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Comparative Study of Impregnated and Non-Impregnated CaO Catalysts for Biodiesel Synthesis

Adinda Tiara Khansa[1], Sabrina Nayla Junaidi[1], Wiliandi Saputro[2], Muhammad Al Muttaqii[3], Ardika Nurmawati[1,4], Erwan Adi Saputro[1,4,5*]

[1]Chemical Engineering Department, Universitas Pembangunan Nasional Veteran Jawa Timur, Surabaya
[2]Mechanical Engineering, Universitas Pembangunan Nasional Veteran Jawa Timur, Surabaya
[3]Research Center for Chemistry, National Research and Innovation Agency of Indonesia, Tanggerang Selatan
[4]Low Carbon Technology Research Centre, Universitas Pembangunan Nasional Veteran Jawa Timur, Surabaya
[5]Environment Science, Universitas Pembangunan Nasional Veteran Jawa Timur, Surabaya
***Corresponding author:** erwanadi.tk@upnjatim.ac.id

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Abstract
Calcium oxide (CaO) is a widely used heterogeneous catalyst in biodiesel synthesis due to its strong basicity and low cost. However, its performance is often limited by low surface area. This study investigates the enhancement of CaO through ammonium carbonate impregnation and evaluates its effect on surface area and catalytic activity. The catalyst was prepared via calcination, impregnation with ammonium carbonate, and recalcination at 900 °C. BET surface analysis showed a substantial increase in specific surface area from 8.242 m²/g to 96.390 m²/g. The modified catalyst was then applied in the transesterification of crude palm oil with methanol at a 1:12 molar ratio. The highest biodiesel yield achieved using the impregnated catalyst was 89% at 2.5 wt% catalyst loading, 3.5 hours reaction time, and 60 °C. outperforming unmodified CaO, which yielded 79.09% under similar conditions. These results demonstrate that ammonium carbonate impregnation not only improves the structural properties of CaO but also significantly enhances its catalytic efficiency in biodiesel production.
Keywords: *biodiesel, calcium oxide, catalyst impregnation, surface area, transesterification*

Abstrak
Kalsium oksida (CaO) merupakan katalis heterogen yang banyak digunakan dalam sintesis biodiesel karena sifat kebasaannya yang kuat dan biaya produksinya yang rendah. Namun, kinerjanya sering kali terbatas oleh luas permukaan yang rendah. Penelitian ini mengevaluasi peningkatan kinerja CaO melalui impregnasi dengan amonium karbonat serta pengaruhnya terhadap luas permukaan dan aktivitas katalitik. Katalis disintesis melalui proses kalsinasi, impregnasi dengan amonium karbonat, dan rekalsinasi pada suhu 900 °C. Analisis BET menunjukkan peningkatan luas permukaan spesifik secara signifikan, dari 8,242 m²/g menjadi 96,390 m²/g. Katalis yang telah dimodifikasi ini kemudian digunakan dalam reaksi transesterifikasi minyak kelapa sawit kasar dengan metanol pada rasio molar 1:12. Hasil biodiesel tertinggi yang diperoleh dengan katalis terimpregnasi adalah 89% pada kondisi optimum: 2,5 wt% katalis, waktu reaksi 3,5 jam, dan suhu 60 °C. Hasil ini melampaui kinerja CaO tanpa modifikasi, yang hanya menghasilkan 79,09% pada kondisi serupa. Temuan ini menunjukkan bahwa impregnasi amonium karbonat tidak hanya memperbaiki struktur fisik CaO, tetapi juga secara signifikan meningkatkan efisiensinya sebagai katalis dalam produksi biodiesel.
Kata Kunci: *biodiesel, kalsium oksida, impregnasi katalis, luas permukaan, transesterifikasi*

1. Introduction

Biodiesel, a renewable and biodegradable fuel, has emerged as a promising alternative to conventional diesel due to its non-toxic nature and its potential to reduce emissions from diesel engines. It can be used directly in existing diesel engines without significant modifications, offering a practical and efficient substitute for petroleum-based fuels. Biodiesel is typically produced via transesterification of vegetable oils in the presence of acid, base, or enzymatic catalysts, a process that contributes significantly to lowering exhaust emissions. Despite these advantages, challenges such as high production costs and limited feedstock availability continue to limit widespread adoption. Nonetheless, the broader utilization of biodiesel is expected to support global sustainability goals and reduce reliance on fossil fuels [1].

Homogeneous catalysts such as sulfuric acid (H_2SO_4), sodium hydroxide (NaOH), and potassium hydroxide (KOH) are commonly used in biodiesel production. However, their application poses notable drawbacks, including the need for complex post-reaction separation and susceptibility to soap formation due to reactions with free fatty acids (FFAs). These limitations reduce biodiesel yield and increase catalyst consumption. Consequently, recent research has focused on the development of heterogeneous catalysts to improve the efficiency, selectivity, and economic viability of biodiesel production [2].

Although calcium oxide (CaO) is highly valued for its strong basicity and cost-effectiveness, its practical application can be compromised by leaching and structural degradation over repeated reaction cycles. A recent study by Istadi (2016) demonstrated that CaO-based catalysts (e.g., K_2O/CaO–ZnO) retained acceptable activity for biodiesel production through multiple reuse cycles after simple regeneration via washing, drying, and recalcination, thereby partially overcoming these drawbacks [3]. Nevertheless, CaO also exhibits limited tolerance to feedstocks with high free fatty acid (FFA) content, which restricts its applicability in processing low-grade oils [4].

One approach to improve the structural properties of CaO is through impregnation, a method that introduces active species into a porous support by adsorption from a precursor solution. The effectiveness of this process depends on variables such as precursor concentration, impregnation time, and drying conditions [5]. Impregnation enhances surface area and active site dispersion, thereby improving catalytic performance [6]. Ammonium carbonate is commonly employed as an impregnating agent to form calcium carbonate precursors, which are then calcined to regenerate CaO with improved basicity [7].

To evaluate these structural improvements, the Brunauer–Emmett–Teller (BET) method is widely used for surface characterization. BET analysis provides measurements of specific surface area (m^2/g), pore diameter, and pore volume. Compared to other techniques such as mercury intrusion porosimetry, BET offers greater accuracy in assessing microporosity in solid catalysts used for transesterification [8]. Surface area is a critical parameter in heterogeneous catalysis since it directly relates to the number of accessible active sites. In fact, Sun (2018) recommends the normalization of catalytic activity to surface area for a fair comparison of catalyst performance, underscoring surface area as a key indicator of catalytic potential [9].

Although CaO exhibits desirable catalytic properties, its inherently low surface area limits its efficiency in biodiesel synthesis. Impregnation with ammonium carbonate has been proposed as a promising strategy to address this limitation by enhancing surface accessibility. This study aims to compare the BET surface areas of pure and impregnated CaO catalysts to assess the extent to which surface modification improves catalyst characteristics and enhances catalytic performance in biodiesel production.

2. Material and Methods

This study consisted of two main experimental stages: catalyst preparation and characterization, followed by the application of the catalyst in biodiesel synthesis via transesterification. All procedures were performed using standard laboratory equipment and analytical protocols to ensure consistency and reproducibility.

2.1 Materials

The chemicals used for catalyst preparation included calcium oxide (CaO), ammonium carbonate ($(NH_4)_2CO_3$), and deionized water. Equipment used in the synthesis process included beaker glasses, a magnetic stirrer, an oven, and a high-temperature furnace. Crude palm oil (CPO) and methanol were used as feedstocks for the transesterification reaction.

2.2 Catalyst Preparation

The catalyst was synthesized via a wet impregnation method. Precisely 3.5 g of CaO (80 mesh) was mixed with 50 g of ammonium carbonate solution and stirred for 30 minutes at 40 rpm. The mixture was then dried in an oven at 110 °C for 5 hours, followed by calcination in a furnace at 900 °C for 90 minutes. The final catalyst was stored in a desiccator to prevent moisture absorption.

2.3 Catalyst Characterization

The characterization of the synthesized superbasic CaO catalyst involved surface analysis using the Brunauer–Emmett–Teller (BET) method, which provides data on specific surface area, pore diameter, and pore volume. The BET analysis was performed at the laboratory of the National Research and Innovation Agency (BRIN) in Tangerang.

2.4 Transesterification Procedure

To evaluate catalytic performance, the synthesized catalyst was applied in the transesterification of crude palm oil with methanol at a 1:12 molar ratio. The reaction was carried out at 60 °C under atmospheric pressure, with variations in catalyst loading (1.5–3.5 wt%) and reaction time (2–4 hours). After the reaction, the mixture was allowed to settle, and the resulting biodiesel layer was separated and analyzed to determine the yield.

3. Results and Discussion

The Brunauer–Emmett–Teller (BET) method was employed to determine the specific surface area of the catalyst, as it is a widely accepted technique for characterizing porous materials. BET analysis quantifies the volume of gas adsorbed onto the catalyst surface under varying pressures, enabling the calculation of surface area based on adsorption isotherms. This model assumes multilayer adsorption, providing a more accurate representation of porous surface behavior. Owing to its precision and reliability, the BET method is extensively used in catalyst research, as an increased surface area is generally correlated with more active sites and improved catalytic activity. The BET characterization results presented below demonstrate the impact of ammonium carbonate impregnation on the surface properties of CaO.

Table 1. BET Surface Area of Pure CaO and CaO Impregnated with Ammonium Carbonate

Catalyst	Surface Area (m2/g)
Pure CaO	8.242
CaO Impregnated	96.390

Impregnation of the CaO catalyst with ammonium carbonate led to a substantial increase in its specific surface area. BET analysis showed that the surface area increased from 8.242 m^2/g (pure CaO) to 96.390 m^2/g after impregnation. This notable enhancement demonstrates the effectiveness of the impregnation process in modifying the pore structure and improving the surface characteristics of the catalyst. **Table 2** presents results from several published studies on catalyst impregnation and the corresponding changes in surface area to contextualize these findings.

Table 2. BET Surface Area Results Before and After Catalyst Impregnation

Catalyst Material	Impregnation Method	Impregnation Material	Surface Area (m2/g)		Source
			Before Impregnation	After Impregnation	
Cu/AC (activated carbon)	Wet impregnation followed by H_2 reduction	$Cu(NO_3)_2 \cdot 3H_2O$	272.77	307.06	[10]
Cu/ZnO dan Cu/ZnO/γ-Al2O3	Impregnation and co-precipitation	$Cu(NO_3)_2$, $Zn(NO_3)_2$, $Al(NO_3)_3$	77.02	151.4	[11]
Fe-Co/HZSM-5 dan meso-HZSM-5	Incipient wetness impregnation with desilication	$Fe(NO_3)_3 \cdot 9H_2O$, $Co(NO_3)_2 \cdot 6H_2O$	266.28	526.03	[12]
Rice husk ash + CaO	Calcium nitrate impregnation into rice husk ash	$Ca(NO_3)_2$	7.23	16.64	[13]
Activated Carbon (pre-pyrolysis + NaOH)	Pre-pyrolysis followed by NaOH impregnation	NaOH	292	2093	[14]

These five studies clearly demonstrate that catalyst impregnation can lead to substantial increases in surface area, with BET values improving from as little as 74 m^2/g to over 1800 m^2/g. The results obtained in the present study are consistent with these findings, reinforcing the conclusion that surface area enhancement via impregnation is both scientifically valid and experimentally reproducible.

Moreover, both the present study and the work of Liou (2016) reported more than a tenfold increase in surface area following impregnation, highlighting the consistency of this phenomenon across various catalyst systems [14]. These results confirm that surface area enhancement via impregnation is not only reproducible but also directly contributes to improved catalytic performance. Supporting this, Rahman et al. (2019) demonstrated that increased surface area is strongly correlated with the number of active sites, thereby enhancing catalytic efficiency in transesterification reactions [15].

In the case of CaO, impregnation with ammonium carbonate has been shown to significantly improve its surface properties. This enhancement is attributed to structural modifications induced during the impregnation process, including increased porosity and the generation of additional active sites. Such changes are essential for enhancing catalyst efficiency in biodiesel synthesis. Syahrir (2017) further reported that CaO impregnated with ammonium carbonate at a concentration of 0.57 g/mL achieved a crystallinity of 96.64%, demonstrating the method's effectiveness in increasing both surface area and structural integrity of the catalyst [16].

An increased surface area in CaO catalysts directly correlates with enhanced catalytic activity, particularly in transesterification reactions for biodiesel production. The higher number of active sites facilitates better interaction with reactant molecules, leading to improved conversion rates and product yields. Previous studies have demonstrated that such modifications not only improve the physical characteristics of the catalyst but also its chemical reactivity, making impregnated CaO a more effective material for biodiesel synthesis.

To verify this hypothesis, the ammonium carbonate–impregnated CaO catalyst was further tested in the transesterification of palm oil with methanol. This experiment aimed to determine whether the observed surface area enhancement translated into improved catalytic performance. The reaction was conducted under various conditions by adjusting catalyst loading and reaction time, while the temperature and methanol-to-oil molar ratio were kept constant. A maximum biodiesel yield of 89% was obtained at 2.5 wt% catalyst loading, 3.5 hours of reaction time, and a temperature of 60°C. This high yield under relatively mild conditions demonstrates the significant improvement in catalytic efficiency brought about by the impregnation treatment.

For comparison, Murti et al. (2015) reported a maximum biodiesel yield of 79.09% using unmodified CaO at 3 wt% catalyst loading, 4 hours of reaction time, and the same temperature of 60 °C [17]. The superior yield obtained in the present study confirms that surface modification through ammonium carbonate impregnation increases the catalyst's surface area and enhances its overall reactivity, making it a more effective catalyst for biodiesel production from palm oil.

4. Conclusion

This study confirms that impregnating calcium oxide (CaO) with ammonium carbonate significantly enhances its physicochemical properties, particularly by increasing the specific surface area from 8.242 m^2/g to 96.390 m^2/g. The improved surface accessibility facilitates the formation of additional active sites, which directly contributes to superior catalytic performance. When applied in the transesterification of palm oil, the impregnated CaO catalyst achieved a maximum biodiesel yield of 89% under optimal conditions: 2.5 wt% catalyst loading, 3.5 hours reaction time, and a reaction temperature of 60 °C. In contrast, unmodified CaO under similar conditions produced a lower yield of 79.09%. These results demonstrate that ammonium carbonate impregnation is an effective surface modification technique that not only improves the structural properties of CaO but also significantly enhances its catalytic efficiency for biodiesel production.

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