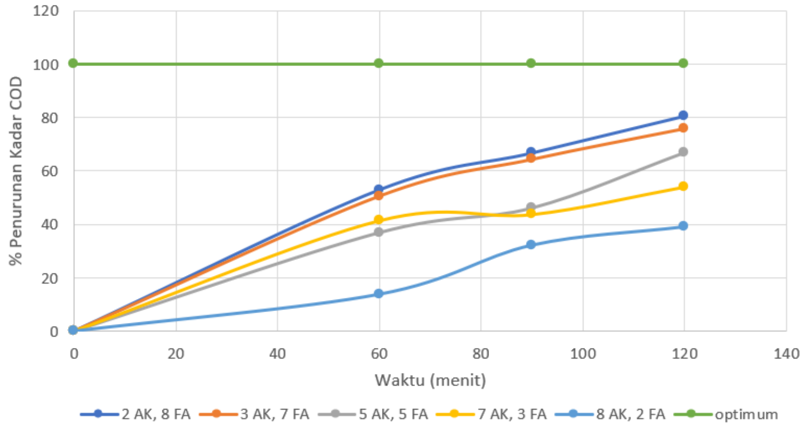


**Figure 7.** COD Reduction Percentage Curve vs. Contact Time

Based on Figure 4.3, the highest COD reduction percentage was 80.46%, with a COD value of 2,761 mg/L, obtained from the adsorption process using a 2:8 ratio of coffee grounds to fly ash and a contact time of 120 minutes.

### 3.6 pH Analysis Results

The pH measurement process in this experiment aimed to determine the acidity level of tofu wastewater after the adsorption process. pH value is a crucial factor for wastewater discharged into the environment, as it directly affects aquatic biological life. Table 3.6 presents the pH analysis results of tofu wastewater after the adsorption process.

**Table 6.** pH Analysis of Tofu Wastewater After Adsorption Process

| Time (min) | pH | | | | |
|---|---|---|---|---|---|
| | 2 ak : 8 fa | 3 ak : 7 fa | 5 ak : 5 fa | 7 ak : 3 fa | 8 ak : 2 fa |
| 0 | 4.44 | 4.44 | 4.44 | 4.44 | 4.44 |
| 60 | 5.02 | 4.76 | 4.66 | 4.58 | 4.53 |
| 90 | 5.68 | 5.11 | 4.87 | 4.67 | 4.61 |
| 120 | 6.21 | 5.43 | 5.1 | 4.72 | 4.71 |

*Note: ak = coffee grounds; fa = fly ash*

Overall, the pH of the wastewater ranged between 4 and 6. This value is still below the quality standard regulated by the Indonesian Ministry of Environment Regulation No. 5 of 2014, which sets the acceptable pH range at 6 to 9. The optimum pH value was obtained from the adsorption process using an adsorbent composition of activated carbon and fly ash in a 2:8 ratio for 120 minutes, resulting in a pH of 6.21. This pH value complies with the quality standard stated in the Indonesian Ministry of Environment Regulation No. 5 of 2014. The trend of increasing pH in tofu wastewater is illustrated in Figure 8. below.

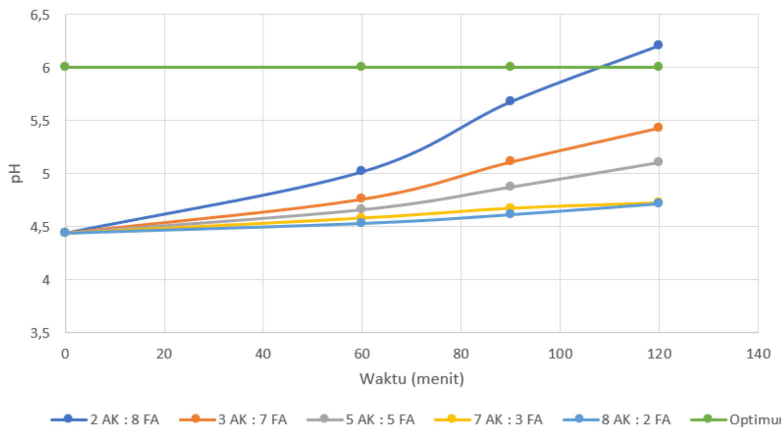


**Figure 8.** pH Curve of Tofu Wastewater with Various Adsorbent Compositions Over Time

Based on Figure 8, it can be observed that the trend shows an increase in pH over time. The increase in pH may occur due to the use of phosphoric acid as an activating agent during the adsorbent activation process, which contributes additional $H^+$ ions into the solution. Over time, these $H^+$ ions are adsorbed by the adsorbent, leading to a decrease in $H^+$ ion concentration in the solution, which consequently increases the pH.

## 4. CONCLUSION

Based on the discussion above, it can be concluded that the optimal composition of coffee grounds and fly ash-based adsorbents can be determined at a ratio of 20:80, with the variable time of the adsorption process operating at maximum capacity within a 120-minute timeframe. The results of this composition show a significant reduction in Chemical Oxygen Demand (COD) levels, starting from the initial condition of industrial soybean curd wastewater with a concentration of 14,132 mg/L to 2,761 mg/L. Although this value does not yet meet the wastewater quality standards for tofu according to Ministry of Environment Regulation No. 5 of 2014, Page 47, which specifies 300 mg/L, the results demonstrate the effectiveness of the coffee grounds and fly ash adsorbent with an efficiency of 80.46%. Additionally, there was an increase in the initial pH of the industrial tofu wastewater, which had a pH of 4.44, to a pH of 6.2, which meets the quality standards specified in Ministry of Environment Regulation No. 5 of 2014, Page 47, regarding soybean processing wastewater (tofu), which requires a pH range of 6–9.

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