



H₂O₂-Modified Geopolymers as Integrated Adsorbent-Catalysts for FFA Removal and Biodiesel Production from Crude Palm Oil

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ABSTRACT

This study investigated hydrogen peroxide (H₂O₂)-modified geopolymers as integrated adsorbent catalysts for biodiesel production from crude palm oil (CPO). Geopolymers were synthesized using metakaolin and modified with varying H₂O₂ concentrations (0-30 wt%). Material characterization through XRD, FTIR, XRF, and BET analysis showed that H₂O₂ modification enhanced porosity and surface area, with modified samples showing a 13-fold increase in specific surface area (9.7068 m²/g) compared to unmodified geopolymer (0.7312 m²/g). The optimal H₂O₂-modified geopolymer (Gpm-3) demonstrated superior performance in separated and combined processes. The separated process achieved 85.38% FFA removal efficiency and 100% biodiesel yield. The combined single-step process maintained impressive performance with 55% FFA removal and 96.47% biodiesel yield. These results highlight the potential of H₂O₂-modified geopolymers as cost-effective, sustainable materials for integrated FFA removal and biodiesel production from CPO.

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1. INTRODUCTION

The global demand for renewable energy and sustainable production systems has driven significant research on biodiesel production, particularly from renewable feedstocks such as Crude Palm Oil (CPO). Biodiesel production, primarily through transesterification, is hindered by the presence of Free Fatty Acids (FFAs) in CPO, which adversely affect the process by inducing saponification and reducing biodiesel yield [1-5]. Removal of FFAs is both an essential preprocessing step and a focal point for improving biodiesel synthesis efficiency, with adsorbent-catalyst systems serving as integrated, multifunctional solutions [6-8].

Geopolymers, a class of inorganic polymers with three-dimensional aluminosilicate structures, have attracted increasing interest as versatile materials for adsorption and catalysis applications because of their tunable porosity, chemical resistance, and thermal stability [9, 10]. The modification of geopolymers using hydrogen peroxide (H₂O₂) is a novel approach for enhancing their pore structure and improving their adsorption properties. The incorporation of H₂O₂ during geopolymer synthesis induces in situ foaming, which creates micro- and mesopores by releasing oxygen gas, leading to enhanced surface area and accessibility [11, 12]. Several studies have demonstrated that the degree of porosity in H₂O₂-modified geopolymers can be controlled through precursor composition and synthesis conditions, such as Si/Al ratio, H₂O₂ concentration, and curing temperature, enabling applications that demand highly porous materials [13-16].

Despite their structural advantages, the use of H₂O₂-modified geopolymers in adsorption processes for Free Fatty Acid (FFA) removal from Crude Palm Oil (CPO) has not been extensively explored. Previous studies have focused on FFA adsorption using materials like magnesium silicate [17], activated carbon [18], and clay-based sorbents [19]. These materials exhibit moderate FFA adsorption efficiency but are limited by challenges such as higher regeneration costs, limited tunability of pore structures, and potential environmental drawbacks. However, H₂O₂-modified geopolymers offer the scalability of synthesis and configurability of both hydrophilic and hydrophobic surface properties, making them promising candidates as adsorbents [16-20].

In addition to adsorption, the functionalization of geopolymers with active catalytic groups, such as sulfonic acid (–SO₃H) or phosphoric acid (–PO₃H₂), has gained attention for enabling simultaneous catalysis and adsorption [12, 21]. Such bifunctional systems are designed to first adsorb FFAs and then catalyze their conversion into methyl esters (biodiesel) via esterification, reduce the number of pre-processing steps, and improve the sustainability of biodiesel production [22, 23]. Studies employing conventional acid catalysts, such as Amberlyst-15 and carbon-zeolite composites, have achieved comparable outcomes in catalysis and operational stability [24, 25]. However, combining these functionalities with materials designed for biodiesel production, particularly geopolymers modified for optimized porosity and recyclability, remains an underexplored domain [26].

H₂O₂-modified geopolymers are particularly interesting candidates for this integration because of their ability to achieve high specific surface areas and tailored pore-size distributions. Porosity enhancements in metakaolin-based geopolymers synthesized with H₂O₂, achieving a surface area up to 94 m²/g and mesopore volume that significantly improved adsorptive capabilities [11]. However, these studies focused on inert adsorption applications, such as heavy metal removal, and failed to address reactive systems, such as FFAs, in biodiesel precursors. Similarly, studies on the catalytic performance of other geopolymer systems have shown promising results in heavy oil upgrading and other catalytic reactions [27, 28]. However, their suitability for biodiesel application remains unclear.

The stability and recyclability of adsorbent catalysts are also critical parameters for economic viability. Conventional materials, such as zeolites and commercial acid catalysts, often suffer from performance degradation during repeated cycles because of fouling, loss of active sites, and structural collapse under reaction conditions [29, 30]. Although H₂O₂-treated geopolymers exhibit promising recyclability in structural applications, comprehensive studies evaluating their reusability in adsorption-catalysis systems for FFAs in CPO and biodiesel reactions are lacking [12, 16].

The advantage of geopolymers over traditional materials lies in their eco-friendly production from industrial by-products (e.g., fly ash and metakaolin) and significantly lower material costs [31, 32]. Integrating H₂O₂-modified geopolymer systems into biodiesel production pipelines can provide a low-cost and scalable pathway for simultaneous FFA removal and biodiesel catalysis. However, extensive experimental validation is necessary to achieve practical implementation, with a specific focus on optimizing the pore structure and functional catalytic sites and ensuring long-term stability over multiple regeneration cycles [16, 20, 31].

This study explored the potential of H₂O₂-modified geopolymers as integrated adsorbent-catalyst materials for removing FFAs from CPO and catalyzing biodiesel production. It addresses critical gaps in pore structure optimization, dual-functionality implementation, and recyclability testing to evaluate the feasibility of these materials as sustainable alternatives to conventional adsorbent-catalyst systems.

2. METHODS

2.1. Chemicals and Materials

Raw materials for geopolymer synthesis such as sodium hydroxide (NaOH, pellet, 98%), sodium silicate solution (containing 26.5% SiO₂, 10.6% Na₂O), hydrogen peroxide (H₂O₂, 30 wt%, analytical grade) and methanol (CH₃OH, analytical grade, 99.8%) were purchased from Merck. Crude Palm Oil (CPO) with 1-5 wt% Free Fatty Acid (FFA) content was obtained from PT. Pundi Lahan Khatulistiwa, West Kalimantan Province, Indonesia. Metakaolin produced from kaolin was obtained from Capkala District, Bengkayang Regency, West Kalimantan, Indonesia.

2.2. Synthesis of H₂O₂-Modified Geopolymers

The modified geopolymer was prepared according to previous research [33]. Preparation of Geopolymer Paste. Dissolve sodium hydroxide pellets in deionized water to prepare an 8 M NaOH solution, then mix with sodium silicate in a 2:3 weight ratio to create the activator solution. Combine the activator solution with metakaolin in a solid-to-liquid ratio of 2:1 and mix thoroughly for 10 minutes to form a homogenous geopolymer paste. Add hydrogen peroxide (H₂O₂) into the paste at three different concentrations (0 wt%, 5 wt%, 10 wt%, 15 wt%, 20 wt%, and 25 wt% relative to total paste weight) and mix for 2–3 minutes to ensure uniform foaming. Cast the H₂O₂-treated paste into molds and cure initially at room temperature for 24 hours, followed by thermal curing at 70°C for 8 hours. H₂O₂ levels varying between 0 and 30% by mass of the binder were used (see **Table 1**).

2.3. Material Characterization

The pore size was determined using SSA (Quantachrome Novatouch Lx4) (see **Table 2**). The geopolymer pattern was determined using X-ray diffraction (XRD) (PANalytical XPERT-PRO). The chemical compositions of metakaolin and geopolymer were determined by X-ray fluorescence (PANalytical Epsilon 3).

Table 1. Concentration of hydrogen peroxide solution.

Metakaolin (%)	NaOH (%)	Na ₂ SiO ₃ (%)	Concentration of H ₂ O ₂
44	4	52	0%
44	4	52	5%
44	4	52	10%
44	4	52	15%
44	4	52	20%
44	4	52	25%
44	4	52	30%

Table 2. The result of BET analysis and comparison with previous research.

Sample	Specific Surface Area (m ² /g)	Total Pore Volume (cm ³ /g × 10 ⁻²)	Average Pore Diameter (nm)	Ref
Metakaolin	0.1591	0.00411	5.171	
Unmodified Geopolymer	0.7312	0.0824	2.255	
H ₂ O ₂ -Modified Geopolymer	97.068	30.281	6.239	
Metakaolin	0.014 - 1.259	N/A	N/A	[34,35]
Unmodified Geopolymer	0.690 - 1.260	0.0067 - 0.120	2.100 - 3.600	[34-36]
H ₂ O ₂ -Modified Geopolymer	8.910 - 10.600	2.980 - 3.120	5.960 - 6.200	[35-37]

2.4. Adsorption of FFAs in Crude Palm Oil

Prepare a fixed amount of CPO (~20 g). Heat the CPO to 60°C in a batch reactor equipped with a magnetic stirrer to simulate typical pre-treatment conditions. Add 1 g of H₂O₂-geopolymer into the oil and stir for 1–3 hours at a constant speed (~200 rpm). Compare adsorption efficiency across geopolymer samples with varying H₂O₂ concentrations (0 wt%, 5 wt%, 10 wt%, 15 wt%, 20 wt% and 25 wt%). Analyze the FFA concentration using the titration method. Dissolve oil samples (2 g) in ethanol containing phenolphthalein indicator and titrate using a 0.1 M NaOH solution. Calculate FFA removal efficiency using equation (1).

$$\text{FFA removal efficiency \%} = \frac{C_0 - C_t}{C_0} \times 100\% \quad (1)$$

C_0 = Initial FFA concentration

C_t = FFA concentration after adsorption

2.5. Catalysis of Biodiesel Production

Place the FFA-reduced CPO (after the adsorption step) into a three-neck glass reactor equipped with a condenser and magnetic stirrer. Add methanol to the oil in a molar ratio of 20:1 (methanol: FFA) and load 5% of the modified geopolymer as a catalyst into the system. Operate the reactor at 65°C for 3 hours under continuous stirring (200 rpm). After the reaction, cool the product mixture and separate the phases by gravity. Extract biodiesel (Fatty Acid Methyl Ester, FAME) and quantify it using a Gas Chromatography-Flame Ionization Detector (GC-FID). FAME yield (%) is calculated using equation (2):

$$\text{FAME content \%} = \frac{\text{mass of FAME calculated by GC-MS analysis}}{\text{Mass of biodiesel phase analyze}} \times 100\% \quad (2)$$

2.6. Integration of Adsorption and Catalysis

Fresh CPO (20 g) directly into the batch reactor operated at 60°C. Simultaneously perform FFA adsorption and esterification by adding 2 g of H₂O₂-modified geopolymer. Add methanol to the system at a 20:1 molar ratio (methanol: FFA) and operate for 1-3 hours at 65°C under

constant stirring. Collect samples of the reaction mixture at 1-hour intervals to track FFA reduction and FAME formation. Plot FFA concentration (%FFA) and FAME yield (%) over time to evaluate the kinetics of the integrated process. Compare the efficiency of the combined system with separate adsorption and esterification steps.

3. RESULTS AND DISCUSSION

3.1. X-ray Diffraction (XRD) Analysis

Figure 1 shows the diffractograms of metakaolin raw materials, unmodified geopolymer, and modified geopolymer. The X-ray diffractogram of the modified geopolymer exhibits three prominent peaks at 2θ values of 26.61° , 32.33° , and 50.13° , corresponding to crystalline phases typically found in metakaolin-based geopolymer systems. The peak at 26.61° is commonly attributed to quartz (SiO_2), a remnant crystalline phase present due to incomplete dissolution of the metakaolin precursor during polymerization, which has been similarly reported in previous studies [3, 15]. The peak at 32.33° can be linked to either nepheline-like or sodalite-type phases (Na-Al-Si structures), which represent newly formed crystalline phases developed during polymerization under strongly alkaline conditions and are indicative of a partially crystallized geopolymer matrix [16]. The third peak at 50.13° is often associated with derivative or secondary quartz or other silicate crystalline phases, reflecting the persistence of unreacted aluminosilicate material and crystalline structures distributed within the amorphous polymeric network [15, 17].

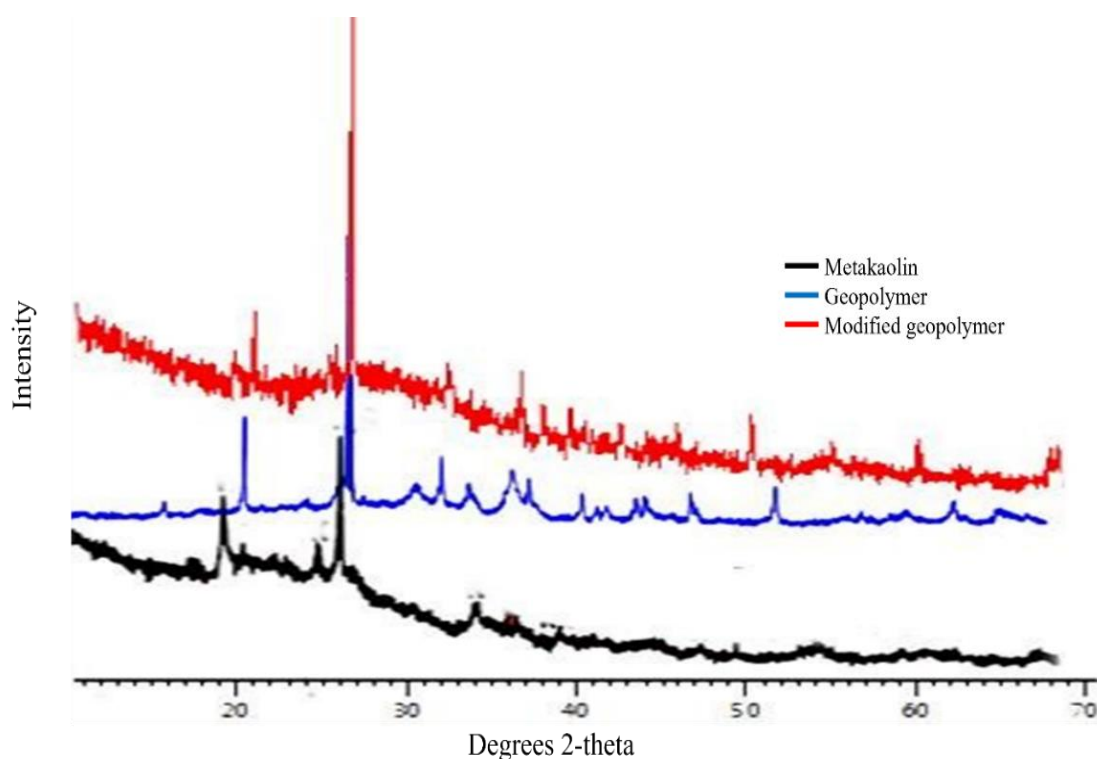


Figure 1. Diffractogram from metakaolin, geopolymer, and modified geopolymer.

The XRD data confirm that the geopolymer structure largely retains an amorphous background with embedded crystalline phases, as evidenced by the broad humps typically observed in the region of $15\text{--}35^\circ$ in 2θ , combined with the presence of specific sharp peaks. This partial crystallinity is in line with the known characteristics of hydrogen peroxide-modified geopolymers, where pore-forming agents such as H_2O_2 influence the degree of structural reorganization, potentially contributing to uneven polymerization reactions during

curing [6, 13]. Comparable peak intensities and positions have been reported in the literature for metakaolin geopolymers incorporating silica-rich additives or those modified for enhanced porosity [3, 16, 17]. It is noteworthy that peaks associated with H₂O₂-modified systems may differ slightly in intensity and phase formation due to the potential redistribution of silicate species within the geopolymer matrix during oxygen bubble formation [6].

3.2. Fourier Transform Infrared (FTIR) Spectroscopy Analysis

FTIR spectroscopic analysis (Figure 2) of the modified geopolymer revealed several characteristic absorption bands, indicating successful geopolymerisation. The FTIR spectrum of the modified geopolymer reveals key vibrational bands that are characteristic of the geopolymer framework and surface chemistry. The primary peaks at 982.64 cm⁻¹ and 1000 cm⁻¹ correspond to the asymmetric stretching vibrations of Si-O-Si and Si-O-Al bonds, which are indicative of the aluminosilicate network formation during polymerization [38, 39]. These values are consistent with other studies on geopolymers where the presence of these vibrational modes confirms the successful polymerization of the aluminosilicate source through alkali activation.

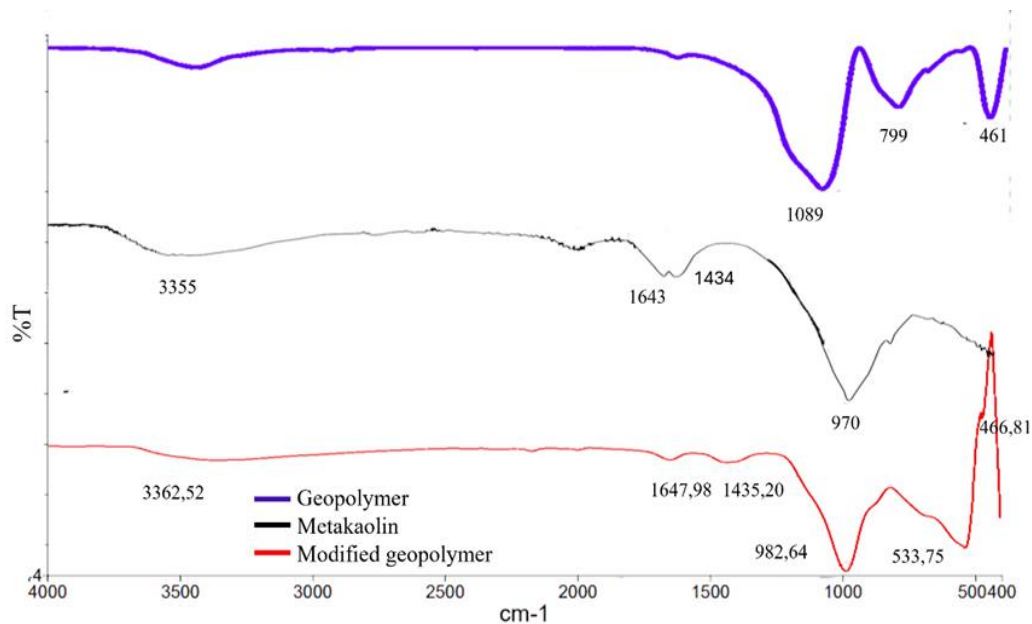


Figure 2. FTIR Spectrum from metakaolin, geopolymer, and modified geopolymer.

Additionally, the peaks at 466.81 cm⁻¹ and 533.79 cm⁻¹ are attributed to the bending vibrations of Si-O-Si bonds [40]. These modes reflect the integrity of the characteristic three-dimensional network structure typically observed in geopolymers. The band at 3362 cm⁻¹ corresponds to O-H stretching vibrations, while the peak at 1647 cm⁻¹ represents H-O-H bending vibrations from adsorbed water molecules or hydroxyl groups in the geopolymer matrix [38]. These results indicate the retention of physically adsorbed water and the presence of structural hydroxyl groups, which are key contributors to surface hydrophilicity and may play a role in adsorption processes, particularly in materials designed for polar adsorbates such as free fatty acids (FFAs).

The presence of a band at 1435 cm⁻¹ is a signature of carbonate (CO₃²⁻) groups, typically formed via carbonation reactions when the geopolymer is exposed to atmospheric CO₂ during curing [40]. The incorporation of carbonate species is common in geopolymer systems and does not detract from the material's functionality; instead, it often enhances surface reactivity by introducing additional active sites [41].

These spectral features are consistent with those observed in other modified geopolymers synthesized with an emphasis on enhancing adsorption potential through structural modifications. For example, studies have shown that the incorporation of pore-forming agents or modifiers like hydrogen peroxide can enhance the presence of hydroxyl groups and porosity, contributing to better adsorption performance [42]. This FTIR analysis confirms the successful formation of a geopolymer system with a well-developed aluminosilicate structure, hydroxyl functionality, and surface characteristics suitable for adsorption applications, including those involving FFAs.

3.3. X-Ray Fluorescence (XRF) Analysis

The X-ray fluorescence (XRF) data (see **Table 3**) for the modified geopolymer exhibits significant differences in elemental composition when compared to previously studied geopolymers based on other precursors, including metakaolin [43], blast furnace slag [44], and kaolin [45]. The silica (Si) content of the modified geopolymer ranges from 71.38 to 73.94%, which is higher than that of blast furnace slag-based geopolymers (25.63–43.24%) and kaolin-based geopolymers (63.82%) while falling within the reported range for metakaolin-based geopolymers (51.4–92.3%). The aluminum (Al) content of the modified geopolymer (17.34–19.75%) is relatively consistent with kaolin-based geopolymers (29.14%) and some metakaolin-based geopolymers (5.2–28.5%) but markedly higher than slag-based systems (6.88–15.63%). These findings reflect the influence of precursor type and synthesis modifications in the Si/Al composition. This Si/Al ratio falls within the optimum range for structural stability and effective crosslinking in geopolymer networks, where higher silica content contributes to improved mechanical performance, thermal resistance, and tailored adsorption properties as supported by prior studies [37, 46].

Table 3. Metallic composition of modified geopolymer catalysts.

Element	Geopolymer modified	Geopolymer base metakaolin [47]	Geopolymer base blast furnace slags [44]	Geopolymer base kaolin [45]
Si	71.38-73.94%	51.4-92.3%	25.629-43.238%	63.82%
Al	17.34-19.75%	5.2-28.5%	6.882-15.631%	29.14%
Fe	1.53-1.87%	-	1.110-1.214%	1.32%
Ti	1.04-2.15%	-	-	13.61%
K	1.55-2.83%	-	-	-
Si/Al ratio	3.62-4.23	1.51-14.88	~2.5-3.7	2.08

The calculated Si/Al ratio for the modified geopolymer is 3.62–4.23, indicating a silica-rich composition that is critical for enhanced network crosslinking and stability in adsorption and mechanical applications. This Si/Al ratio is notably higher than that of kaolin-based geopolymers (2.08) and some blast furnace slag-based systems (~2.5–3.7) while slightly overlapping with the broad range of ratios reported for metakaolin-based geopolymers (1.51–14.88). The iron (Fe) and titanium (Ti) contents in the modified geopolymer (Fe: 1.53–1.87%, Ti: 1.04–2.15%) are trace elements not commonly emphasized in slag- or kaolin-based geopolymers, but they can impart functional properties, such as enhanced adsorption or redox capabilities. The potassium (K) content (1.55–2.83%) is also consistent with alkali activation processes, contributing to the ion exchange capacity and adsorption efficiency of the material.

These observations suggest that the modified geopolymer has a tailored aluminosilicate structure with high silica content, an optimized Si/Al ratio, and minor constituents contributing to its functionality. Compared to other precursor systems, the chemical

composition indicates a successful synthesis and modification process that enhances the material's application potential in adsorption and environmental remediation, consistent with findings in similar studies [39, 43-45].

3.4. BET Analysis

Table 2 presents the BET analysis results for the metakaolin, unmodified geopolymer, and modified geopolymer. The results of the BET analysis demonstrate the successful synthesis of geopolymers from metakaolin, as evidenced by the significant increases in specific surface area and pore volume when compared to the raw precursor. The specific surface area of the unmodified geopolymer increased from 0.1591 m²/g (metakaolin) to 0.7312 m²/g, with a concurrent increase in total pore volume from 0.00411 ×10⁻² cm³/g to 0.0824 ×10⁻² cm³/g. This transformation highlights the microstructural changes associated with the polymerization process, where aluminosilicate dissolution and subsequent three-dimensional network formation create interconnected pore structures [34, 48, 49]. However, the average pore diameter decreases (from 5.171 nm in metakaolin to 2.255 nm in the unmodified geopolymer), consistent with network densification during curing [49].

Upon modification with H₂O₂, the properties of the geopolymer are dramatically enhanced. The specific surface area increases to 9.7068 m²/g, the total pore volume increases to 3.0281 ×10⁻² cm³/g, and the average pore diameter expands to 6.239 nm. These results confirm the effectiveness of H₂O₂ as a porosity-enhancing agent. The decomposition of H₂O₂ during synthesis releases oxygen gas, introducing mesopores and increasing the structural accessibility of the material's surface area [34, 37, 50]. Such structural modifications align with findings in other studies, where H₂O₂-based pore engineering has been linked to significantly improved adsorption capabilities for heavy metals, organics, and FFAs [37, 38, 50]. Overall, these findings indicate that geopolymers synthesized with H₂O₂ modification are highly promising candidates for adsorption applications due to their high surface area, enhanced pore structure, and tailored mesoporosity.

3.5. Adsorption of FFAs in Crude Palm Oil

Free fatty acid adsorption from CPO using hydrogen peroxide (H₂O₂)-modified geopolymers is shown in **Table 4**. The data from this study demonstrate the significant improvement in FFA adsorption efficiency and adsorption capacity achieved by hydrogen peroxide (H₂O₂)-modified geopolymers when compared to both unmodified geopolymers and alternative adsorbents from the literature. Among the samples tested in this study, H₂O₂-modified geopolymer sample Gpm-3 achieved the highest FFA adsorption efficiency (85.3%) and adsorption capacity (222 mg/g). Other H₂O₂-modified samples, such as Gpm-2, also realized high efficiency (83%) and capacity (216 mg/g), showcasing the impact of H₂O₂ in enhancing geopolymers through porosity and increased surface interactions. In contrast, the unmodified geopolymer (Gp) exhibited poor adsorption efficiency (30.7%) and adsorption capacity (80 mg/g), underscoring the critical role of H₂O₂ in optimizing geopolymer structure. Adsorption efficiency is shown in **Table 5**.

When compared with other adsorbents in the literature, the H₂O₂-modified geopolymer (Gpm-3) outperformed mesoporous silica derived from palm oil boiler ash (MS-POBA), which achieved an efficiency of 35.54% [51], and NaOH-activated zeolites, which recorded efficiencies up to 50% [53]. Similarly, the H₂O₂-modified geopolymers surpassed bleaching earth-based adsorbents, such as attapulgite and kaolin, with efficiencies of 45% and ~10–15%, respectively [55]. Even K₂CO₃-modified zeolites exhibited limited performance in the range of

30–50% [54]. This demonstrates the advantages of the H₂O₂-modified geopolymer not only in adsorption efficiency but also in adsorption capacity, a parameter not commonly reported in other studies but highly relevant in quantifying FFA uptake.

However, one comparison highlights room for further development. Calcium silicate modified with glycerol achieved the highest reported efficiency (94.78%) in FFA adsorption. [52], slightly higher than Gpm-3. While adsorption capacity was not reported for calcium silicate, its observed performance underscores the importance of optimized mesoporous structures and enhanced functional groups. Further optimization of geopolymer synthesis, such as fine-tuning the H₂O₂ concentration, could potentially close or surpass this gap.

The higher performance of H₂O₂-modified geopolymers can be attributed to the decomposition of H₂O₂ during geopolymerization, which releases oxygen and creates extensive mesoporosity. This is critical for physical adsorption by providing enhanced surface area and pore volume suitable for trapping FFA molecules. Studies have shown that mesopores ranging from 2 to 50 nm align well with medium-chain FFAs, facilitating effective adsorption [56]. The role of surface functional groups, such as hydroxyl (Si-OH, Al-OH), in hydrogen bonding with FFAs' carboxylic groups further contributes to the chemical adsorption mechanism [57].

Additionally, the comparison with poor-performing unmodified geopolymers <10% [55] and kaolin ~10–15%; [55] Underscores the limitations of adsorbents with insufficient porosity or active functionalities. Gpm-3's adsorption capacity of 222 mg/g reflects nearly a threefold increase over unmodified geopolymer (80 mg/g), signifying the profound influence of porosity and active site availability created through H₂O₂ modification.

Table 4. Free fatty acid (FFA) adsorption from crude palm oil (CPO) using hydrogen peroxide (H₂O₂)-modified geopolymers.

Sample	Initial FFA content (%)	Contact time (hour)			After adsorption (%)	Adsorption efficiency (%)	Adsorption Capacity (mg/g)
		1	2	3			
Gp	1.3	1.20	0.90	0.90	0.90	30.70	80
Gpm-1	1.3	0.54	0.49	0.32	0.32	75.30	196
Gpm-2	1.3	0.32	0.27	0.22	0.22	83.00	216
Gpm-3	1.3	0.25	0.20	0.19	0.19	85.30	222
Gpm-4	1.3	0.32	0.25	0.25	0.25	80.70	210
Gpm-5	1.3	0.31	0.25	0.22	0.22	83.00	216
Gpm-6	1.3	0.28	0.25	0.20	0.20	84.60	220

Table 5. Comparison of free fatty acid (FFA) adsorption efficiency using different adsorbents.

Adsorbent	Modifier	FFA Adsorption Efficiency (%)	Reference
H ₂ O ₂ -modified geopolymer (Gpm-3)	H ₂ O ₂	85.3	This study
H ₂ O ₂ -modified geopolymer (Gpm-2)	H ₂ O ₂	83	This study
Unmodified geopolymer (Gp)	None	30.7	This study
Mesoporous silica (MS-POBA)	Methyl ester sulfonate (MES)	35.54	[51]
Modified calcium silicate (CaSiO ₃)	Glycerol	94.78	[52]
Zeolite (NaOH-activated)	NaOH	50	[53]
K ₂ CO ₃ -modified zeolite	K ₂ CO ₃	30–50	[54]
Bleaching earth (attapulgitite)	None	45	[55]
Kaolin	None	~10–15	[55]
Unmodified metakaolin geopolymer	None	<10	[34, 56]

3.6. Catalytic Transesterification of CPO

The performance of geopolymer materials as heterogeneous catalysts for biodiesel production has markedly progressed, as demonstrated in this study and prior research (**Table 7**). The current data (**Figure 3**) on H₂O₂-modified geopolymer catalysts (e.g., Gpm-3 and higher variants) reveals superior catalytic activity, achieving 100% fatty acid methyl ester (FAME) yield from crude palm oil (CPO) within just 1 hour. This rapid reaction time and maximum efficiency highlight the transformative impact of H₂O₂ modification, which enhances the number and diversity of active catalytic sites by introducing Brønsted acid sites and surface oxygen functional groups [57]. These properties not only improve reaction kinetics but also mitigate the inhibitory effects of free fatty acids (FFAs) or water, common in crude oils.

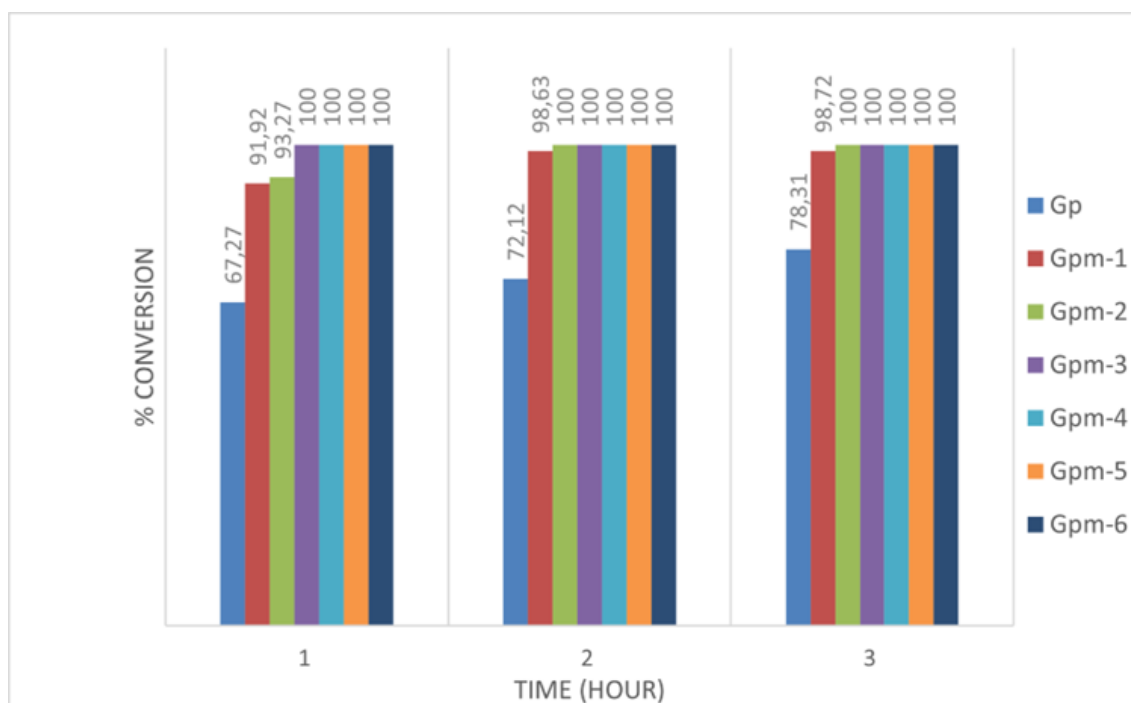


Figure 3. Transesterification yield of CPO using geopolymer catalyst.

Comparable studies further support the enhanced catalytic potential observed with specialized geopolymer materials. For example, calcium-modified geopolymer catalysts achieved near-100% FAME conversion within the same reaction time (1 hour), emphasizing the role of strong base sites integrated into the aluminosilicate framework [57]. However, unlike calcium-modified variants, H₂O₂-modified geopolymers exhibit acidic sites that support bifunctional activity, enabling simultaneous esterification of FFAs and transesterification of triglycerides, which is particularly critical when using CPO as a feedstock [57].

By comparison, unmodified or alkali-based geopolymer catalysts (e.g., K-based geopolymer [58]) have slower reaction times and often require milder feedstocks such as refined oils. This limitation arises from the limited density and specialization of active sites, which fail to address the chemical complexity of CPO effectively. For instance, potassium-based geopolymers reached only 89.9% FAME yield after 4 hours while maintaining low leaching rates. Waste-derived geopolymer catalysts, such as those based on rice husk ash, also demonstrate relatively high yields (~87%) under moderate conditions, yet they lack the rapid kinetics essential for industrial-scale biodiesel production [59].

To place these findings in a broader context, even non-geopolymer catalysts, such as choline hydroxide (an ionic liquid), achieve nearly comparable results for CPO

transesterification, with a 99.45% yield achieved in 1 hour [60]. However, the sustainability, robustness, and potential reusability of geopolymer-based systems provide distinct advantages over ionic liquids, especially when paired with appropriate post-synthesis modifications such as H₂O₂ treatment.

Free fatty acid adsorption from CPO using hydrogen peroxide (H₂O₂)-modified geopolymers is shown in **Table 4**. The data from this study demonstrate the significant improvement in FFA adsorption efficiency and adsorption capacity achieved by hydrogen peroxide (H₂O₂)-modified geopolymers when compared to both unmodified geopolymers and alternative adsorbents from the literature. Among the samples tested in this study, H₂O₂-modified geopolymer sample Gpm-3 achieved the highest FFA adsorption efficiency (85.3%) and adsorption capacity (222 mg/g). Other H₂O₂-modified samples, such as Gpm-2, also realized high efficiency (83%) and capacity (216 mg/g), showcasing the impact of H₂O₂ in enhancing geopolymers through porosity and increased surface interactions. In contrast, the unmodified geopolymer (Gp) exhibited poor adsorption efficiency (30.7%) and adsorption capacity (80 mg/g), underscoring the critical role of H₂O₂ in optimizing geopolymer structure. Adsorption efficiency is shown in **Table 5**.

Table 6. Catalytic performance of geopolymer catalysts in biodiesel production.

Catalyst	Feedstock	FAME Yield (%)	Reaction Time	Key Advantages	Ref.
Gpm-3 (H ₂ O ₂ -modified)	Crude Palm Oil	100%	1 hour	High efficiency, rapid reaction kinetics, robust water/FFA tolerance	This study
Ca-modified geopolymer	Soybean oil	~100%	1 hour	Strong base sites, minimal leaching, stable after reuse cycles	[57]
K-based geopolymer	Soybean oil	89.9%	4 hours	Enhanced basicity due to K ions, good leaching resistance	[58]
Rice husk ash-based geopolymer	Calophyllum inophyllum oil	87.68%	~3 hours	Waste-derived feedstock, high yield under moderate conditions	[59]
Choline hydroxide (non-geopolymer)	Crude Palm Oil	99.45%	1 hour	Short reaction time, high methyl ester yield	[60]

3.7. Catalytic Activity and FFA Removal Using Modified Geopolymers

The simultaneous removal of free fatty acids (FFAs) and the transesterification of crude palm oil (CPO) into biodiesel represent an efficient strategy for biodiesel production. In this study, modified geopolymer materials (Gpm-1 to Gpm-6) were evaluated alongside an unmodified geopolymer (Gp) for their ability to simultaneously reduce FFA content and catalyze the transesterification process. The findings reveal that modifications significantly enhance both FFA adsorption and catalytic activity for simultaneous biodiesel production when compared with the unmodified geopolymer.

FFA removal and yield data indicate that the modified geopolymers substantially outperform the unmodified geopolymer (Gp) (**Figure 4**). At 1 hour, FFA levels were reduced to 0.97% with Gpm-1, compared to 1.38% with Gp. This reduction further improved at 3 hours, with Gpm-1 achieving an FFA level of 0.72%, while Gp still retained 1.19% FFA. Simultaneously, biodiesel yield significantly increased with the modified geopolymers. At 1 hour, yield reached 89.78% for Gpm-1, 91.23% for Gpm-2, and peaked at 93.92% for Gpm-3, compared to only 60.72% with Gp. At 3 hours, all modified geopolymers exhibited yields

above 93%, with Gpm-3 achieving 96.47%, while Gp lagged at 72.52%. These results indicate that H₂O₂ modification successfully enhances the dual-functionality of the geopolymer by promoting active sites conducive to both FFA adsorption and catalytic conversion. The introduction of surface functional groups (e.g., hydroxyl and carboxyl groups) during H₂O₂ treatment likely increases the material's polarity, improving FFA adsorption via acid-base interaction, and simultaneously boosting catalytic sites essential for alcoholysis [23, 61].

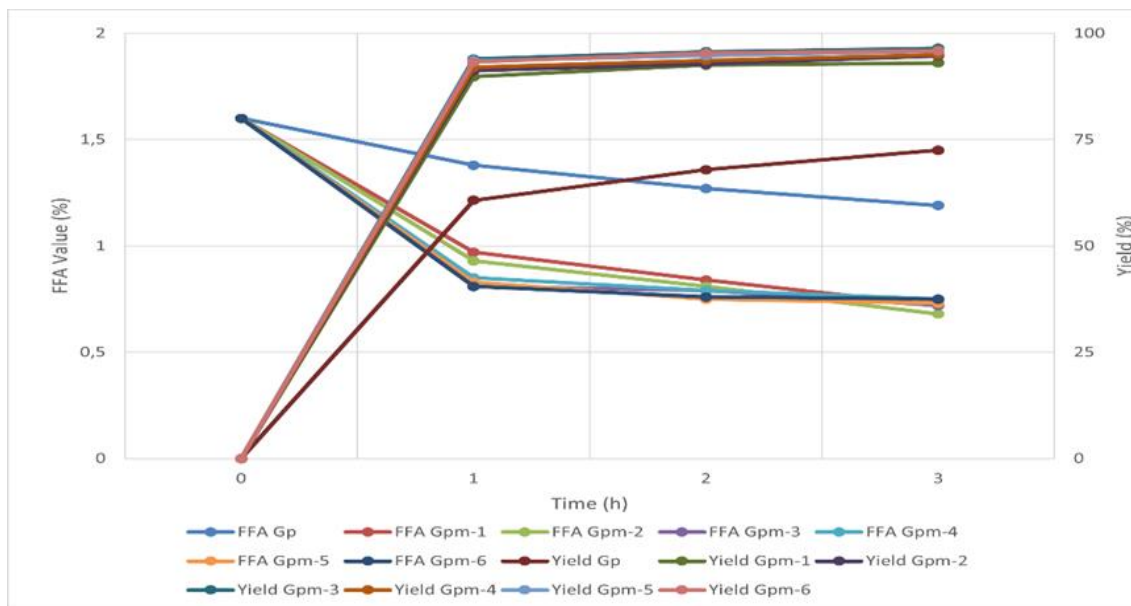


Figure 4. FFA value and yield percentage over time.

Compared to previous studies, the simultaneous integration of adsorption and catalysis remains underexplored. Conventional catalysts often require separate pre-treatment for FFA removal, such as Amberlyst-15 for esterification (1) or zeolite-based adsorbents (12), before transitioning to catalytic transesterification using solid acid or base catalysts (e.g., CaO/Al₂O₃ [62] or sulfated tin oxides [63]). These segmented processes yield relatively lower biodiesel outputs. For example, combinations of acid/base catalysts [62, 63] without integrated adsorption functionality reached yields of 89% to 94% over longer reaction times (~3–5 hours), while requiring specific pre-treatment of high-FFA oils. In contrast, the modified geopolymers in this study perform the dual adsorption and catalytic functions in a single-step process, achieving comparable or superior yields (~96%) in a simplified system. This integration eliminates the need for additional FFA reduction steps (e.g., using homogeneous chromosulfuric acid catalysts [64]) and underscores the efficiency of the geopolymer-based approach.

The performance comparison of the modified geopolymers and conventional methods is summarized in **Table 8**. The results reinforce the conclusion that H₂O₂-modified geopolymers offer a compelling solution to the challenges of biodiesel production from high-FFA feedstocks such as CPO, offering high thermal stability, improved chemical reusability, and cost-effectiveness by reducing procedural complexity and enhancing yield efficiency [25, 61].

Table 8. Comparison of FFA removal and yield from modified geopolymers and previous studies.

Process Type	Material Used	Step(s) Conducted	FFA Removal Efficiency (%)	FAME Yield (%)	Ref.
Separated Process (Gpm-3)	H ₂ O ₂ -Modified Geopolymer Gpm-3	FFA removal + FAME	85.38	100	This Study
Combined Process (Gpm-3)	H ₂ O ₂ -Modified Geopolymer Gpm-3	FFA removal + FAME	55.00	96.47	This Study
Bifunctional Catalyst	CaO/Al ₂ O ₃	FAME only (No adsorption)	N/A	~89	[62]
Sulfonic Acid Mesostructured	Mesostructured silica w/sulfonic acid	FAME only (No adsorption)	N/A	~95	[61]
Amberlyst-15	Amberlyst-15 resin	FFA removal only	~98	N/A (no transesterification)	[12]
KOH-Impregnated Zeolite	Zeolite w/KOH	FFA removal only	~94	N/A (no transesterification)	[12]
Sulfated Metal Oxide	SO ₄ ²⁻ /ZrO ₂	FFA pre-treatment then FAME	~75	~94	[65]

The performance of H₂O₂-modified geopolymers (Gpm-1 to Gpm-6) for the simultaneous removal of free fatty acids (FFA) and methyl ester (FAME) production via crude palm oil (CPO) transesterification was evaluated and compared between separated (two-step) and combined (one-step) processes (**Figure 5**). The data highlight the significant potential of modified geopolymers as dual-functioning materials for catalytic and adsorptive applications. Notably, the modified geopolymers delivered better FFA removal and biodiesel yields compared to the unmodified geopolymer (Gp), though their performance was influenced by process type.

In the separated process, where FFA removal and catalytic transesterification occur in two distinct steps, the modified geopolymers demonstrated higher FFA removal efficiency, ranging from 75.38% (Gpm-1) to 85.38% (Gpm-3), compared to only 30.77% for the unmodified geopolymer (Gp). The yield of biodiesel was also exceptionally high in the separated system, with most modified geopolymers (Gpm-2 to Gpm-6) achieving 100% yield. Conversely, in the combined process, where FFA adsorption and transesterification were integrated, FFA removal efficiency decreased slightly for the modified geopolymers, ranging from 53.13% (Gpm-4 and Gpm-6) to 57.50% (Gpm-2). Correspondingly, FAME yields were also slightly reduced in the combined method but remained high, ranging from 93.02% (Gpm-1) to 96.47% (Gpm-3). Though the combined process shows a small trade-off in individual performance metrics, it eliminates the need for multiple steps, simplifying biodiesel production and improving process efficiency.

Compared to previous studies, the use of modified geopolymers in the combined process demonstrates competitive performance. For example, sulfonated metal oxides like SO₄²⁻/ZrO₂ achieved biodiesel yields of ~94% but required separate FFA pre-treatment steps (65,67), while KOH-impregnated zeolites and Amberlyst-15 primarily addressed FFA reduction and lacked catalytic transesterification functionality [1, 12]. The ability of H₂O₂-modified geopolymers to integrate both adsorption and catalytic activities into a single step represents a clear advantage over traditional methods, particularly for high-FFA feedstocks like CPO.

While the separated process achieves higher absolute efficiencies due to dedicated optimization of each step, the combined process simplifies production, reduces energy and solvent use, and still delivers high biodiesel yields (~96%).

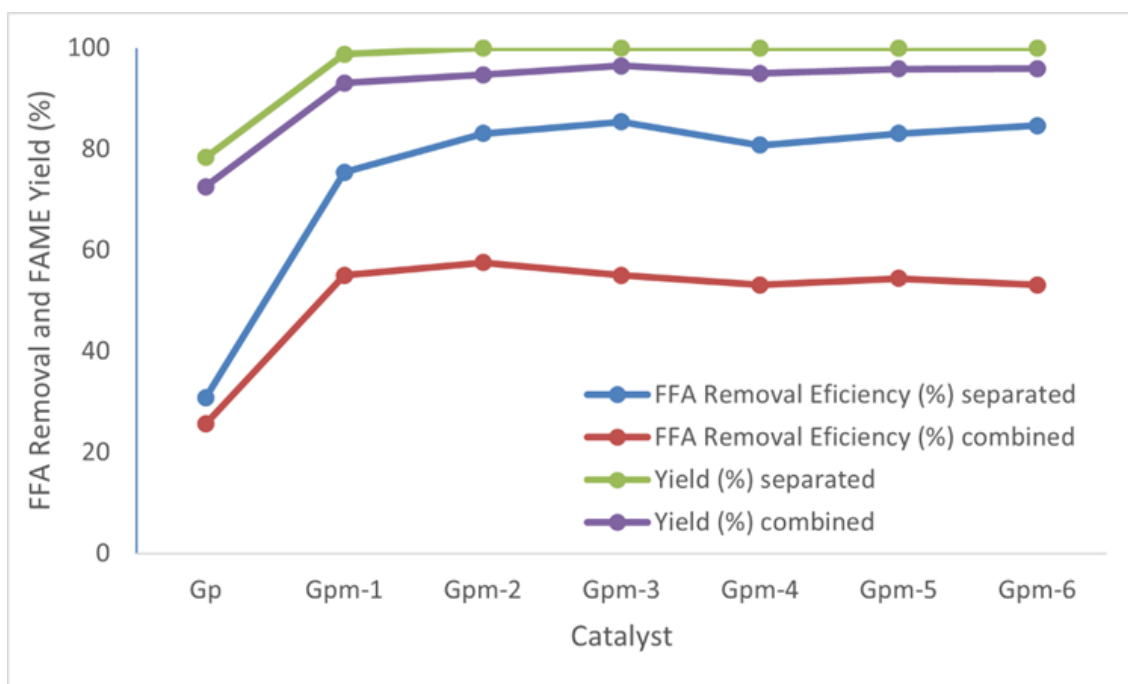


Figure 5. FFA removal efficiency and yield comparison.

4. CONCLUSION

This study has successfully demonstrated the remarkable potential of H₂O₂-modified geopolymers as integrated adsorbent-catalysts for biodiesel production from crude palm oil. The optimal H₂O₂-modified geopolymer (Gpm-3) demonstrated exceptional performance, achieving 85.38% FFA removal efficiency and 100% biodiesel yield in the separated process, while maintaining impressive performance (55% FFA removal and 96.47% biodiesel yield) in the combined process. The successful integration of adsorption and catalytic functions in a single material and process represents a significant advancement in biodiesel production technology, offering a simplified, more sustainable approach compared to conventional multi-step methods. These findings establish H₂O₂-modified geopolymers as promising candidates for industrial-scale biodiesel production, combining cost-effectiveness, environmental sustainability, and high performance in a single material system.

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6. AUTHORS' NOTE

The authors declare that there is no conflict of interest regarding the publication of this article. The authors confirmed that the paper was free of plagiarism.

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