

# Free Fatty Acid Adsorption of Crude Palm Oil by Modified Fly Ash

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## ABSTRACT

This study used modified fly ash as a material to adsorb free fatty acid in Crude Palm Oil. One or two types of modification treatments in previous studies were used to investigate the effect on the absorption process. Therefore, this study focused on the capacity of the modified fly ash in adsorbing FFA with varying concentrations and durations of adsorption. Modification of fly ash began through leaching process using hydrochloric acid (HCl), followed by activation using hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>), and reactivation using cocamidopropyl betaine (CAPB) surfactant. Scanning Electron Microscope Energy Dispersive X-Ray (SEM-EDX) is used to analyze the morphology and elements, while a Gas Sorption Analyzer (GSA) is used to analyze the pore radius, surface area, and pore volume of modified fly ash. Based on SEM-EDX result, fly ash had an amorphous shape with silicon (32.15%) and oxygen (50.29%) as the major elements. While GSA showed that the surface area, total pore volume, and average pore diameter were 15.34 m<sup>2</sup>/g, 0.02 cc/g, and 3.23 nm, respectively. Furthermore, modified fly ash had adsorption efficiency and capacity of 44.38%±0.21 and 140 mg/g±0.21, respectively. Then, adsorbent mass of 6% w/w and adsorption duration of 60 minutes recorded as the optimal condition of FFA adsorption process using fly ash. The maximum permissible FFA in cooking oil based on Standar Nasional Indonesia (2019) was 0.30%. Because of that, multi-stage adsorption was carried out to reduce the FFA in order to comply with Standar Nasional Indonesia (2019). The adsorption with twice repetition can produce CPO with FFA < 0.30% (initial FFA content of 1.2%). Due to the reduction level of FFA, the yield of the CPO obtained from each step was ±60-80%. This study could be applied in industry to reduce the FFA content and enhance the CPO quality.

**Keywords:** Crude palm oil, fly ash, free fatty acid, leaching, surfactant

## INTRODUCTION

Free fatty acid (FFA) is one of the factors that determine the quality of crude palm oil (CPO). The existence of triglyceride hydrolysis process by lipase enzyme causes the formation of FFA in CPO. This formation is accelerated by high temperatures and rusty storage. High content of FFA impacts the oil acidity, produces an unpleasant odor, reduces its shelf life, and affects the oxidative stability of palm oil (Sopianti et al., 2017). According to SNI 7709 (2019), palm cooking oil must contain FFA below 0.3%. CPO normally contains

FFA in the range from 1-5%, and the market value reduces when the value is above 5%. Reducing FFA can be achieved through adsorption using fly ash, where pores enable Van der Waals interactions and active sites form chemical bonds with FFA (Ahn & Kwak, 2020).

Previous studies on FFA adsorption have been widely conducted, including the use of bleaching earth. Although it has good effectiveness in the adsorption process, bleaching earth also has weaknesses. This material can increase acidity and FFA content in the oil (Bielaca and Zagospodarowania, 2013). A study conducted by Rengga et al. (2021) found that active

carbon adsorbent formed from banana peel with activation using KOH and calcination could adsorb FFA in waste cooking oil until 62 mg/g. Bonassa et al. (2016) also found that fly ash adsorbent without activation could adsorb FFA in waste cooking oil up to 63-68%. Therefore, this study used another material (fly ash) with several activation methods that are feasible due to its ability to adsorb FFA through physical and chemical adsorption using pores, surface area, and several active sites. The aim was to increase the effectiveness of fly ash in adsorbing FFA.

Fly ash (FA) is a by-product of the coal combustion process. The total global production of FA is 600 million tons per year, while Indonesia produced 8.3 million tons in 2019, and it is increasing 5% every year (Aigbe et al., 2021). In addition, FA is widely used as the base material for glass and concrete. Fly ash has pores with average size of 1-2 nm, therefore it also can be an adsorbent. Moreover, the pores can be enlarged through modification. Fly ash contains several metal oxide compounds, with silica oxide ( $\text{SiO}_2$ ) as its dominant component. Hence,  $\text{SiO}_2$  in fly ash provides high adsorption capacity because it has pore-forming ability and large, also easily modified surface area (Ahn & Kwak, 2020). Susilowati et al. (2019) found that fly ash which has pore radii ranging from 1-5 nm after treatments adsorbs up to 87.44% of FFA in waste cooking oil.

Adsorption capacity of fly ash is also influenced by the high silica content in FA. Silica makes FA more hydrophobic, preventing its pores from binding with water and ensuring dryness, which improves adsorption of FFA. Silica also provides more active sites (-Si-OH and Si-O-Si) that enhance the adsorption process (Mushtaq et al., 2022). However, treatments are needed to increase the silica content in FA that can influence the effectiveness of adsorption process.

The effectiveness of FA as an adsorbent can be enhanced by modifying its characteristics. Leaching and activation are two of treatments that can be used to remove certain metal oxides, enlarge the pores, and increase the surface area. Cao et al. (2018) reports that the most effective leaching process is using hydrochloric acid (HCl). Furthermore, several studies have examined modification methods to increase the pore size of FA. Then, the modification of FA through activation with hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) and surfactants is a method to enhance the adsorption effectiveness. According to Meirinna et al. (2013),  $\text{H}_2\text{O}_2$ -activated fly ash removed 94.99% of chromium (III) compared to 52.37% for unactivated FA. Therefore, Ghafar et al. (2020) reports that CAPB surfactant-activated clay has wider pore radii (7 nm) compared to the unactivated clay (2 nm).

This study applies leaching (with HCl) and activation (with  $\text{H}_2\text{O}_2$  and CAPB surfactant) to evaluate the effect on FA and its capacity to adsorb FFA. Hydrophobic sites in CAPB surfactant will interact with FA and hydrophilic sites will interact with FFA. Several active sites in fly ash are capable of forming hydrogen bonds and Van der Waals interactions with FFA, thereby improving adsorption effectiveness (Bariyah et al., 2017). This study also examines optimum adsorption conditions, focusing on adsorbent mass and contact time. Adsorbent mass influences efficiency and capacity through surface area and active site availability, while contact time affects the process by increasing the reaction rate and strengthening the interaction between adsorbent and adsorbate.

## METHODS

### Materials

The materials used in this study included  $\text{C}_2\text{H}_5\text{OH}$  (SmartLab, 99%),  $\text{H}_2\text{O}$ ,  $\text{H}_2\text{O}_2$  (Merck, 30%), HCl (SmartLab, 37%), phenolphthalein indicator (Merck), potassium hydrogen phthalate (Merck), NaOH (Merck), and CAPB surfactant (31.8%). Fly ash was obtained from PT Indonesia Chemical Alumina (ICA), and CPO from PT Pundi Lahan Khatulistiwa, Indonesia.

The tools used included a hot plate (IKA C-Mag HS 7, Germany), oven (Mettler, Germany), pH meter (Hana Instrument, US), shaker (Certomat Sartorius, Germany), and a set of glassware. The instruments were Scanning Electron Microscope-Energy Dispersive X-Ray (SEM-EDX) (magnification 10,000 times, Jeol JSM 6510 (LA)) and Gas Sorption Analyzer (GSA) (full isothermal 20 pts., Quantachrome TouchWin v1.22).

### Modification of Fly Ash

Fly ash (FA) was rinsed with distilled water three times with a ratio of 1:5 w/v. The mixture was then separated using the decantation method, and the residue was collected and dried using an oven at 105 °C for 7 hours. FA was sieved through an 80-mesh sieve and then subjected to leaching by a reflux method in 3 M HCl at a 1:8 w/v ratio, maintained at 90 °C for 5 hours. The residues were collected using decantation method. The residues were rinsed with distilled water until the filtrate pH was neutral (Cao et al., 2018) and dried using an oven at 105 °C for 7 hours (FA1). Subsequently, 10% (v/v) hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) solution in a ratio of 1:10 (w/v) (FA1: $\text{H}_2\text{O}_2$ ) was used to activate FA1. Then, the mixture was stirred for 60 minutes using a hotplate at room temperature. The mixture was allowed to stand for 24 hours and then decanted. The residue was dried using an oven at 150 °C for 4 hours. Then, the residue

was rinsed using distilled water until the filtrate pH was neutral and dried in the oven for 7 hours at 105 °C (Meirinna et al., 2013). After that, it is also sieved using an 80 mesh siever (FA2).

The second activation was using 3 mM cocamidopropyl betaine (CAPB) solution at a ratio of 1:10 (w/v) (FA2:CAPB). The mixture was stirred using a hot plate for 60 minutes at room temperature. Subsequently, the residue was collected and dried using an oven for 10 hours at 105-110 °C. The residue was rinsed using distilled water until the filtrate pH was neutral and dried for 7 hours at 105°C (Mobarak et al., 2018). Afterward, the residue was sieved with an 80 mesh siever (FA3), and fly ash yield obtained was calculated using Equation 1.

$$\% \text{ Yield} = \frac{\text{FA3 mass}}{\text{FA mass}} \times 100\% \quad (1)$$

### Characterization of FA, FA1, FA2, and FA3

FA, FA1, FA2, and FA3 were characterized using SEM-EDX (10,000 times magnification, Jeol JSM 6510 (LA)) to identify the structure, shape, and pore morphology of the adsorbent. In addition, characterization using GSA (full isothermal 20 pts., Quantachrome TouchWin v1.22) was also conducted to determine the surface area, pore size, volume, and pore distribution of fly ash.

### Preparation of CPO

CPO was prepared through a degumming process to reduce gum (phosphatides and other impurities). A total of 1 kilogram of CPO was placed in a beaker and heated to 100 °C, then 1% w/w H<sub>3</sub>PO<sub>4</sub> was added while stirring on a hotplate for 20 minutes. The CPO was filtered to separate the gum, and the filtrate was taken. For the neutralization process, 2 M NaOH (15% w/w) was added to the filtrate, and the mixture was heated to 75 °C and stirred for 20 minutes using a hotplate. The CPO was filtered, and the filtrate was collected and rinsed using warm water (60 °C). This was followed by separation in a separatory funnel, which the oil was collected and dried in an oven at 105 °C for 2 hours to remove water (Onwumelu & Onwumelu, 2022).

### Preparation for FFA Test

NaOH 0.1 N served as the titrant in FFA test and was standardized with potassium hydrogen phthalate (KHP), which had been dried in an oven at 120 °C for 2 hours. A mass of 0.40 ± 0.02 g KHP was dissolved in 50 mL of distilled water, and a few drops of phenolphthalein indicator were added. The solution was titrated with the NaOH titrant until a pink color appeared. The titration

was replicated thrice, and the normality of the NaOH solution was calculated using Equation 2.

$$N = \frac{W \times 1000}{V \times 204,2} \quad (2)$$

N is the normality of NaOH, W is the weight of potassium hydrogen phthalate (g), V is the volume of titrant solution used (mL), and 204.2 is the equivalent weight of potassium hydrogen phthalate (SNI 01-2901-2006).

### Adsorption of CPO FFA on Varying Adsorbent Mass

A sample of 25 g CPO was taken, and the adsorbent mass (FA3) was varied at 2, 4, 6, 8, and 10% w/w. CPO was mixed with adsorbent at each mass variation, shaken (250 rpm) for 60 minutes, and filtered to collect the filtrate. Each treatment was replicated thrice, and the data were presented as averages with standard deviation.

### Adsorption of CPO FFA on Varying Adsorption Duration

Adsorbent (FA3) was added to each sample (25 g CPO) with the optimum mass obtained in the previous treatment. Several time variations are used in the CPO shaking process (250 rpm in 30, 60, 90, 120, and 150 minutes). The CPO was filtered and the filtrate was taken. Each process was carried out in three repetitions, and the data were presented as averages with standard deviation.

### Multi-stage Adsorption of FFA

A sample of 25 g CPO was taken for the adsorption process. Adsorbent (FA3) was added to each sample at the optimum mass. CPO was shaken at 250 rpm for 60 minutes and then filtered to obtain the filtrate. The application of optimum adsorbent mass and adsorption duration aimed to achieve the best results and reduce the FFA content to meet the standard of SNI (2019). When FFA content in CPO did not meet the maximum limit of 0.30% stated in Standar Nasional Indonesia 7709 (2019), the adsorption process was repeated with a fresh adsorbent. Each adsorption was replicated thrice, and the data were presented as averages with standard deviations.

### Determination of FFA Content

A test sample of 5 g CPO was taken for determining FFA content. CPO was heated at 60-70 °C and stirred until homogeneous. A total of 50 mL of hot ethanol and phenolphthalein indicator was added. Subsequently, the CPO was titrated using 0.1 N NaOH until a pink color appeared. Determination of FFA content was conducted

thrice, and the content was calculated using Equation 3 (SNI 7709, 2019).

$$\%FFA = \frac{25,6 \times N \times V}{W} \quad (3)$$

N is the normality of NaOH, V is the volume of NaOH (mL), W is the weight of the sample (g), and 25.6 is the constant for calculating FFA as palmitic acid (SNI 7709, 2019). Adsorption capacity and efficiency were calculated using Equations 4 and 5, respectively.

$$Q = \frac{(C_o - C_i) \times V}{W_{adsorbent}} \quad (4)$$

$$\% \text{ Adsorption efficiency} = \frac{(C_o - C_i)}{C_o} \times 100\% \quad (5)$$

Q is adsorption capacity (mg/g), C<sub>o</sub> is the initial FFA concentration (mg/L), C<sub>i</sub> is the final FFA concentration (mg/L), V is the solution volume (L), and W is the adsorbent weight (g).

## RESULTS AND DISCUSSION

### Characterization of Fly Ash Using SEM-EDX and GSA

Fly ash (FA) requires specific treatments to enhance its adsorption effectiveness. In this study, three modification processes were applied, namely leaching with HCl (FA1), activation with hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) (FA2), and CAPB surfactant (FA3). Figure 2 showed that raw FA appeared as a black powder, while FA1, FA2, and FA3 had finer particle sizes. The treated samples were also lighter in color than raw FA, a change attributed to the drying process, since moisture content influenced the darkness of FA.

Energy Dispersive X-ray (EDX) analysis (Table 1) was conducted to determine the chemical composition of fly ash. The results showed that oxygen (44–50%) and silicon (25–26%) were the dominant elements, while sodium, calcium, titanium, magnesium, iron, and potassium were < 10%. The reduction in some metal oxides was attributed to the interaction with HCl during

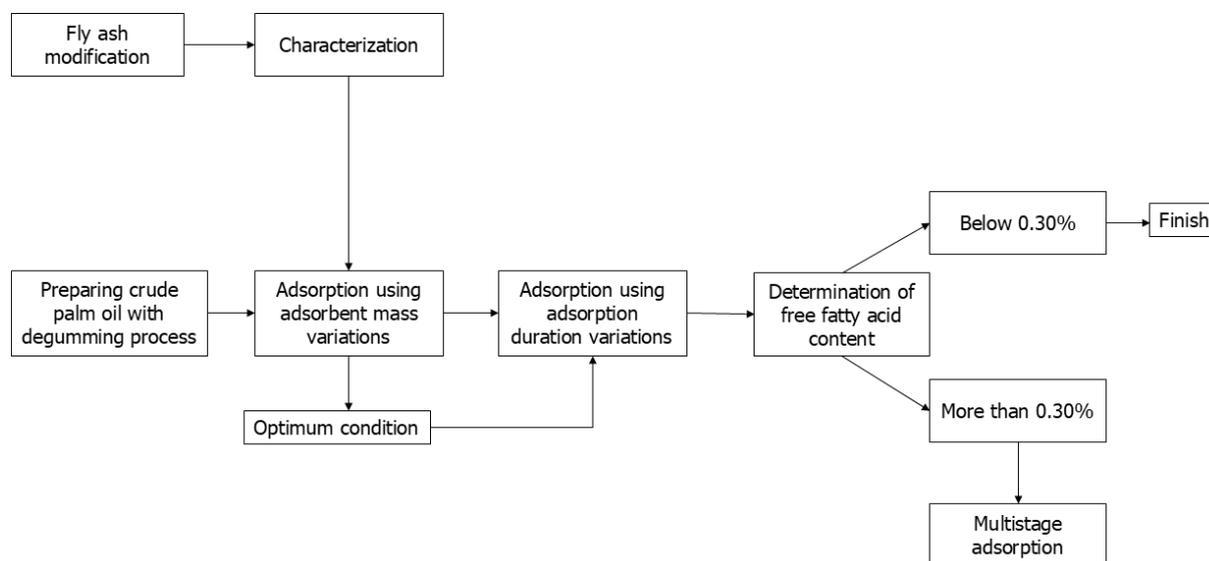


Figure 1. Study Methods

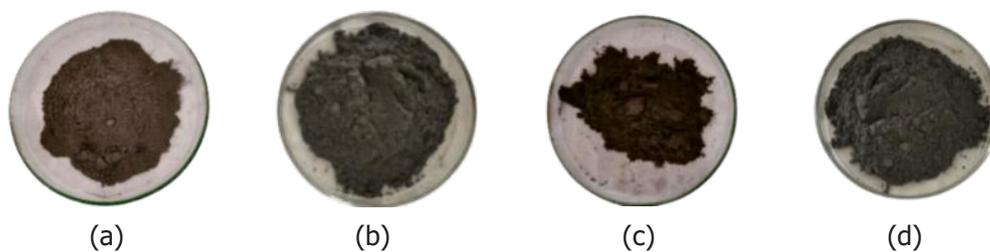


Figure 2. (a) Fly ash; (b) Chloride acid-activated fly ash; (c) Hydrogen peroxide-activated fly ash ; (d) CAPB surfactant-activated fly ash.

Table 1. Elements of fly ash, chloride acid-activated fly ash, hydrogen peroxide-activated fly ash, and CAPB surfactant-activated fly ash based on SEM-EDX analysis (%mass)

Sample	Elements								
	C	O	Na	Mg	Al	Si	Ca	Ti	Fe
FA	7.60	44.03	1.11	0.77	12.98	26.96	0.96	0.27	5.33
FA1	5.19	50.76	0.17	0.12	3.96	37.25	0.24	0.61	1.69
FA2	8.42	46.00	2.11	0.46	14.64	25.78	0.69	0.46	1.4
FA3	8.83	50.29	0.46	0.24	6.39	32.15	0.12	1.01	0.50

leaching. Table 1 showed that Fe and Al had the greatest decrease because the higher electronegativity made these elements more soluble in HCl compared to Na, Mg, Ti, and Ca (Cao et al., 2018). These results suggested that HCl treatment effectively reduces metal oxides that could otherwise block the pores and active sites of fly ash.

A Scanning Electron Microscope (SEM) was used to analyze the surface characteristics of fly ash. Figure 3 (a) shows the morphology of fly ash before modification, (b) after leaching, (c) after activation with hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>), and (d) after modification with CAPB surfactant. Based on these figures, fly ash particles were dominantly sphere-shaped and amorphous. Figures 3 (b) and 3 (c) showed that there was disaggregation of fly ash particles. In addition, leaching with HCl and activation using H<sub>2</sub>O<sub>2</sub> caused the formation of a more homogeneous size of pore size, which impacted the total volume (Buema et al., 2021). Figure 3 (d) showed re-aggregation that reduced the surface area. The suboptimal heating process left residual surfactant in fly ash pores (Fungaro et al., 2021). Several pores in FA3 were closed, which decreased the total pore volume.

The distribution of elements in fly ash, identified in Table 1, was visualized through SEM-EDX mapping (Figure 4). There were some reduced elements (metal oxides) in FA1, FA2, and FA3. In addition, some other elements had a larger percentage (such as Si, Al, and O) were more clearly visible. This was because SEM-EDX detected the interaction of electrons in the instrument with atoms in

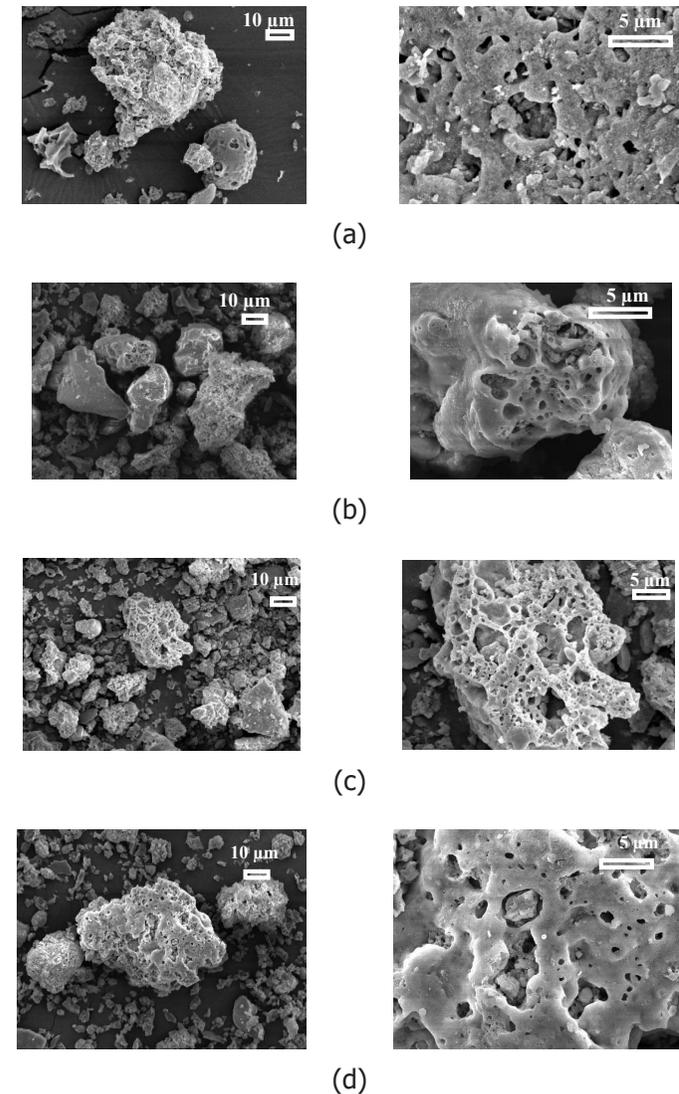


Figure 3. Surface morphology of (a) Fly ash; (b) Chloride acid-activated fly ash; (c) Hydrogen peroxide-activated fly ash; (d) CAPB surfactant-activated fly ash

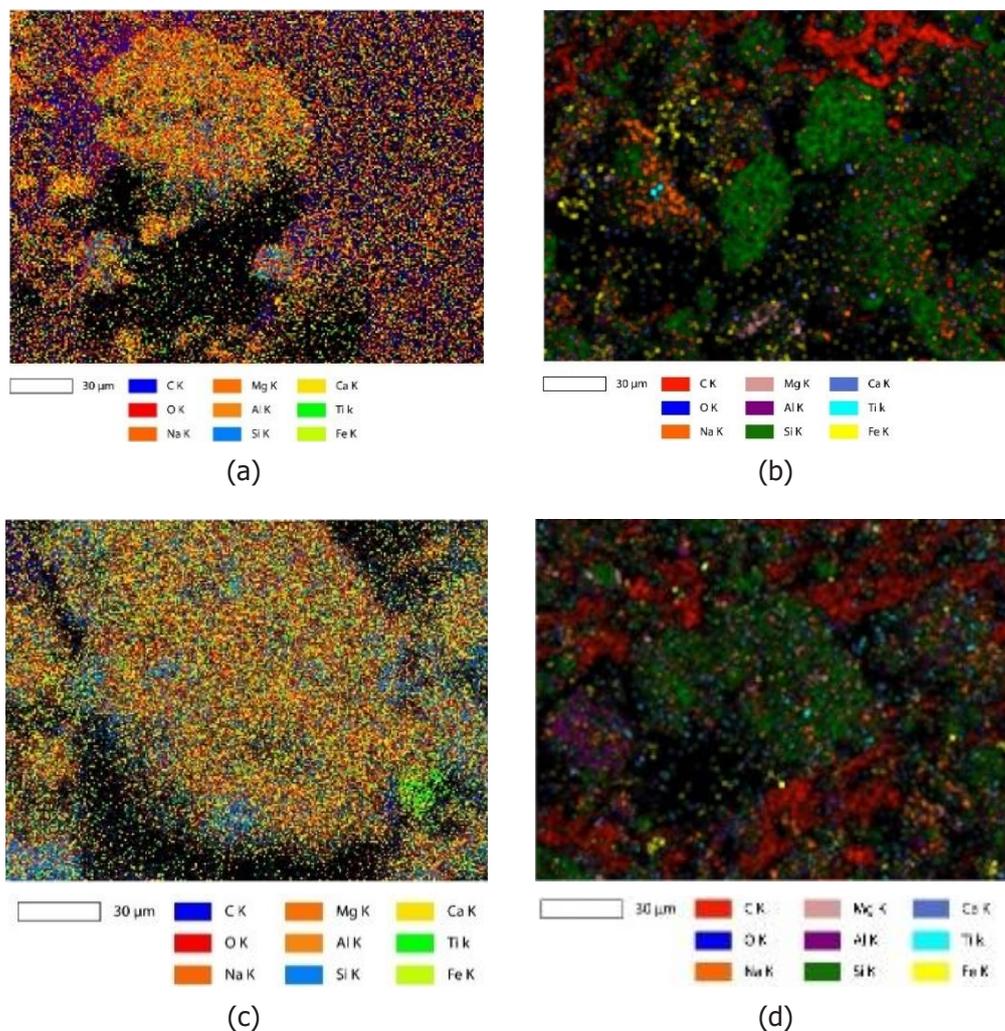


Figure 4. EDX mapping of (a) Fly ash; (b) Chloride acid-activated fly ash; (c) Hydrogen peroxide-activated fly ash; (d) CAPB surfactant-activated fly ash

Table 2. Characterization results of FA, FA1, FA2, FA3 using GSA and its adsorption capacity

Sample	BET Specific Surface Area (m <sup>2</sup> g <sup>-1</sup> )	Total Pore Volume (ccg <sup>-1</sup> )	Average Pore Radius (nm)	Adsorption Capacity (mg/g)
FA	4.806	0.00913484	3.8016	76.53 ± 0.21
FA1	31.766	0.0345199	2.17336	87.73 ± 0.33
FA2	30.040	0.0356173	2.37132	85.87 ± 0.34
FA3	15.337	0.0247563	3.22826	141.87 ± 0.21

fly ash. Therefore, the interaction of elements with a larger percentage increased and impacted the lighter color (Sunjidmaa et al., 2019).

The surface area, pore size and volume, and pore distribution characteristics of fly ash were analyzed using GSA (Table 2). FA had a surface area, total pore

volume, and average pore diameter of 15.337 m<sup>2</sup>g<sup>-1</sup>, 0.025 ccg<sup>-1</sup>, and 3.228 nm, respectively. The increase in specific surface area in FA1, FA2, and FA3 was due to the disaggregation of fly ash particles. In FA1 and FA2, the total pore volume increased, while the average pore diameter decreased due to the formation of smaller

Table 3. Adsorption results on varying adsorbent (FA3) mass

Adsorbent Mass (%w/w)	Adsorption Duration (minutes)	Initial FFA (%)	Final FFA (%)	Adsorption Efficiency (%)
0	60	1.69 ± 0.12	1.69 ± 0.12	0
2	60	1.69 ± 0.12	1.24 ± 0.17	26.63 ± 0.17
4	60	1.69 ± 0.12	1.09 ± 0.16	35.50 ± 0.16
6	60	1.69 ± 0.12	0.94 ± 0.21	44.38 ± 0.21
8	60	1.69 ± 0.12	0.94 ± 0.10	42.38 ± 0.10
10	60	1.69 ± 0.12	1.09 ± 0.13	35.50 ± 0.13

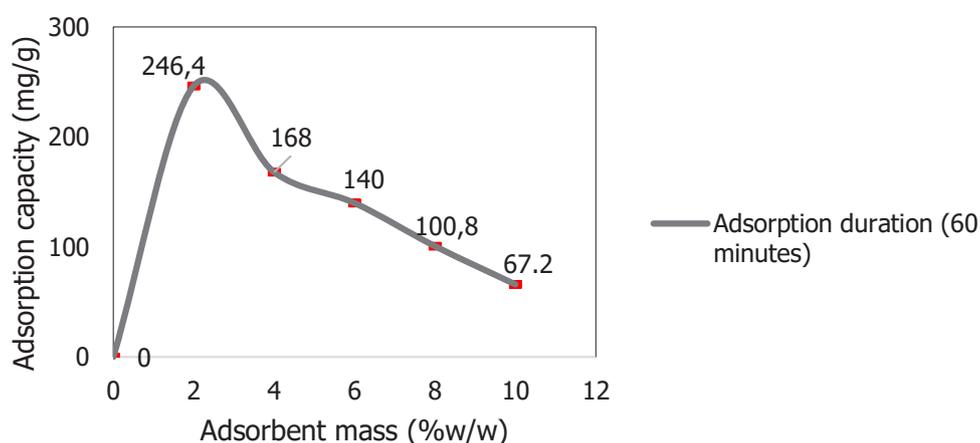


Figure 5. FFA adsorption capacity on varying adsorbent (FA3) mass (25 g CPO)

pores. However, FA3 showed reduced pore volume and surface area, along with larger pore diameters, suggesting that the surfactant enlarged the pores at micelle concentration but reduced the number and caused particle aggregation. The increase in particle aggregation after activation with surfactant was caused by the excessive volume of surfactant used (Mushtaq et al., 2022).

### FFA Adsorption Results

The optimum adsorbent mass (FA3) was 6% w/w with adsorption efficiency and capacity of 44.38% ± 0.21 and 140 mg/g ± 0.21, respectively (Table 3). Increasing the adsorbent mass decreased adsorption efficiency and capacity because it had reached equilibrium conditions, as shown in Table 3 and Figure 5. Equilibrium conditions were achieved when adsorption and desorption rates were equal. This showed that at an adsorbent mass above 6% w/w, the desorption rate tended to be more dominant because it had passed the equilibrium condition. Therefore, FFA bound to

the adsorbent surface was released into the CPO and decreased adsorption efficiency. Excessive adsorbent mass also caused aggregation, which limited the interaction between fly ash surfaces and FFA in CPO. As a result, adsorption efficiency decreased when the mass exceeded 6% w/w (Herawati et al., 2018).

The optimum adsorption duration using FA3 adsorbent was 60 minutes, giving adsorption efficiency and capacity of 44.38% ± 0.21 and 140 mg/g ± 0.21 (Table 4 and Figure 6), respectively. This duration was selected because the result was similar to 90 minutes, but avoided unnecessary process extension that could increase energy consumption and production costs in industrial applications. A longer duration reduced adsorption efficiency as the pore surface became saturated and adsorption rate reached equilibrium (Priyanto et al., 2021).

The adsorption of FFA with the FA adsorbent used a physical adsorption mechanism (pores) because FA3 had a larger pore size compared to FA1 and FA2 (Table 2). The active sites of the modified FA compound

Table 4. Adsorption results on varying adsorption duration using FA3

Adsorption duration (minutes)	Adsorbent Mass (%w/w)	Initial FFA (%)	Final FFA (%)	Adsorption Efficiency (%)
0	6	1.69 ± 0.12	1.69 ± 0.12	0
30	6	1.69 ± 0.12	1.29 ± 0.17	23.67 ± 0.17
60	6	1.69 ± 0.12	0.94 ± 0.21	44.38 ± 0.21
90	6	1.69 ± 0.12	0.94 ± 0.17	44.38 ± 0.17
120	6	1.69 ± 0.12	1.14 ± 0.14	32.54 ± 0.1
150	6	1.69 ± 0.12	1.24 ± 0.27	26.63 ± 0.27

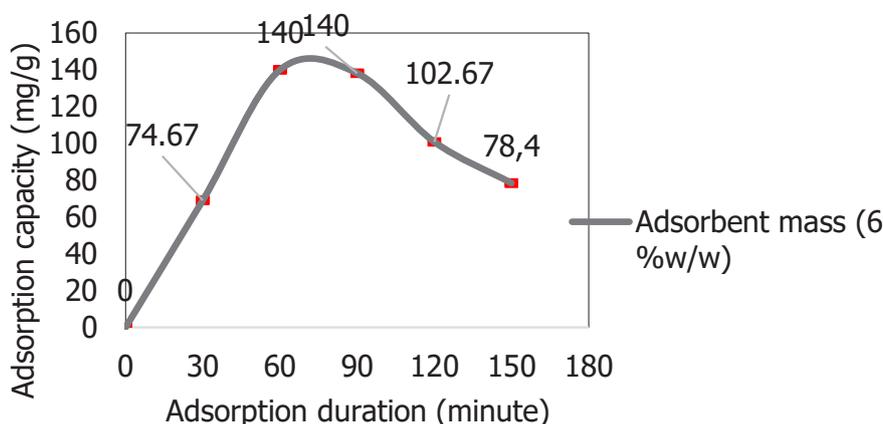


Figure 6. FFA adsorption capacity on varying adsorption duration using FA3

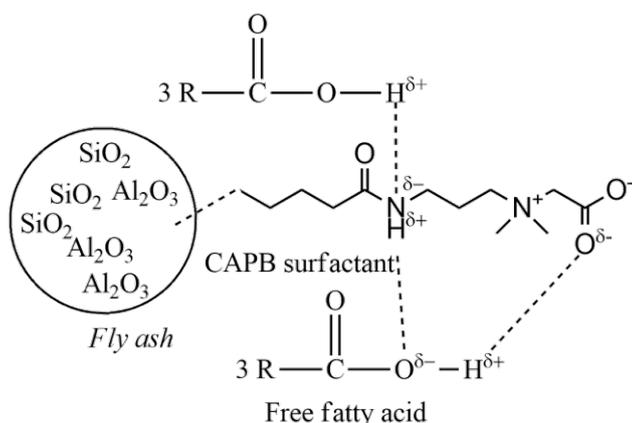


Figure 7. Hypothesized interaction of fly ash with CAPB surfactant and FFA

also bind to FFA molecules. Therefore, the increase in surface area (chemical adsorption mechanism) also affected the adsorption process. Activation with CAPB surfactant enhanced dipole, electrostatic, and hydrogen

bonding interactions between fly ash and FFA (Figure 7). The alkyl groups (-CH) in CAPB interacted with Si-O and Al-O on fly ash (Mushtaq et al., 2022). CAPB attached to fly ash and then interacted with FFA. CAPB was amphoteric with both hydrophilic and hydrophobic sites while FFA had polar properties. The hydrophilic sites bonded with FFA (Bariyah et al., 2017). The amine group (-NH) in CAPB also formed hydrogen bonds with oxygen atoms (-COOH) in FFA. Therefore, FA3 adsorbed more FFA than FA.

The lowest FFA content was 0.94%, while the maximum allowed in cooking oil is 0.30% (SNI 7709, 2019). In this context, multi-stage adsorption using FA3 adsorbent (Table 7) was conducted to produce FFA lower than 0.30%. Table 7 showed that the 6% adsorbent composition produced the lowest FFA content, which was 0.09%.

The study conducted by Susilowati et al. (2019), which adsorbed FFA in cooking oil using calcination-activated fly ash, had adsorption effectiveness of up to 80%. Although adsorption effectiveness in this study

Table 7. Results of multi-stage adsorption using FA3

Adsorbent	Stage of adsorption	Adsorbent mass (%w/w)	Adsorption duration (minutes)	Initial FFA (%)	Final FFA (%)	AE (%)	Q (mg/g)
FA3	1	6	60	1.28 ± 0.10	0.54 ± 0.16	57.81 ± 0.16	138.13 ± 0.16
	2	4	60	0.54 ± 0.16	0.39 ± 0.10	27.78 ± 0.10	28 ± 0.10
FA3	1	6	60	1.28 ± 0.10	0.54 ± 0.16	57.81 ± 0.16	138.13 ± 0.16
	2	6	60	0.54 ± 0.16	0.09 ± 0.07	83.33 ± 0.07	84 ± 0.07



Figure 8. (a) CPO before degumming; (b) CPO after degumming; (c) CPO after adsorption

reached only 40%, the result was reasonable because CPO contained more impurities than cooking oil.

Figure 8 shows a comparison of CPO color before degumming and adsorption, as well as after adsorption using FA3. The color of CPO after adsorption was clearer compared to before and after degumming due to the  $\beta$ -carotene (the cause of the red color in CPO) that was adsorbed (Onwumelu & Onwumelu, 2022).

## CONCLUSION

In conclusion, fly ash activation using  $H_2O_2$  after the leaching process with HCl did not significantly affect the pore diameter of the modified fly ash. However, modified fly ash has a larger surface area, total pore volume, and adsorption efficiency than FA before modification. In this study, the most effective adsorbent is fly ash treated by HCl leaching, followed by  $H_2O_2$  activation and CAPB surfactant reactivation. The optimum conditions are 6% w/w adsorbent mass and 60 minutes of adsorption duration. Adsorption efficiency and capacity of FFA are  $44.38\% \pm 0.21$  and  $140 \text{ mg/g} \pm 0.21$ , respectively. This study carried out multi-stage adsorption to reduce FFA content until  $< 0.3\%$ . At the optimum adsorbent mass and adsorption duration, there is a need for 2 replications to achieve FFA content  $< 0.3\%$ .

## CONFLICT OF INTEREST

All authors declare no conflicts of interest with any party in this article.

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