



Transformasi Limbah Pertanian Menjadi Microsponge Adsorben Silika–Selulosa untuk Optimasi Minyak Jelantah sebagai Bahan Baku Biodiesel

Transformation of Agricultural Waste into Silica–Cellulose Microsponge Adsorbent for Optimization of Waste Cooking Oil as Biodiesel Feedstock

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ABSTRAK

Pemanfaatan minyak jelantah sebagai bahan baku biodiesel terkendala oleh tingginya kadar asam lemak bebas yang menghambat proses transesterifikasi. Penelitian ini bertujuan untuk menurunkan FFA melalui metode adsorpsi menggunakan microsponge berbasis silika–selulosa yang disintesis dari sekam padi dan bonggol jagung. Variabel proses yang dikaji meliputi % massa adsorben (1, 3, 5), suhu (30, 45, 60 °C), waktu kontak (30, 60, 90 menit), serta rasio silika:selulosa (40:60, 50:50, 60:40). Analisis FTIR menunjukkan adanya pita karakteristik Si–O–Si (~1053 cm⁻¹) dan Si–O (~795 cm⁻¹) yang mengarah pada keberadaan silika amorf, sedangkan pita O–H (~3200–3500 cm⁻¹) dari selulosa tampak melemah akibat dominasi silika pada permukaan. Hal ini menunjukkan keberhasilan pembentukan komposit silika–selulosa dengan interaksi kimia yang baik. Selanjutnya, hasil uji Brunauer-Emmett-Teller (BET) menunjukkan luas permukaan spesifik 87,77 m²/g dengan karakteristik pori meso, menegaskan struktur microsponge yang berafinitas tinggi terhadap molekul polar seperti FFA. Kondisi optimum diperoleh pada massa adsorben 5%, suhu 30 °C, waktu 60 menit, dan rasio 60:40, dengan penurunan FFA sebesar 49,62%. Karakteristik fisik dan kimia tersebut menjadikan adsorben ini efektif, hemat energi, dan ramah lingkungan, sekaligus menunjukkan potensi besar transformasi limbah pertanian sebagai material aktif untuk pra pemurnian minyak jelantah menuju produksi biodiesel berkelanjutan.

Kata kunci: Adsorpsi, Asam lemak bebas (FFA), Biodiesel berkelanjutan, Minyak jelantah, Silika–selulosa

ABSTRACT

The utilization of used cooking oil as a biodiesel feedstock is hindered by its high free fatty acid (FFA) content, which interferes with the transesterification process. This study aims to reduce FFA levels through adsorption using a silica–cellulose-based microsponge adsorbent synthesized from rice husk ash and corn cob waste. The investigated process variables include % mass adsorbent (1, 3, 5), temperature (30, 45, 60 °C), contact time (30, 60, 90 minutes), and silica-to-cellulose ratios (40:60, 50:50, 60:40). FTIR analysis revealed characteristic peaks of Si–O–Si (~1053 cm⁻¹) and Si–O (~795 cm⁻¹) indicating the presence of amorphous silica, while the O–H stretching (~3200–3500 cm⁻¹) of cellulose appeared weaker due to silica dominance on the surface. This confirms the successful formation of the silica–cellulose composite with strong chemical interaction. Furthermore, Brunauer–Emmett–Teller (BET) analysis showed a specific surface area of 87.77 m²/g with mesoporous characteristics, confirming the microsponge structure with high affinity toward polar molecules such as FFA. The optimum conditions were obtained at 5% wt adsorbents, 30 °C, 60 minutes contact time, and a 60:40 silica-to-cellulose ratio, achieving an FFA reduction efficiency of 49.62%. These physicochemical properties make the adsorbent efficient, energy-saving, and environmentally friendly, highlighting the great potential of agricultural waste transformation into active materials for pre-purification of used cooking oil toward sustainable biodiesel production.

Keywords: Adsorption, Free fatty acid (FFA), Silica–cellulose, Sustainable biodiesel, Waste cooking oil

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INTRODUCTION

Indonesia's high dependence on fossil energy amid declining reserves and increasing emissions has driven renewable energy targets of 23% by 2025 and 31% by 2050 (Pertamina, 2020; Kementerian ESDM, 2014). Biodiesel from low-cost waste such as used cooking oil is a promising alternative (Haryanto et al., 2015); however, high free fatty acid (FFA) content reduces biodiesel yield, requiring pretreatment in accordance with SNI 7182:2015 (Cerón Ferrusca et al., 2023; Sipayung & Budiyono, 2022). Adsorption offers a simple and environmentally friendly pretreatment method (Syauqiah et al., 2011), while agricultural biomass such as corn cobs and rice husks provides low-cost, porous, and silica-rich adsorbents that support waste valorization and emission reduction (Fatah et al., 2021; Okoro et al., 2022).

Previous studies have shown that rice husk ash reduces the FFA content of used cooking oil from 0.46% to 0.23% (Pasaribu et al., 2023), while maize cob charcoal achieves up to 38.44% reduction (Bavaresco et al., 2021). However, conventional adsorbents are limited by low surface area and poor structural stability, leading to the development of highly porous microsp sponge materials with good thermal stability (Manique et al., 2012). Therefore, this study aims to develop and evaluate a silica-cellulose microsp sponge derived from agricultural waste to enhance FFA removal from used cooking oil while supporting waste valorization and sustainable energy goals.

MATERIALS AND METHODS

The research involved was carried out experimentally in the laboratory, including used cooking oil as the test sample and agricultural wastes (rice husk and corn cob) as the raw materials for adsorbent synthesis. The chemicals used were NaOH, HCl, glutaraldehyde, technical ethanol, distilled water, and phenolphthalein indicator. The major equipment included an oven, furnace, grinder, 100-mesh sieve, analytical balance, hot plate stirrer, Buchner funnel, filter paper, desiccator, and characterization instruments like FTIR and BET analyzers. Figure 1 illustrates all of the research procedure as a flow diagram.

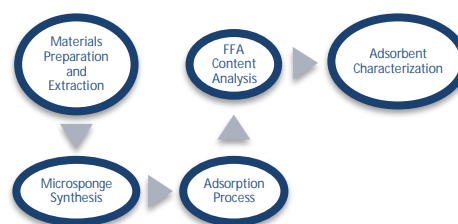


Figure 1. Research Framework of Adsorbent for Optimization of Biodiesel Feedstock

Materials Preparation and Extraction

Rice husk was first cleaned and oven-dried at 110 °C for 3 h, followed by calcination at 500 °C for 4 h to obtain rice husk ash. Silica was extracted by dissolving the ash in 2 M NaOH at a solid-to-liquid ratio of 1:10 (w/v) and heating at 100 °C for 1 h. The resulting solution was precipitated with 2 M HCl under stirring for 1 h until the pH reached 7–9. The precipitate was then washed and dried to obtain silica powder (Figure 2).



Corn cobs were treated with 4% NaOH at 85 °C for 2 h to extract cellulose, followed by washing to neutral pH, drying at 60 °C for 4 h, grinding, and sieving through a 100-mesh screen. The cellulose extraction process is shown in Figure 3.

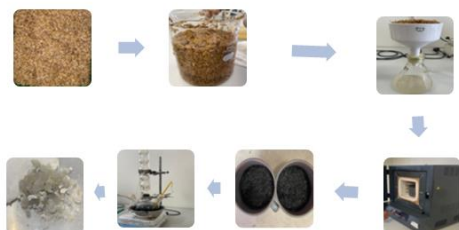


Figure 2. Flow Chart of Procedure Used for Silica Extraction



Figure 3. Flow Chart of Procedure Used for Cellulose Extraction

Microsponge Synthesis

The obtained silica and cellulose were mixed in mass ratios of 40:60, 50:50, and 60:40 (w/w). The mixture was added to an ethanol–water solvent (50:50 v/v) at a solid-to-solvent ratio of 8:1 (w/v) and stirred for 30 minutes. A 2.5% (v/v) glutaraldehyde solution was added as a crosslinking agent, and the crosslinking reaction was carried out at 65 °C for 6 hours. The resulting product was then dried and activated in a furnace at 180 °C for 6 hours to obtain the silica–cellulose adsorbent.

Adsorption Process

Prior to treatment, the used cooking oil was filtered for approximately 10 minutes using Whatman No. 1 filter paper (pore size ~11 µm) to remove suspended solids and then heated at 100 °C for 30 minutes to reduce moisture content. Batch adsorption experiments used 10 g of used cooking oil in each trial. The experiments tested different conditions, including adsorbent amounts of 1%, 3%, and 5% (w/w), temperatures of 30, 45, and 60 °C, and contact times of 30, 60, and 90 minutes, while stirring constantly at 300 rpm. Once the contact time was over, the mixture was filtered again using the same filter paper to remove the adsorbent.

FFA Content Analysis

The free fatty acid (FFA) content was determined using the acid-base titration method, with 0.02 N NaOH and phenolphthalein as the indicator, following the AOAC standard method (2005). The FFA percentage was then calculated using the following equation:

$$\% \text{FFA} = \frac{V_{\text{NaOH}} \times N_{\text{NaOH}} \times 28.2}{W_{\text{sample}}} \quad (1)$$

where V_{NaOH} is the titrant volume (mL), N_{NaOH} is NaOH normality, and W_{sample} is the oil sample mass (g). Once the FFA percentage was obtained from the titration, the reduction in FFA was calculated to evaluate the adsorption efficiency using:

$$\% \text{FFA Reduction} = \frac{\text{FFA}_{\text{initial}} - \text{FFA}_{\text{final}}}{\text{FFA}_{\text{initial}}} \times 100\% \quad (2)$$

Adsorbent Characterization

The prepared adsorbents were analyzed to verify their structure and physical properties.



FTIR analysis was employed to identify key functional groups, such as -OH, Si-O-Si, and C-O-C, which indicate the presence of silica and cellulose. BET analysis was carried out to evaluate the adsorption potential of the materials by examining their specific surface area and pore size distribution. This approach to characterization is commonly used in studies of biomass-based porous materials (Saleh & Gupta, 2022; Wibawa et al., 2020; Song et al., 2023).

RESULTS AND DISCUSSION

The results showed that adsorbent mass, temperature, contact time, and the silica-to-cellulose ratio significantly affected the reduction of free fatty acids (FFA) in used cooking oil. As presented in Table 1 and Figures 4–6, analysis using the Box–Behnken design within the Response Surface Methodology (RSM) revealed nonlinear interactions among these factors. The combined variations of silica to cellulose ratio, temperature, and adsorbent mass enhanced FFA removal efficiency. This analysis also helped determine the optimal conditions and clarified the contribution of each parameter to the overall adsorption process.

Table 1. FFA Adsorption Results for Used Cooking Oil

| No | % wt adsorbent | t (min) | T °C | Silica: Cellulose | FFA Reduction (%) |
|----|----------------|---------|------|-------------------|-------------------|
| 1 | 1 | 60 | 30 | 40:60 | 41.67 |
| 2 | 1 | 60 | 45 | 40:60 | 25.76 |
| 3 | 1 | 60 | 60 | 40:60 | 20.45 |
| 4 | 5 | 60 | 30 | 40:60 | 39.02 |
| 5 | 5 | 60 | 45 | 40:60 | 31.06 |
| 6 | 5 | 60 | 60 | 40:60 | 20.45 |
| 7 | 1 | 30 | 45 | 50:50 | 33.71 |
| 8 | 3 | 30 | 30 | 50:50 | 33.71 |
| 9 | 3 | 30 | 60 | 50:50 | 15.15 |
| 10 | 5 | 30 | 45 | 50:50 | 28.41 |
| 11 | 1 | 60 | 30 | 50:50 | 17.80 |

| | | | | | |
|----|---|----|----|-------|-------|
| 12 | 1 | 60 | 60 | 50:50 | 23.11 |
| 13 | 3 | 60 | 45 | 50:50 | 23.11 |
| 14 | 3 | 60 | 45 | 50:50 | 20.45 |
| 15 | 3 | 60 | 45 | 50:50 | 15.15 |
| 16 | 5 | 60 | 30 | 50:50 | 33.71 |
| 17 | 5 | 60 | 60 | 50:50 | 31.06 |
| 18 | 1 | 90 | 45 | 50:50 | 23.11 |
| 19 | 3 | 90 | 30 | 50:50 | 20.45 |
| 20 | 3 | 90 | 60 | 50:50 | 20.45 |
| 21 | 5 | 90 | 45 | 50:50 | 20.45 |
| 22 | 1 | 60 | 30 | 60:40 | 17.80 |
| 23 | 1 | 60 | 45 | 60:40 | 23.11 |
| 24 | 1 | 60 | 60 | 60:40 | 31.06 |
| 25 | 5 | 60 | 30 | 60:40 | 49.62 |
| 26 | 5 | 60 | 45 | 60:40 | 15.15 |
| 27 | 5 | 60 | 60 | 60:40 | 44.32 |

Increasing the adsorbent mass from 1 to 5 %wt led to a substantial rise in the percentage of FFA removal. At 30 °C and a silica-to-cellulose ratio of 40:60, FFA reduction increased from 17.80% to 39.02%, likely due to the higher number of available active sites, which enabled more FFA molecules to be adsorbed. This observation supports the concept that adsorption capacity is strongly related to the availability of active sites (Foo & Hameed, 2012) and is consistent with the findings of Putri et al. (2021), who reported that lignocellulosic adsorption efficiency increases with adsorbent dosage until saturation is reached.

Temperature also influenced the adsorption performance. At 30 °C, using a silica-to-cellulose ratio of 60:40 and 5%wt of adsorbent, FFA removal reached 49.62%, whereas increasing the temperature to 60 °C reduced the removal efficiency to 44.32%. This decrease at higher temperature suggests that the adsorption of FFA onto the silica–cellulose microsp sponge is predominantly exothermic, where elevated temperatures may weaken adsorbate–adsorbent interactions and promote partial desorption of FFA molecules from the active sites. Similar temperature-dependent behavior has been widely reported



for physical adsorption systems (Foo & Hameed, 2010).

Figure 4 shows that increasing both the silica content and the adsorbent dose significantly enhances FFA reduction, with the optimal conditions occurring at a 60% silica composition and 5%wt adsorbent, resulting in an efficiency of approximately 49–50%. This improvement can be attributed to the increased specific surface area and the higher number of available active sites provided by the silica-rich microsphere structure, which enhance the interaction between the adsorbent surface and FFA molecules. Beyond the optimal condition, a decrease in adsorption efficiency was observed, which may be caused by particle agglomeration, leading to a reduction in the effective contact area between the oil phase and the adsorbent surface.

The silica-to-cellulose ratio of 60:40 achieved the highest FFA reduction (49.62%) compared to 40:60 (41.67%), indicating a favorable balance between surface area contribution from silica and the presence of polar functional groups (such as –OH) from the lignocellulosic component that facilitate interaction with FFA. Similar enhancement in adsorption performance has been reported for silica–biopolymer and silica–lignocellulosic composites, where improved porosity and surface polarity play a key role in strengthening adsorbate–adsorbent interactions (Foo & Hameed, 2012; Miri et al., 2023; Rengga et al., 2020).

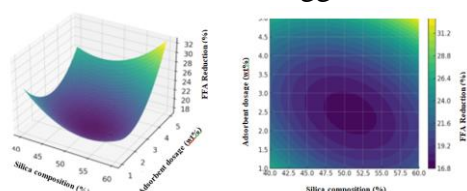


Figure 4. The implications of Composition and Adsorbent Dosage (%) on FFA decrease.

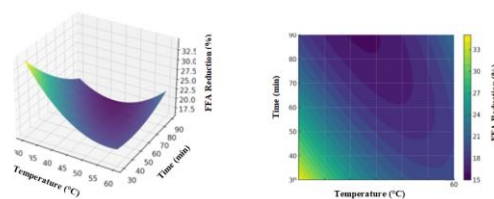


Figure 5. The implications of Temperature and Time on FFA decrease.

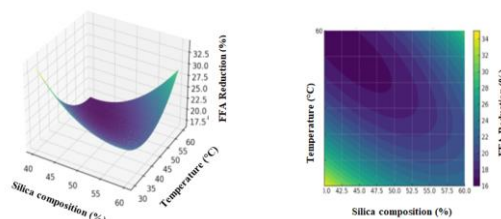


Figure 6. The implications of Composition and Temperature on FFA decrease.

Figure 5 illustrates the combined effect of temperature and contact time on FFA reduction. Based on the experimental data (Table 1), relatively high FFA reductions were already achieved at 30 °C, such as 41.67% (run 1) and 39.02% (run 4), indicating that effective adsorption can occur at low temperatures. An increase in temperature from 30 to 60 °C generally enhances molecular mobility, which facilitates the diffusion of FFA molecules toward the adsorbent surface; however, further improvement in FFA reduction was not consistently observed at 60 °C. Contact time also plays a significant role. At 30 minutes, FFA reduction values were lower (e.g., 15.15–33.71%) compared to those obtained at 60 minutes, suggesting that diffusion and occupation of active sites were not yet complete. The highest FFA reduction (49.62%) was achieved at 60 minutes, after which no substantial improvement was observed at longer contact times (90 minutes), indicating that adsorption equilibrium had been reached. This trend is consistent with Ahmad et al. (2019), who reported that the optimal contact time for FFA adsorption using activated carbon was approximately 60 minutes.



Figure 6 shows that combining a high silica concentration with a moderate temperature produces the largest reaction surface. This highlights the role of silica as a source of active sites, while a temperature around 60 °C promotes diffusion without compromising the microporous structure. Adsorption efficiency decreases when silica concentration is lower or temperatures are higher, due to dehydration of active hydroxyl groups and reduced stability of the mesoporous structure. FTIR analysis shows absorption bands of Si-O-Si ($\sim 1053\text{ cm}^{-1}$) and O-H ($\sim 3200\text{--}3500\text{ cm}^{-1}$), confirming the dominance of active silica groups on the adsorbent surface and indicating optimal performance at the 60:40 ratio.

The FTIR characterization results shown in Figure 7 indicate chemical interactions between silica and cellulose, which are further supported by Brunauer-Emmett-Teller (BET) analysis to examine the pore properties of the adsorbent. As illustrated in Figure 8, the silica-cellulose adsorbent with a 60:40 ratio exhibits a specific surface area of $87.77\text{ m}^2/\text{g}$, a C constant of 100.64, and a correlation coefficient of 0.999872, indicating good agreement with the BET model and strong interactions between nitrogen molecules and the adsorbent surface.

The nitrogen adsorption isotherm shows a notable increase in adsorbed volume at relative pressures between 0.05 and 0.3, indicating a mesoporous structure (2–50 nm) suitable for accommodating FFA molecules ($\sim 2\text{ nm}$). This mesoporous structure facilitates free diffusion and enhances adsorption efficiency. The addition of cellulose contributes to forming a three-dimensional hollow structure resembling a sponge (microsponge), while silica provides thermal stability and surface activity.

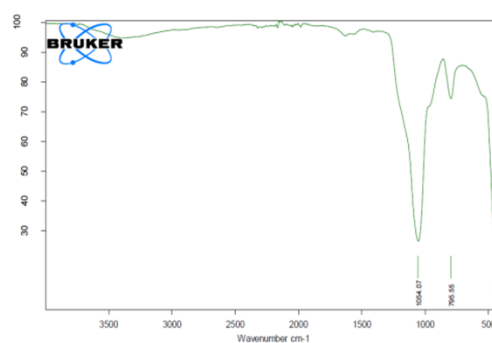


Figure 7. FTIR Spectrum of Silica-Cellulose Adsorbent (60:40)

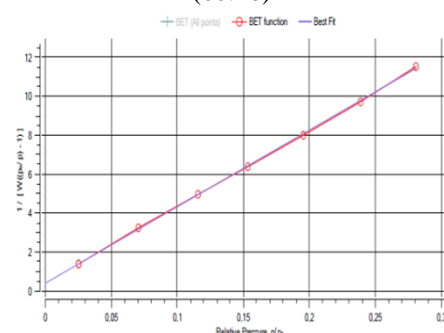


Figure 8. BET Analysis Results of Silica-Cellulose Adsorbent (60:40)

The FTIR and BET results indicate that the 60:40 silica-cellulose adsorbent possesses both chemically active surfaces and a mesoporous structure, which together enhance FFA adsorption. The large surface area provides numerous adsorption sites, while the polar surface properties improve interactions with the carboxyl groups of FFA molecules. Additionally, the adsorbent demonstrates good thermal stability after 720 minutes of degassing at 100 °C, making it suitable for adsorption processes at the typical temperatures of waste cooking oil (60–70 °C).

The composite adsorbent derived from rice husk silica and corncob cellulose exhibits a mesoporous microsponge structure with a high surface area and strong affinity for polar molecules. Optimal conditions were achieved at 5 %wt adsorbent, a temperature of 30–60 °C, a contact time of 60 minutes, and a silica-to-cellulose ratio of 60:40, resulting in 49.62% FFA reduction. These combined physical and chemical properties make the adsorbent effective, environmentally friendly, and



promising for waste cooking oil purification in sustainable biodiesel production using local biomass.

CONCLUSIONS

This study demonstrates that adsorbent mass, temperature, contact time, and silica-to-cellulose ratio significantly affect FFA reduction in waste cooking oil. Optimal conditions were achieved at 5%wt adsorbent, 30 °C, 60 minutes, and a 60:40 silica-to-cellulose ratio, resulting in 49.62% FFA removal. BET analysis confirmed a mesoporous microsphere structure with a specific surface area of 87.77 m²/g, indicating strong affinity for polar FFA molecules. These characteristics highlight the potential of rice husk-silica and corncob-cellulose adsorbents as an effective, energy-efficient, and environmentally friendly pretreatment for sustainable biodiesel production.

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