



Synthesis and Application of Activated Carbon from Oil Palm Fronds Biomass for One-Step Glycerol Purification

Fitri Rowiyah Rambe^{1,*}, Viqry Pramananda¹, Tubagus Rayyan Fitra Sinuhaji², Hari Pratama Sura¹, Muhammad Rifqi Ananda¹, Kelvin Hadinatan¹

¹Department of Chemical Engineering, Universitas Sumatera Utara, Medan 20115, Indonesia

²Department of Chemical Engineering, Diponegoro University, Semarang 50275, Indonesia

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ABSTRACT

This study aims to synthesize and apply oil palm frond activated carbon (OPFAC) as an adsorbent for one-step processing of crude glycerol. OPFAC was prepared through chemical activation using 10% of Na_2CO_3 followed by carbonization at 400°C . Several techniques, including SEM-EDX, FTIR, and SAA, were performed to evaluate its morphology, elemental composition, functional groups, and pore characteristics. In crude glycerol purification, the OPFAC dosage was varied at 1%, 3%, 5%, and 7% (w/w) relative to glycerol mass to determine its effect on purification performance. The initial crude glycerol purity of 50.64% increased progressively with increasing OPFAC dosage, reaching a maximum of 96.66% at 7%, which met SNI 06-1564-1995, BS 2621:1979, and classified as USP Degree II grade. FTIR analysis revealed the presence of methylene groups and aromatic C–H vibrations in OPFAC. Surface area analysis indicated a micropore surface area of $44.41 \text{ m}^2/\text{g}$ and a micropore volume of 0.0132 cc/g . The external mass-transfer coefficient (k_f) decreased from 1.32×10^{-4} to $3.78 \times 10^{-5} \text{ cm/min}$ with increasing adsorbent mass, consistent with film-diffusion theory and supported by previous studies. Overall, the results demonstrate that OPFAC is a promising, low-cost, and sustainable adsorbent capable of achieving high-purity glycerol through a simplified one-step process.

Keywords: Activated carbon; Adsorbent; External mass transfer coefficient; Glycerol; Oil palm fronds

1. Introduction

Glycerol ($C_3H_8O_3$) is a tri-hydroxyl alcohol obtained as a by-product of biodiesel production through the transesterification of vegetable oils or animal fats. High-purity glycerol is an important industrial feedstock due to its wide applications in pharmaceutical, cosmetic, food, and chemical industries [1]. Biodiesel production via transesterification typically generates approximately 10% (w/w) crude glycerol [2]. On a global industrial scale, crude glycerol production reached 4 billion liters in 2020 [3]. Another study reported that biodiesel production in the European Union (EU) increased from 3.7 million tons in 2006 to approximately 11 million tons in 2015, with a slight increase expected by 2050. This biodiesel production will yield over 1.2 million tons of crude glycerol [4].

The increasing volume of crude glycerol, driven mainly by the expansion of the biodiesel industry, has created new challenges related to handling, disposal, environmental impacts, and improper waste management, which may lead to contamination of aquatic and terrestrial ecosystems [2]. Besides direct disposal, another method for handling crude glycerol is incineration. However, uncontrolled incineration can produce unsaturated aldehydes, which are harmful to human health [5]. Furthermore, the presence of inorganic impurities in crude glycerol can trigger particulate emissions and contaminate the furnace during combustion [6]. Consequently, purification and valorization of crude glycerol are necessary to mitigate environmental risks and enhance its economic value.

Crude glycerol is a viscous brown liquid containing glycerol, alcohols (primarily methanol), free fatty acids, water, inorganic salts, and various non-glycerol organic impurities [7]. Current purification practices, whether at laboratory or industrial scale, still rely on multiple pretreatment steps such as acidification, neutralization,

evaporation, and other auxiliary procedures. These steps require additional chemicals, equipment, and processing time, contributing to higher operational costs. Therefore, the development of a one-step purification process without pretreatment is of considerable interest, as it offers lower chemical consumption, shorter processing times, and reduced equipment requirements [8].

Adsorption is a well-established separation technique capable of effectively removing organic and inorganic impurities to enhance product purity. Among various adsorbents, activated carbon is widely recognized for its efficiency due to its large surface area, well-defined pore structure, thermal stability, and low acid–base reactivity. These properties enable its extensive use in pollution control, solvent recovery, food processing, chemical and pharmaceutical manufacturing, wastewater treatment, metal recovery, catalysis, and aroma enhancement [9]. In the context of crude glycerol purification, several studies have utilized activated carbon derived from waste materials, including sludge [10], oil palm empty fruit bunches [11], durian peels [12], and passion fruit peels [8].

Recent advances in activated carbon synthesis have focused on utilizing biomass-based waste materials. Oil palm fronds represent a promising precursor due to their abundance in oil palm plantations, with an estimated production of 5,500 kg/ha/year [13]. In other literature, it is stated that the oil palm fronds production in Indonesia in 2015 reached 124,032,861 tons [14]. The chemical composition of oil palm fronds comprises 31.5% cellulose, 19.2% hemicellulose, and 14% lignin [13]. Maulina et al. [15] characterized oil palm fronds activated carbon (OPFAC) using Na_2CO_3 as an activator, reporting a moisture content of 8.80%, ash content of 2.20%, volatile matter of 14.80%, fixed carbon of 68.60%, and an iodine adsorption capacity of 888.51 mg/g. These characteristics highlight the strong

potential of OPFAC, both material availability and adsorptive performance.

Based on this background, a preliminary study was conducted to synthesize and evaluate OPFAC as an adsorbent for one-step glycerol purification. The objectives of this study were: (i) to characterize the OPFAC, and (ii) to investigate the effect of OPFAC dosage on glycerol purity. In addition to purity enhancement, the external mass transfer coefficient (k_f) was calculated to provide deeper insight into the one-step purification process. To the best of our knowledge, the use of OPFAC for crude glycerol purification—whether in a single-step or multi-step scheme—has not been previously reported, offering a novel contribution and distinguishing this work from existing studies.

2. Materials and Methods

2.1 Materials and equipment

This study utilized oil palm fronds collected from the Universitas Sumatera Utara campus area, together with chemical reagents including Na_2CO_3 , NaOH , NaIO_4 , H_2SO_4 , ethylene glycol, distilled water, and bromothymol blue indicator, while the crude glycerol used was obtained as a byproduct from a previous biodiesel production study employing coconut oil as the feedstock and coconut husk ash as a heterogeneous catalyst [16]. In general, impurities contained in crude glycerol originating from the transesterification reaction can be in the form of residual color pigments, residual methanol, free fatty acids, water, and matter organic non-glycerol matter (MONG).

The equipment comprised a blender, 32-mesh sieve, furnace, desiccator, hot plate and magnetic stirrer, as well as a set of glassware. Characterization of the OPFAC was conducted using a Scanning Electron Microscope–Energy Dispersive X-Ray (SEM-EDX, Thermo Scientific), Fourier Transform Infrared Spectroscopy (FTIR,

Perkin), and a Surface Area Analyzer (SAA, Quantachrome Novatouch Lx4).

2.2 Synthesis of OPFAC

The synthesis of OPFAC is referred to the study by Maulina et al. [15] with minor modification. The oil palm fronds were ground and sieved to a particle size of 32 mesh. The resulting powder was then activated in a 10% Na_2CO_3 solution with a powder-to-activator ratio of 1:10 (w/v). Activation was carried out for 2 hours at 80°C , followed by impregnation at room temperature for 24 hours. The mixture was then filtered, and the solid was dried in an oven and carbonized in a furnace at 400°C for 1 hour. The resulting OPFAC was subsequently characterized for its morphology and elemental composition using SEM-EDX, functional groups using FTIR, as well as surface area and pore volume using SAA.

2.3 Application of OPFAC in glycerol purification

The OPFAC and crude glycerol were placed into a beaker, and the mixture was stirred using a magnetic stirrer at 250 rpm while being heated at 60°C for 90 minutes. The mixture was then allowed to stand at room temperature for 1 hour and subsequently filtered.

Purity analysis of the glycerol was carried out with reference to Indonesian National Standard (SNI) 06-1564-1995, as also applied by Misran et al. (2025). A total of 0.1 g of glycerol was dissolved in 10 mL of distilled water and one drop of bromothymol blue indicator was added. The mixture was acidified with 0.2 N H_2SO_4 until a yellowish-green color was obtained. Subsequently, 10 mL of 10% NaIO_4 was added to the mixture and stirred gently. The mixture was then covered and kept in a dark room at ambient temperature for 30 minutes. Afterward, 2 mL of ethylene glycol was added, followed by covering and standing again in the dark at ambient temperature for

20 minutes. The mixture was then diluted with 60 mL of distilled water, three drops of bromothymol blue indicator were added, and the solution was titrated slowly with 0.5 N NaOH until a blue color appeared. The same procedure was performed for the blank titration. The glycerol purity was calculated using Eq. (2.1).

$$\text{Purity level (\%)} = \frac{(V_1 - V_2) \times N \times 9.209}{w} \times 100\%, \quad (2.1)$$

where V_1 and V_2 refer to the volume of NaOH (in mL) for sample titration and blank titration (by employing aquadest), respectively, N is the normality of NaOH (0.5 N), 9.209 refers to glycerol factor (m_{eq} of glycerol $\times 100$ to adjust for percentage), and w is glycerol mass (g).

2.4 Calculation of external mass transfer coefficient (k_f)

In batch adsorption, adsorbate absorption occurs through two mechanisms: intraparticle diffusion and external mass transfer. A well-stirred adsorption batch results in rapid mixing within the liquid phase. As a result, the concentrations of both adsorbate and adsorbent are uniform throughout the adsorption process [17]. This condition forms the basis for determining the external mass transfer coefficient (k_f). Generally, k_f can be calculated using Eq. (2.2) based on the adsorption data obtained at various adsorbent masses.

$$k_f = -\frac{V}{A_p} \left[\frac{d\left(\frac{C_t}{C_0}\right)}{dt} \right]_{t=0}. \quad (2.2)$$

V refers to volume of solution (mL), A_p is the external surface area of the adsorbent (cm^2), C_t and C_0 are the adsorbate concentration at time t and initial condition (in % unit), respectively. In this study, adsorbate is all impurities in the crude glycerol, calculated as 100% minus the glycerol purity ($C_t = 100\% - \text{glycerol purity}$).

The A_p value is obtained from the surface area analysis using SAA.

The general and linearized form of the correlation for the k_f value can be represented by Eqs. (2.3)-(2.4), where A and B are constants and w is the mass of OPFAC used. These approaches are also used by McKay et al. [17] in dye adsorption using bagasse pith and by Misran et al. [8] in crude glycerol purification using passion fruit peel-based activated carbon.

$$k_f = A \log(w)^B, \quad (2.3)$$

$$\log k_f = \log A + B \log(w). \quad (2.4)$$

3. Results and Discussion

3.1 Characteristics of OPFAC

3.1.1 Morphology and Elemental Composition

Fig. 1 presents the OPFAC along with its morphology observed using SEM at magnifications of 5,000 times (1b) and 20,000 times (1c). In Fig. 1b, the surface of the OPFAC appears non-homogeneous with a coarse texture, and several darker regions are distributed across certain areas. These dark regions indicate the formation of pores or cavities on the OPFAC surface. During activation, volatile compounds on the material surface are released, allowing the carbon pores to open [18]. The uneven surface becomes more apparent when the OPFAC morphology is magnified up to 20,000 times.

Furthermore, EDX analysis reveals several elements contained in OPFAC, as summarized in Table 1. OPFAC is dominated by carbon (40.2%), followed by oxygen (36.2%) and sodium (20.7%). The relatively high sodium content is attributed to the 24-hour impregnation step in the OPFAC synthesis procedure, which allows a considerable amount of sodium to adhere to the surface of the oil palm fronds. Furthermore, Na_2CO_3 will decompose during carbonization to form sodium metal and CO.

These sodium metal atoms can enter the carbon structure, thereby widening existing

pores and even creating new pore structures [18].

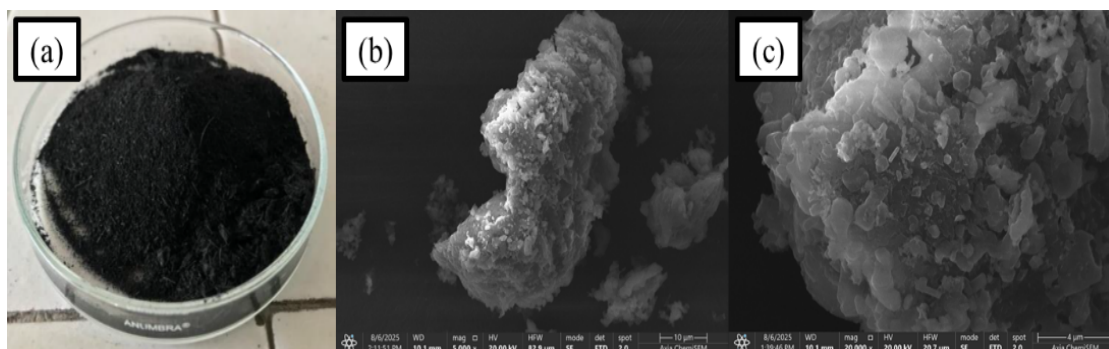


Fig. 1. (a) Appearance of OPFAC; (b) OPFAC morphology at 5,000 times magnification; (c) OPFAC morphology at 20,000 times magnification.

Table 1. Elemental composition in OPFAC.

Element	Composition (wt. %)
C	40.2
N	1.9
O	36.2
Na	20.7
Mg	0.1
K	0.1
Ca	0.8

3.1.2 Functional groups

Fig. 2 presents the infrared spectra of oil palm fronds and OPFAC. Oil palm fronds have more absorption peaks than OPFAC, indicating the presence of more functional groups. The broad absorption band between 3650 cm^{-1} and 3250 cm^{-1} indicates the presence of hydrogen-bonded groups in oil palm fronds, which can be hydroxyl (-OH), hydrate (H_2O), ammonium, or amino acids [19]. The absorption at 2917.77 cm^{-1} and 2850.27 cm^{-1} refers to the aliphatic stretching (C-H) groups, which are commonly found in polysaccharides [20]. An absorption peak at 1735.62 cm^{-1} indicates the presence of carbonyl groups (C=O). Generally, carbonyl groups are characterized between 1850 cm^{-1} and 1650 cm^{-1} [19]. The absorption band at 1463.71 cm^{-1} indicates methylene groups ($>\text{CH}_2$), which is characterized between $1485\text{--}1445\text{ cm}^{-1}$ [19]. The absorption peak at 1033.66 cm^{-1} indicates the presence of compounds with cyclohexane rings, which

are generally marked at 1005 to 925 cm^{-1} [19].

On the other hand, the broad absorption of hydrogen bonding, as well as aliphatic stretching groups, are no longer visible in the OPFAC spectrum, indicating those bonds were lost during activation and carbonization. A sharp peak appears at 1472 cm^{-1} indicating the methylene groups. Additional peaks detected at 879 cm^{-1} and 701.5 cm^{-1} correspond to aromatic C-H out-of-plane bending, which is generally characterized in the range of $900\text{--}670\text{ cm}^{-1}$ [19, 21].

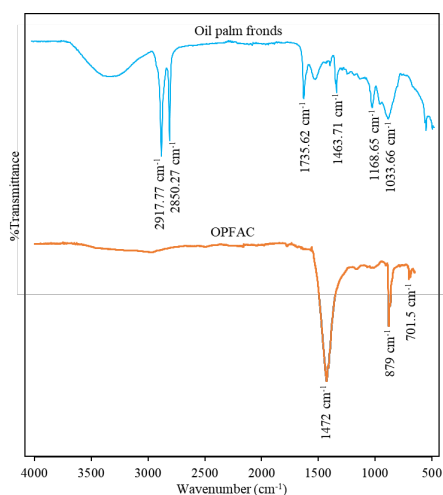


Fig. 2. Infra-red spectra of oil palm fronds and OPFAC.

3.1.3 Surface area and pore volume

Characterization of porous materials using surface area analysis (SAA) generally involves two main analytical methods for determining their specific surface area, namely the Brunauer–Emmett–Teller (BET) method and the t -plot method (where t denotes thickness). The BET method describes the volumetric adsorbate uptake (V_A) as a function of relative pressure (P/P_0), the BET constant (C), and the monolayer volume (V_m). In contrast, the t -plot method is based on comparing the adsorption isotherm of a porous adsorbent with a reference isotherm of a chemically similar material that is ideally non-porous. This method is used to determine micropore volume and specific surface area, assuming flat surfaces during multilayer adsorption as well as sequential micropore filling and capillary condensation (22). Therefore, the BET and t -plot methods can complement each other, providing more comprehensive information about a material's surface area.

In this study, characterization using the BET method yielded a specific surface area of $0.000 \text{ m}^2/\text{g}$ for OPFAC. This indicates the inability of the BET method to produce a positive linear regression within the relative pressure range employed for the characterization ($P/P_0 = 0.05\text{--}0.30$). This issue arises from the highly microporous structure of OPFAC, which prevents the formation of a linear region within the pressure range required for BET analysis. The sole concept of a “BET monolayer capacity” is inadequate for microporous materials, as the monolayer has no clear physical or theoretical meaning in such systems [23]. As a conclusion, the BET method cannot accurately detect the surface area of the microporous structure in the OPFAC, resulting in a surface area reading of $0.000 \text{ m}^2/\text{g}$ (as if the OPFAC had no pores at all).

Conversely, the t -plot method successfully provided valid estimates, yielding a micropore surface area of 44.408

m^2/g and a micropore volume of $0.0132 \text{ cm}^3/\text{g}$. Accordingly, the synthesis method applied in this study—particularly the 24-hour impregnation step at room temperature—proved effective in generating a microporous structure in OPFAC.

3.2 Effect of OPFAC dose on the glycerol purity improvement

Fig. 3 shows the effect of OPFAC dosage on the improvement of glycerol purity. Initially, the glycerol had a purity of 50.64%. The adsorption process successfully enhanced the glycerol purity progressively with increasing OPFAC mass. The highest purity (96.66%) was achieved using an OPFAC dosage of 7%. This purity level exceeds the minimum standards specified in SNI No. 06-1564-1989 and British Standard (BS) 2621:1979, which require a minimum purity of 80%. A similar trend was also reported by Misran et al. [8] in the glycerol purification by adsorption using passion fruit peel-activated carbon (PFPAC), where increasing the PFPAC dosage to 11% progressively increased the glycerol content from 64.46% to 96.69%.

In general, glycerol is classified into three categories based on its purity level. The first category is Degree I (technical grade), with a purity of ~96%. This type of glycerol is used in the chemical industry but is not suitable for use in food or beverage applications. The second category is Degree II (USP grade), with a purity ranging from 96% to 99.5%. This grade is appropriate for use in food, pharmaceuticals, and cosmetic products. The final category is glycerol with the highest purity level, 99.5–99.7%, known as Kosher grade. This type of glycerol meets Kosher certification standards [24]. Accordingly, the glycerol purified using OPFAC in this study can be classified as Degree II glycerol.

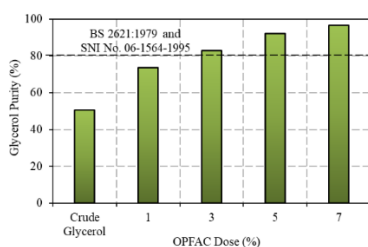


Fig. 3. Glycerol purity at various OPFAC dosage.

Table 2 compares our findings with previous studies, particularly those that used activated carbon for crude glycerol purification. Only our study and the one by Misran et al. [8] used a single-step (adsorption) process for glycerol purification, while other studies employed pretreatment such as acidification, neutralization, evaporation, and drying before the adsorption stage. Multi-stage processes can remove many impurities from

glycerol, so adsorption serves only as the final step to remove any remaining impurities. For example, the study by Tambun et al. [12] used a multi-stage process prior to adsorption, so the specific performance of the durian peel-activated carbon synthesized for purifying glycerol is unknown. On the other hand, a single-stage approach with adsorption can reduce costs and operational time, making it highly suitable for industrial applications.

Our results are also very similar to the findings of Misran et al.[8]; however, we use a lower adsorbent dosage of 7% (compared to 11% by Misran et al.), resulting in lower operational costs. Overall, OPFAC demonstrates superior performance and significant potential for single-stage glycerol purification, requiring further development for industrial-scale application

Table 2. Comparison to previous work.

Adsorbent	Purification Stage	Adsorbent Dose (%wt)	Glycerol Purity (%)			Ref.
			Crude Glycerol	Before Adsorption	After Adsorption	
Commercial activated carbon	• Acidification • Adsorption	10	39.10	-	64.90	(25)
Durian peel activated carbon	• Acidification • Neutralization • Evaporation • Adsorption	25	58.21	-	96.26	(12)
Sludge-derived activated carbon	• Acidification • Neutralization • Drying	1 g in 15 ml CG	27.20	35.70	93.00	(10)
Pasion fruit peel activated carbon	• Adsorption	11	64.46	64.46	96.69	(8)
OPFAC	• Adsorption	7	50.64	50.64	96.66	This work

3.3 External mass transfer coefficient

Fig. 4 presents the adsorption profile of purities at various OPFAC masses. The adsorption profile (expressed as C/C_0) decreases with increasing OPFAC mass. This indicates that the amount of OPFAC influences the transfer of adsorbates (impurities) to the adsorbent film layer. A similar phenomenon was reported by Guiza et al. [26] in fluoride adsorption using activated silica, where the adsorption kinetics increased with higher activated silica dosage. In the context of glycerol purification using

activated carbon, Misran et al. [8] also observed a decrease in impurity adsorption profiles with increasing doses of activated passion fruit peel carbon.

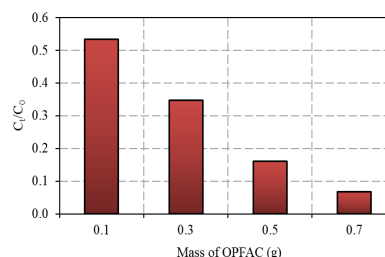


Fig. 4. C/C_0 profile.

Fig. 5 presents the correlation between the log OPFAC mass with the log k_f to obtain the constants A and B through a linear regression equation. The k_f values, as well as the constants A and B, are summarized in Table 2. Increasing the OPFAC mass results in lower k_f values. The linear regression equation shown in Fig. 5 exhibits a coefficient of determination (R^2) of 0.9969, indicating that the linear regression model accurately represents the external mass-transfer mechanism. These results align with the findings of McKay et al. [17], who analyzed k_f values in dye adsorption using bagasse and reported that increasing bagasse mass led to lower k_f values. Misran et al. [8] also reported that increasing activated carbon dosage in glycerol purification reduced the k_f value.

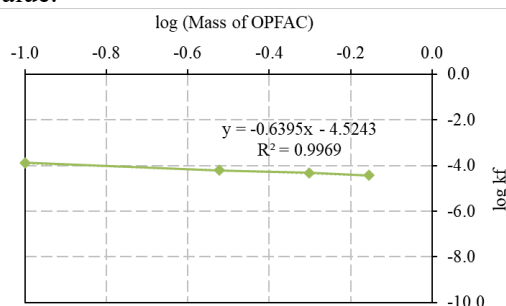


Fig. 5. log mass of OPFAC vs log k_f .

Table 2. The Results of External Mass Transfer Coefficient (k_f).

OPFAC Dosage (%)	k_f (cm/min)	A	B
1	1.32×10^{-4}	0.0108	-0.6395
3	6.18×10^{-5}		
5	4.77×10^{-5}		
7	3.78×10^{-5}		

Based on the regression relationship between log mass of OPFAC and log k_f , the slope $B = -0.6395$ was obtained. The negative value of B confirms that increasing the OPFAC mass leads to a decrease k_f . This result is consistent with the experimental data, where k_f decreased from 1.32×10^{-4} to 3.78×10^{-5} cm/min as the OPFAC mass increased. The decrease in k_f indicates that increasing the adsorbent mass enhances the external diffusion resistance. As the amount

of adsorbent increases, both the number of particles and the total external surface area also increase. At the same time, however, a thicker particulate layer is formed, which reduces turbulence and increases the thickness of the boundary layer (film layer) surrounding the particle surfaces. The increased film thickness intensifies mass-transfer resistance, resulting in a lower k_f value.

These findings demonstrate that increasing the OPFAC mass is not always beneficial in adsorption processes, particularly when the objective is to enhance the rate of external mass transfer. This condition is important to consider in adsorption system design, especially in batch reactors or packed-bed systems, where mass-transfer efficiency significantly affects the overall adsorption rate. While using a larger adsorbent mass may improve total adsorption capacity, it can slow the initial adsorption rate due to the decline in k_f .

4. Conclusion

This study demonstrates that OPFAC synthesized through Na_2CO_3 activation followed by carbonization at 400°C can be employed as an effective adsorbent for one-step glycerol purification. A series of characterization techniques confirmed the potential of OPFAC as an efficient adsorbent, evidenced by the presence of active functional groups and a microporous structure. Batch adsorption experiments showed that increasing the OPFAC dosage consistently improved glycerol purity, reaching a maximum of 96.66% at a dosage of 7% (w/w). This purity level satisfies both the Indonesian National Standard (SNI) and the British Standard, and can be classified as Degree II glycerol.

The obtained external mass-transfer coefficient (k_f) exhibited an inverse relationship with adsorbent mass, with k_f decreasing from 1.32×10^{-4} to 3.78×10^{-5} cm/min. This decline indicates that higher adsorbent dosages increase external

diffusion resistance due to the formation of a thicker particulate layer and an expanded boundary-layer film.

Overall, OPFAC is shown to be an inexpensive, environmentally friendly, and effective adsorbent capable of enhancing glycerol quality through a single-step process. These findings highlight the potential for further development, including optimization of adsorption conditions and implementation in industrial-scale systems such as continuous reactors or packed-bed configurations. From a practical standpoint, the post-use management of OPFAC should also be considered; regeneration through thermal reactivation or solvent washing could enable multiple reuse cycles, thereby improving process sustainability and cost efficiency. Alternatively, spent OPFAC may be valorized as a solid fuel or safely disposed of following appropriate environmental guidelines, minimizing waste generation and supporting circular resource utilization.

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