Optimization Process of Oil Palm Biomass-Based Activated Carbon for Palm Oil Mill Effluent Treatment

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ABSTRACT

The rapid expansion of the palm oil industry has significantly increased the volume of palm oil mill wastewater, creating a growing need for its effective treatment and management. Filtration is one of the unit operations involved in treating Palm Oil Mill Effluent (POME), and activated carbon is a commonly used medium in filtration systems. In this study, activated carbon was synthesized from oil palm biomass, including empty fruit bunch (EFB) and palm kernel shell (PKS). The synthesis involved a series of experiments with varying concentrations of H_3PO_4 (20–60% w/w), activation temperatures (70–100 °C), and activation times (30–60 min). The operating conditions for activation were varied using a 23-factor complete factorial design with one center point (analyzed in the Minitab Program). Performance analysis was conducted by evaluating the ability of activated carbon to reduce pollutant parameters in POME, including biochemical oxygen demand (BOD), chemical oxygen demand (COD), and color. Activated carbon from EFB and PKS was successfully synthesized with larger pores, ranging from 10.91 µm to 15.22 µm, compared to raw EFB and PKS, which had pore sizes ranging from 1.52 µm to 2.11 µm. It was also found that activation temperature significantly affected the percentage of COD and BOD removal compared to phosphoric acid concentration and activation time. The optimum adsorbent was a 75% PKS:25% EFB mixture, activated with 20 wt% H_3PO_4 at 70 °C for 30 min, achieving a COD removal of 64.0% along with a maximum BOD of 91.2%.

1. Introduction

Palm oil accounted for approximately 74.5% of Indonesia's primary plantation export commodities in 2021, covering a plantation area of 14.62 million hectares. According to data from the Indonesian Central Bureau of Statistics, Indonesia has become the world's largest producer of crude palm oil (CPO), producing approximately 48.2 million tons of CPO in 2022. In addition to producing CPO, the industry also generates substantial amounts of biomass, particularly from waste associated with palm oil milling activities. Every 1 ton of CPO yields approximately 1.50–1.64 tons of EFB, 0.4 tons of PKS, and 2.5 m^3 of POME [1], [2]. According to these data, it can be estimated that around 72.3–79 million tons of EFB/year, 19.3 million tons of PKS/year, and 120.5 million m^3 POME/year. If these wastes are not properly processed, they will generate large volumes of organic waste that can pollute the environment and harm aquatic life.

POME contains high organic components; on average, the final discharge of POME can contain up to 900 mg/L COD and 200 mg/L BOD [3]. Most palm oil mills process POME using an open pond

system because of its low cost and simple construction. The system necessitates an extended hydraulic retention time (up to 120 days) [4], [5]. This system also requires regular monitoring and stable maintenance to ensure it runs smoothly. According to the Regulation of the Minister of Environment of the Republic of Indonesia No. 5 of 2014, the BOD and COD values permitted before POME is discharged into water bodies are 100 mg/L and 350 mg/L, respectively. Many new and advanced technologies/systems on the market claim to be able to reduce the organic content in POME to meet standards. For example, ultrafiltration (UF), membrane bioreactors, conventional activated sludge, sequencing batch reactor, and coagulation processing [6], [7]. Although POME has been treated with various technologies, concerns persist because POME discharged into water bodies remains brownish, sometimes blackish, or is still visible in water bodies.

Another alternative for POME treatment is adsorption using activated carbon. This method is quite simple and produces high efficiency because the pores of the adsorbent have high adsorption capacity [8]. Activated carbon has been extensively utilized for the adsorption of color and pollutants from conventional and non-conventional wastewater. Activated carbon can be obtained from carbonaceous materials (carbonaceous materials), for example, coal, biomass derived from agricultural products, industrial waste, animal waste, and household waste [9]. Commercial activated carbon is obtained from non-renewable materials, which are relatively expensive, such as coal, which is an economic consideration.

This has led to increasing research interest in the production of low-cost activated carbon, particularly for wastewater treatment applications. In previous studies, the adsorption capability of activated carbon was evaluated using artificial pollutants, including methylene blue, phenol, nickel, and copper [10–13]. Only a few studies have stated the adsorption capability of activated carbon using industrial waste directly [6], [14].

While previous studies have explored oil palm biomass as a source of activated carbon, few have optimized the chemical activation parameters specifically for the remediation of raw POME using a statistical design of experiments. Therefore, this study aimed to determine the optimum activation process variables using EFB and PKS-based activated carbon for POME processing. The process variables to be optimized are activation temperature, activation time, and acid concentration, which serve as the activation agent. Unlike earlier works, this study employs a 23-factorial complete experimental design with one center point, providing a statistically rigorous methodology to systematically evaluate and optimize the interaction effects of multiple variables. This approach represents a novel application of factorial design in the context of POME treatment, ensuring that the resulting activated carbon is not only cost-effective but also customized for real industrial wastewater applications.

2. Research Methodology

Firstly, a summary of the experimental design is illustrated in Fig. 1. As shown in the diagram, it can be observed that activated carbon synthesis involves cleaning, carbonizing, grinding, sieving, and drying. The activation process was optimized in phosphoric acid concentration, temperature, and time. Activated carbon was used for POME adsorption, and then SEM, COD, and BOD were analysed.

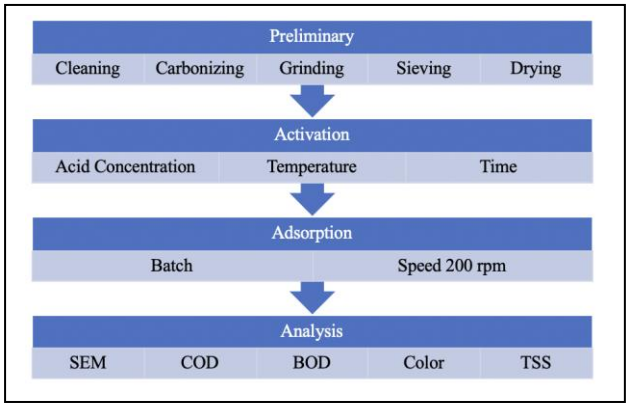


Fig. 1. Experimental design

The empty fruit bunch (EFB), palm kernel shell (PKS), and POME samples were supplied by the mini palm oil plant (as a teaching factory) at Politeknik Teknologi Kimia Industri Medan. The POME sample used for testing and analysis was obtained from the final discharge of the plant. The commercial activated carbon, as a comparative adsorbent, is derived from a local manufacturer. Phosphoric acid (H_3PO_4) (AR grade: 85% purity) was obtained from Sigma Aldrich.

2.1.Procedures

1) Synthesis of Biochar from Oil Palm Biomass

Empty fruit bunches and palm kernel shells were washed and dried at 105°C for 24 hours. Next, specifically for EFB, it was ground into particles with a diameter of 1–2 mm. EFB and PKS were then carbonized at 450°C for 2 hours using a furnace to produce biochar. The biochar was cooled at room temperature overnight. Next, it was weighed, ground, and sieved until the particles were less than 150 microns [15].

2) Activation Process for Activated Carbon Production

The dried biochar was chemically activated by being placed in a 500 mL beaker containing a phosphoric acid solution with a specific concentration (20–60% w/v). The mixture in the beaker was heated at 70–100°C for 30–60 minutes using a heat stirrer. Furthermore, the mixture was allowed to cool to room temperature for 2 hours [16], [17]. The activated carbon derived from EFB and PKS was then rinsed with distilled water three to four times until the pH reached seven and subsequently dried in an oven at 105°C for 12 hours. To determine the variables that influence the activation process, a two-factor experimental design will be employed, as presented in Table 1.

3) Batch Adsorption Study

The adsorption process was carried out in batches by mixing 0.75 g (2.5% w/v) of adsorbent (activated carbon) with 30 mL of POME. The mixture was stirred at 45°C and 200 rpm for 15 minutes. Mixing was carried out to ensure that the POME and activated carbon were mixed homogeneously, thereby maximizing the adsorption capacity of the mixture. The purification efficiency for all parameters was calculated as follows [2],[18].

$$\boldsymbol{Percentage\ removal} = \frac{\boldsymbol{C_0 - C_f}}{\boldsymbol{C_0}} \boldsymbol{x\ 100\%} \quad (1)$$

Where Co and Cf are POME concentrations at initial and final, respectively, the unit is mg/L for COD & BOD and Pt-Co for color.

Table 1. Experimental Design of a 2^3 Full Factorial Experiment with One Center Point

Run	Phosphoric acid concentration (%-w)	Temperature activation (°C)	Time (minute)
A1	20	70	30
A2	20	70	60
A3	60	70	30
A4	60	70	60
A5	20	100	30
A6	20	100	60
A7	60	100	30
A8	60	100	60
A9	40	85	45

4) Analytical Procedure

The surface morphology of the activated carbon was analysed at a micrograph magnification of 5000x using a Scanning Electron Microscope (SEM) (Nova NanoSEM 450). Standard methods were used to detect BOD, COD, TSS, and color. SNI 06-6989.14-2004 was employed to determine the BOD of the sample stored in a Winkler bottle by the iodometric titration method. While SNI 06-6989.2-2004 was employed to determine the COD of the sample using a UV-Vis spectrophotometer. TSS was measured in accordance with the standard SNI 06-6989.3-2004. A UV-Vis spectrophotometer was also used to detect color, following the procedure outlined in the SNI 6989.80-2011 standard method.

3. Results and Discussion

3.1. Effect of EFB-PKS Composition on Adsorption Performance

The formulation of the EFB and PKS mixture-based activated carbon was varied across five compositions, as illustrated in Fig. 2. The results revealed that the percentage of COD removal for pure EFB and PKS was still relatively low, reaching only 18.9% and 27.4%, respectively. This low COD removal percentage indicates that pure EFB and PKS-based activated carbon have limitations in absorbing organic and inorganic compounds contained in POME [19]. Based on Fig. 2, activated carbon produced from a mixture of EFB and PKS exhibits a higher adsorption capacity than activated carbon produced from pure EFB and PKS. EFB has a high lignocellulose content, while PKS contains more dominant lignin. The combination of the two creates a more varied pore structure, comprising micro-, meso-, and macropores, thereby increasing the adsorption capacity for molecules of various sizes [20]. The composition using more carbon from the shell (75% PKS and 25% EFB) has the best adsorption capacity, as proven by the highest COD removal percentage value.

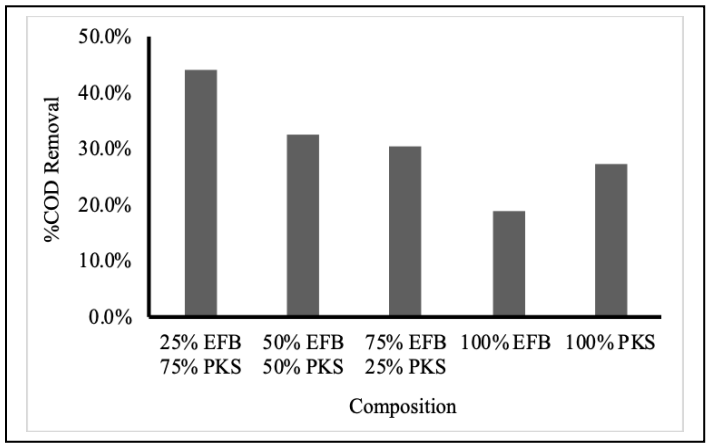


Fig. 2. Percentage of COD removal on various activated carbon compositions (Note: EFB-empty fruit bunch and PKS-palm kernel shell)

3.2. Optimization of Carbon Activation Conditions

Considering the data from the previous experiment, the effect of the activation conditions process will be analyzed using 0.75 g of activated carbon (2.5% w/v of POME) at the optimum composition, namely 25% EFB and 75% PKS. The adsorption experiments, which focused on optimizing the carbon activation conditions, were extended to 4 hours. A longer duration was applied in this case to allow equilibrium to be reached, ensuring that the effect of activation parameters on adsorption capacity could be evaluated without the limitation of diffusion. The operating conditions for activation were varied using a 2^3 complete factorial design with one center point (shown in Table 2). Based on Table 2, the higher the activation temperature and phosphoric acid concentration, the lower the percentage of COD and BOD removal obtained. The result of this study is consistent with the research in the literature [21]. Among the operating conditions tested, run A3 (60% phosphoric acid concentration, 70°C, 30 minutes) achieved the highest COD removal of 65.2%, while run A1 (20% phosphoric acid concentration, 70 °C, 30 minutes) achieved the highest BOD removal of 91.2%. Despite the slightly higher COD removal in run A3, run A1 was considered the optimal condition because it provided a more balanced performance with both high COD and the highest BOD removal. Therefore, the following experiment was conducted at a 20%-w phosphoric acid concentration and a temperature of 70°C for 30 minutes.

Analysis of variance using the Minitab program was conducted to investigate the main variables that affect the percentage of COD and BOD removal (illustrated in Fig.3.). Based on Fig. 3. (a), the most significant variable that effect regarding the reduction in the percentage of COD removal in succession are: temperature > interaction between temperature and phosphoric acid concentration > phosphoric acid concentration > time. Meanwhile, based on Fig. 3. (b), the key variables that influence the lower percentage of BOD removal are as follows: temperature > time > phosphoric acid concentration > interaction between phosphoric acid concentration and temperature.

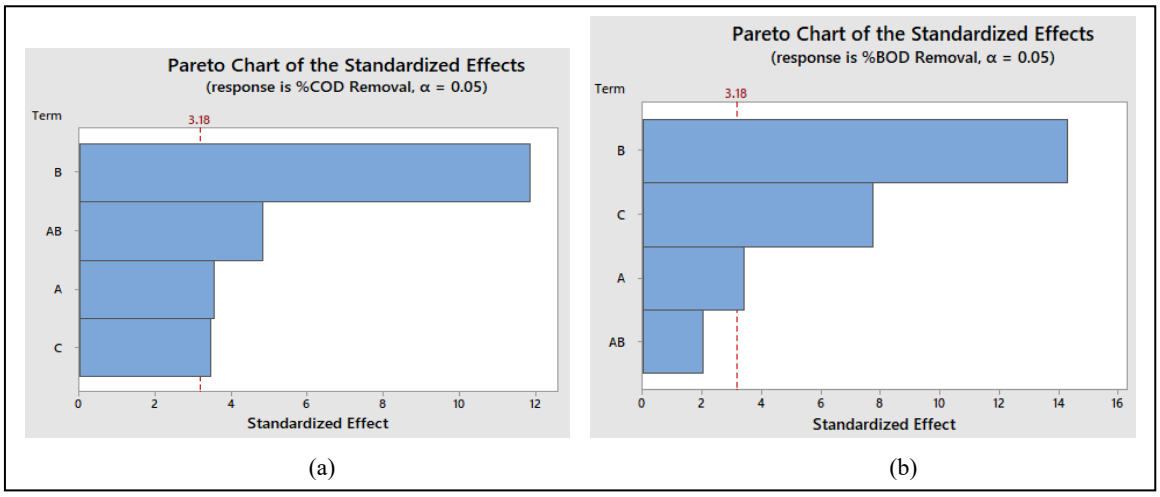
The regression models for COD and BOD removal demonstrated excellent fit, with R^2 values of 98.43% and 98.95%, respectively. The adjusted R^2 values (95.82% for COD and 97.19% for BOD) indicate minimal overfitting and confirm that the models explain nearly all variability in the responses. The lack-of-fit test (curvature $p > 0.05$) suggests that the linear model is adequate for describing the system. The equations model can be used to predict %COD removal and %BOD removal at various conditions.

$$\text{COD removal (\%)} = 161.5 - (1.042 \times \text{H}_3\text{PO}_4 \text{ Concentration}) - (1.258 \times \text{Temp}) - (0.2017 \times \text{Time}) + (0.01408 \times \text{H}_3\text{PO}_4 \text{ Concentration} \times \text{Tem} \quad (2)$$

$$\text{BOD removal (\%)} = 134.08 + (0.269 \times \text{H}_3\text{PO}_4 \text{ Concentration}) - (0.460 \times \text{Temp}) - (0.3467 \times \text{Time}) - (0.00450 \times \text{H}_3\text{PO}_4 \text{ Concentration} \times \text{Temp}) \quad (3)$$

Table 2. Adsorption Capability Using Synthetic Activated Carbon

Run	Phosphoric acid concentration (%-w)	Activation Temperature (°C)	Time (min)	%COD removal	%BOD removal
A1	20	70	30	64.0%	91.2%
A2	20	70	60	62.4%	79.5%
A3	60	70	30	65.2%	86.7%
A4	60	70	60	56.7%	80.3%
A5	20	100	30	38.5%	74.9%
A6	20	100	60	29.3%	62.8%
A7	60	100	30	51.0%	67.3%
A8	60	100	60	46.1%	55.9%
A9	40	85	45	52.1%	76.7%



(a) (b)

Fig. 3. Analysis of variance effects on (a) %COD removal and (b) %BOD removal using the Minitab program (Note: A-phosphoric acid concentration (%-wt); B-activation temperature (°C); and C-time (minutes))

Additionally, the impact of activation time on the color removal of POME was also analysed. The activation time was varied at a constant concentration of phosphoric acid and temperature, which were 20%-w and 70°C, respectively. The color of processed POME and the percentage of color removal are displayed in Fig. 4. As shown in Fig. 4, it can be observed that POME adsorbed using synthetic activated carbon is brighter/clearer compared to POME before adsorption (raw). This indicates that the adsorption process is running effectively. The percentage of color removal increases from 28 to 33% with increasing activation time. The increase in the percentage of color removal is probably attributed to the enhanced surface area. This is because the activator (phosphoric acid) can penetrate more deeply, thereby increasing the porosity of activated carbon at longer activation times. A larger surface area provides more sites for the adsorption process to occur [22].

Table 3. ANOVA Results for COD Removal

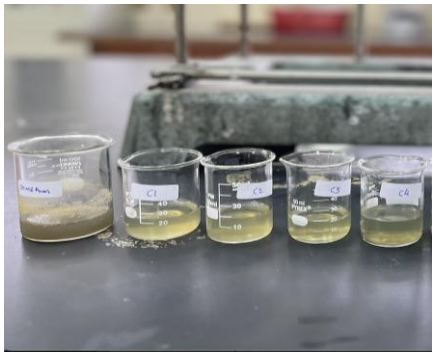
Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	5	1162.51	232.503	37.65	0.007
Linear	3	1019.53	339.843	55.04	0.004
A	1	76.88	76.880	12.45	0.039
B	1	869.44	869.445	140.8	0.001
C	1	73.20	73.205	11.86	0.041
2-Way Interactions	1	142.80	142.805	23.13	0.017
A*B	1	142.80	142.805	23.13	0.017
Curvature	1	0.18	0.18	0.03	0.875
Error	3	18.52	18.52	6.175	
Total	8	1181.04			

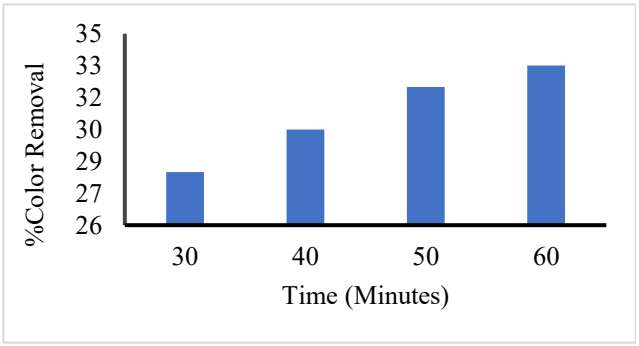
Model adequacy: R^2 = 98.43%, Adjusted R^2 = 95.82%, S = 2.485

Table 4. ANOVA Results for BOD Removal

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	5	1012.71	202.542	56.31	0.004
Linear	3	995.01	331.668	92.22	0.002
A	1	41.40	41.405	11.51	0.043
B	1	737.28	737.280	204.99	0.001
C	1	216.32	216.320	60.14	0.004
2-Way Interactions	1	14.58	14.580	4.05	0.138
A*B	1	14.58	14.580	4.05	0.138
Curvature	1	3.13	3.125	0.87	0.420
Error	3	10.79	3.597		
Total	8	1023.51			

Model adequacy: R^2 = 98.95%, Adjusted R^2 = 97.19%, S = 1.896





(a) (b)

Fig. 4. Analysis of the effect of activation time on (a) qualitative color changes, (b) percentage of color removal

The activated carbon synthesized in this study was compared with commercial activated carbon under identical adsorption conditions, including a 2.5% w/v dosage and a 15-minute contact time, as summarized in Table 3. The activated carbon used was a mixture of 25% EFB and 75% PKS-based activated carbon, which was activated at a concentration of 20%-w phosphoric acid and a temperature of 70°C for 30 minutes (variation A1). As shown in Table 5, it can be observed that the raw POME has BOD and TSS values that exceed the threshold set by the Regulation of the Minister of Environment of the Republic of Indonesia Number 5 of 2014 regarding Wastewater Quality Standards. All BOD, COD, and TSS levels of POME after the adsorption process using synthetic and commercial activated carbon have met the Regulation of the Minister of Environment of the Republic of Indonesia Number 5 of 2014. Synthetic activated carbon has a higher percentage reduction in COD and TSS compared to commercial activated carbon.

Table 5. Comparison of Synthetic and Commercial Activated Carbon Performance

Parameter	Initial POME	Treated POME with activated carbon		Threshold value*
		Commercial	*Synthetic*	
COD (mg/L)	342	277.2	228	350
BOD(mg/L)	175	46	80.8	100
TSS (mg/L)	458	135	98	200

Source: Regulation of the Minister of Environment of the Republic of Indonesia No. 5 of 2014

3.3.SEM Surface Analysis

In this study, SEM analysis was used to observe the surface morphology of EFB- and PKS-based activated carbon at a magnification of 5,000× (Fig. 5). The surface of the raw EFB and PKS was markedly different from that of the activated carbon. The raw materials showed irregular cavities partially covered by impurities, whereas the H_3PO_4-activated carbon displayed cleaner, fractured surfaces with large visible voids [23]. These visible voids (≈1–15 µm) represent macroporous channels and surface cracks produced by the release of volatiles during activation and are not the actual adsorption pores of activated carbon [24]. Activated carbon's primary adsorption sites are typically micropores (<2 nm) and mesopores (2–50 nm), which cannot be resolved at the 5,000× magnification used here. Therefore, the micrometer-scale dimensions reported (10.91–15.22 µm for activated carbon versus 1.52–2.11 µm for raw biomass) describe the size of these external surface cavities rather than the nanometer-scale pore network that provides high surface area. To fully characterize the actual microporous structure and pore size distribution, higher-magnification SEM (≥50,000×) or, preferably, nitrogen adsorption–desorption (BET) analysis is recommended.

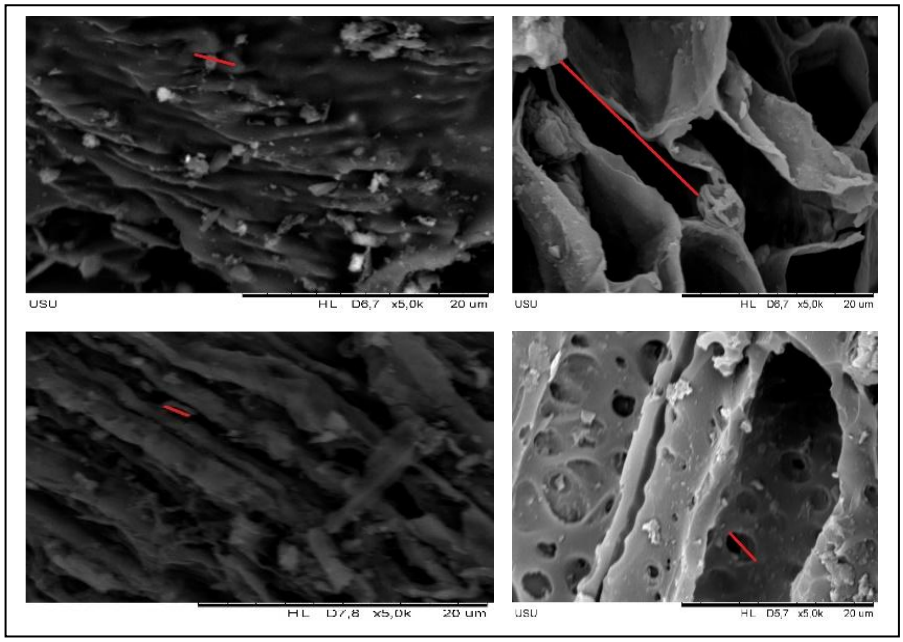


Fig. 5. Porosity images observed by SEM of raw PKS (upper left), PKS-based activated carbon (upper right), raw EFB (lower left), and EFB-based activated carbon (lower right)

3.4.Batch Adsorption

The impact of adsorption time on the percentage of COD removal is presented in Fig. 6. As shown in Fig. 6, the rate of COD removal increases with increasing adsorption time. The same trend is seen for all activated carbon masses that are varied. At 0–4 hours, the percentage of COD removal exceeds the increase in the subsequent adsorption time interval. This is likely because the contact surface of the activated carbon used is still large, resulting in a high absorption capacity. The longer the adsorption time, the longer organic and inorganic compounds can penetrate through the pores of the activated carbon. This indicates that the greater the number of pores that can be passed, the higher the absorption of POME with EFB and PKS-based activated carbon, and the greater the percentage of

COD removal [25]. The optimal adsorption time obtained was 4 hours. After 4 hours, the increase in COD removal percentage was no longer significant because the active site was nearly saturated.

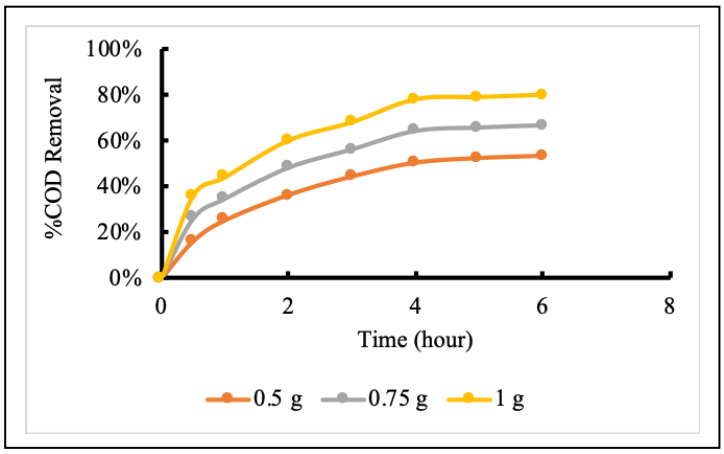


Fig. 6. Effect of adsorption time on percentage of COD removal

4. Conclusion

Activated carbon derived from empty fruit bunch (EFB) and palm kernel shell (PKS) was successfully synthesized and activated, as evidenced by the enlarged surface structures observed after activation. The optimal composition was 75 wt% PKS and 25 wt% EFB, with activation at 20 wt% phosphoric acid, 70 °C, and 30 min, achieving removals of 33.3% COD, 53.9% BOD, and up to 33% color. Among the studied variables, activation temperature exerted the most significant influence on COD and BOD reduction. Compared with commercial activated carbon, the synthesized material provided higher COD and TSS removal under identical adsorption conditions. The optimized activated carbon shows potential for use as a polishing treatment in POME systems. Future work should focus on quantifying microporous characteristics using BET analysis, continuous column adsorption studies, regeneration of spent AC, and a detailed economic analysis to assess scalability.

Acknowledgment

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