

Article

From Waste to Catalyst: Transforming Mussel Shells into a Green Solution for Biodiesel Production from *Jatropha curcas* Oil

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Abstract: This study introduces an innovative approach to sustainable biodiesel production using mussel shell-derived calcium oxide (CaO) as a catalyst for converting *Jatropha curcas* oil into biodiesel. By repurposing waste mussel shells, the research aims to provide an eco-friendly and cost-effective solution for environmentally responsible biodiesel production, aligning with global standards. The study involves characterizing the catalyst, optimizing reaction conditions, and achieving a remarkable 99.36% Fatty Acid Methyl Ester (FAME) yield, marking a significant step toward cleaner and more economically viable energy sources. Biodiesel, recognized for its lower emissions, is produced through transesterification using mussel shell-derived CaO as a sustainable catalyst. This research contributes to cleaner and economically viable energy sources, emphasizing the importance of sustainable energy solutions and responsible catalytic processes. This research bridges the gap between waste management, catalyst development, and sustainable energy production, contributing to the ongoing global shift towards cleaner and more economically viable energy sources.

Keywords: biodiesel production; mussel shell-derived CaO catalyst; sustainable catalyst; transesterification; *Jatropha curcas* oil; renewable energy



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1. Introduction

The global energy landscape is currently characterized by increased greenhouse gas emissions, including CO₂, and how combusting fossil fuels affect the environment. In response, there is a pressing need to transition towards sustainable alternative fuels, including biodiesel, bioethanol, and biomass, which align with the sustainability goals outlined in Saudi Arabia's Vision 2030 strategy [1,2]. This transition is essential to mitigate the environmental challenges posed by the current energy paradigm [3].

Biodiesel, as a sustainable energy source, offers a compelling alternative to traditional petroleum diesel. Its green credentials include renewability, biodegradability, non-toxicity, reduced CO and CO₂ emissions, sulfur-free composition, and straightforward production [4]. Moreover, biodiesel is environmentally friendly because it minimizes hazardous exhaust emissions [5,6].

Mussel shell formation is a vital natural process in marine ecosystems, where mussels actively produce calcium carbonate for their protective shells. These shells contribute significantly to marine habitats, providing shelter and influencing local biodiversity. Beyond its ecological importance, researchers explore mussel shell production for potential applications in materials science and environmental remediation [7]. The annual production of mussel shells thus stands as a crucial element in the intricate balance of marine ecosystems, influencing biodiversity and contributing to the overall health of coastal environments [7].

Catalysts are essential in biodiesel production, with three primary types: homogeneous, heterogeneous (basic or acidic), and biocatalysts [8]. Heterogeneous catalysts are gaining popularity due to their environmental and economic advantages [9]. Biodiesel production is shifting towards non-edible oils like *J. curcas*, which offer high oil content and can grow in adverse conditions without competing with food sources [10]. Hence, *J. curcas* oil is a promising candidate for biodiesel transesterification [11,12].

Catalysts are crucial in facilitating the dominant technique to produce biodiesel, the transesterification process [13]. Homogeneous catalysts, heterogeneous catalysts, and biocatalysts constitute the three primary categories, each further classified into acid and base catalysts [14]. Homogeneous catalysts, such as KOH and NaOH, are commonly used as base catalysts. However, their retrieval and the substantial volume of wastewater from catalyst separation present significant drawbacks [15]. In contrast, heterogeneous catalysts offer several advantages, notably in mitigating environmental and economic drawbacks related to homogeneous catalysts [16]. Heterogeneous catalysts can be separated and reused more efficiently, minimizing the environmental footprint. This streamlined separation process reduces wastewater generation and simplifies biodiesel synthesis, eliminating the need for certain process steps [17].

The transesterification process, a key step in biodiesel synthesis, is influenced by various factors, such as the catalyst type, reaction conditions (like temperature, time, and reactant ratios), and the ability to reuse the catalyst [18,19]. Understanding how these factors impact biodiesel production is crucial for optimizing the process and ensuring high-quality biofuels. Additionally, exploring the use of natural waste products like chicken eggshells, crab shells, mussel shells, and clamshells, all rich in calcium carbonate (CaCO_3), presents an interesting opportunity. These discarded shells could create CaO-based catalysts, especially suitable for biodiesel synthesis at high temperatures [20–22].

Transesterification using mussel shells as a heterogeneous catalyst offers several advantages [23]. Firstly, they show potential for high catalyst yields. Secondly, waste materials promote environmental sustainability. Thirdly, they are cost-effective, lowering biofuel production costs and supporting the shift to sustainable energy sources. Lastly, their ready availability in substantial quantities locally adds to their appeal as a catalyst source [24,25].

This study introduces a fresh perspective to the field of biodiesel production. Its primary objective is to investigate the use of discarded mussel shells as a renewable source for a solid base catalyst, which represents a departure from traditional catalyst materials. This novel application addresses environmental concerns and taps into the abundance of cost-effective waste materials. The choice of mussel shell-derived CaO as a catalyst is motivated by its potential advantages over commercially available CaO. Additionally, the research aims to identify the optimal operating conditions that balance the quantity and quality of biodiesel, addressing efficiency challenges. This study underscores the research's dedication to addressing modern energy and environmental issues through inventive, environmentally conscious methods.

2. Results

2.1. Transesterification Reaction

The study examined the process parameters for trans-esterifying *J. curcas* oil into biodiesel using the mussel shell waste catalyst. It investigated the influence of reaction temperature, time, methanol-to-oil ratio, calcination temperature, and catalyst amount.

2.1.1. Impact of Reaction Time

The reaction time is a crucial factor in optimizing biodiesel yield. Batch experiments were conducted with consistent conditions: 50 g of oil, 110°C, methanol-to-oil ratio of 1:12, 6 wt.% catalyst calcined at 900°C, and varying reaction times of 3, 4, 5, and 6 h. Figure 1a indicates that the conversion percentage rose from 40.90% to 46.54% as the

transesterification time extended from 3 to 6 h. However, at 5 h, there was a slight drop to 44.79%, but the highest conversion of 62.51% was achieved after 6 h.

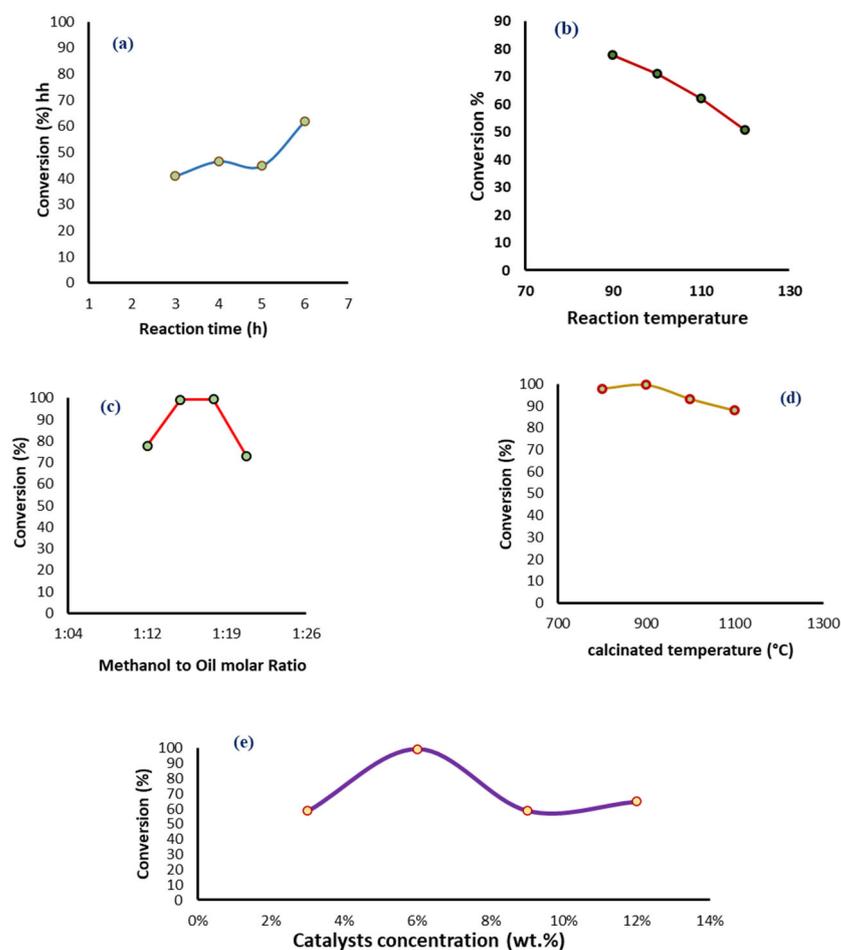


Figure 1. (a) Effect of reaction time on biodiesel yield. (b) Effect of reaction temperature on biodiesel yield. (c) Effect of molar ratio on biodiesel yield. (d) Effect of catalyst calcination temperature on biodiesel yield. (e) Effect of catalyst concentration.

2.1.2. Effects of Different Reaction Temperatures

Temperature significantly affects the biodiesel reaction and its yield. This study conducted transesterification for 6 h with a 12:1 molar ratio, using 6 wt.% of mussel waste shell catalyst calcined at 90 °C. Different temperature ranges (90, 100, 110, and 120 °C) were investigated. As shown in Figure 1b, the reaction rate decreased with increasing temperature, resulting in reduced yield, especially at 120 °C. The maximum methyl ester yield, approximately 77.78%, was achieved at 90 °C, while it dropped to 71.03% at 100 °C, 62.01% at 110 °C, and 50.53% at 120 °C.

2.1.3. Impact of Various Methanol to Oil Ratios

The molar ratio between methanol and oil is a crucial factor in manufacturing biodiesel. While the stoichiometric molar ratio for transesterification is 3:1, higher ratios are often used to expedite the reaction and increase product yield. In this study, *J. curcas* oil was subjected to transesterification with varying methanol to oil molar ratios (12:1, 14:1, 18:1, and 21:1) using 6 wt.% of calcined mussel shell catalyst for 6 h at 90 °C. Figure 1c shows how the molar ratio affects the amount of biodiesel produced. Initially, increasing the molar ratio led to a higher yield. The highest yield of 99.36% was achieved at a 1 molar ratio of 18:1, but it started to decline when the ratio was further increased to 21:1.

2.1.4. Effect of the Calcined Catalyst Temperatures

Mussel shell catalysts were subjected to different calcination temperatures (800, 900, 1000, and 1100 °C) and then used in transesterification processes with a methanol-to-oil molar ratio of 18:1 for 6 h at 90 °C. The influence of calcination temperature on the oil yield percentage is depicted in Figure 1d. It is evident that the percentage yield increased as the calcination temperature rose. The highest yield was 99.36% at 900°C, which is consistent with SEM results. However, when the calcination temperature exceeded 900 °C, the yield percentages dropped to 87.73% at 1100 °C.

2.1.5. Impact of Catalyst Concentration

To assess the impact of catalyst quantity on the maximum conversion of *J. curcas*, methyl ester catalysts calcined at 900 °C were used in amounts of 3, 6, 9, and 12 wt.% at a temperature of 90 °C for 6 h, and with a molar ratio of 18:1. As depicted in Figure 1e, the quantity of catalyst added significantly influences the conversion of *J. curcas* oil to biodiesel.

Increasing the catalyst amount from 3 to 6 wt.% resulted in a significant increase in the methyl ester content of *J. curcas* oil, rising from approximately 58.66% to 99.36%. However, as the catalyst quantity increased to 9%, the conversion efficiency dropped to 65.89%. The yield increased to 63.88% when the catalyst quantity was raised to 12%.

2.2. Catalyst Characterization

2.2.1. Thermogravimetric Analysis

To find the optimal calcination temperature for CaO production, we conducted a DSC/TGA analysis on the mussel shell sample, and the results are depicted in Figure 2. Initially, between 50 and 400 °C, a decrease in mass occurred due to the removal of surface-adsorbed water. The most significant mass loss, observed from 400 to 600 °C, was attributed to eliminating mineral or volatile components in the mussel shell. Notably, between 600 and 800 °C, there was a maximum weight reduction, indicating complete decomposition at 800 °C with the simultaneous release of CO₂, as shown in reaction 1. Beyond 800 °C, the sample's weight remained constant at 53.31 wt.%.

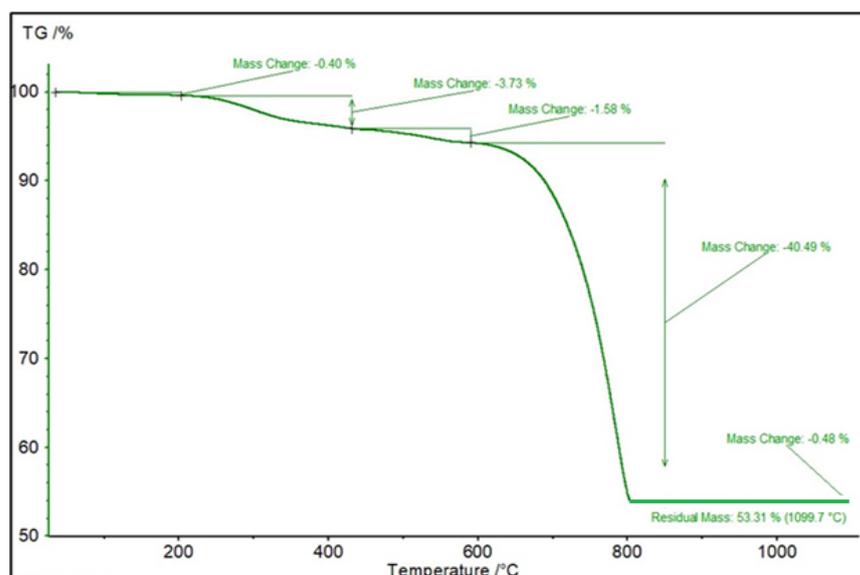


Figure 2. DSC/TGA analysis of mussel shells.

2.2.2. Thermogravimetric Analysis

The Fourier transform infrared spectra of non-calcined and calcined mussel shell catalysts at temperatures of 800, 900, 1000, and 1100 °C are depicted in Figure 3. Initially, absorption bands in the non-calcined mussel shell are observed at 705 cm⁻¹, 858 cm⁻¹,

1099 cm^{-1} , and 1457 cm^{-1} . During calcination, as CaCO_3 in the mussel shell decomposes into CaO , releasing CO_2 , the strength of these absorption bands at 3693 cm^{-1} , 1436 cm^{-1} , and 865 cm^{-1} decreases. Additionally, the production of basic OH groups attached to the calcium atoms produces a distinct sharp band at 3639 cm^{-1} .

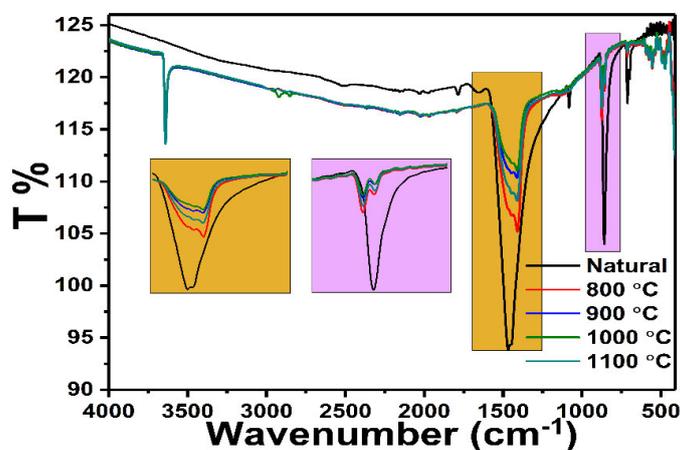


Figure 3. FTIR spectra of mussel shell catalyst non-calcined and calcined at 800, 900, 1000, and 1100 °C.

2.2.3. Thermogravimetric Analysis

Figure 4 displays the chemical constitution of waste mussel shells upon being calcined for 4 h at temperatures of 800, 900, 1000, and 1100 °C. The primary mineral component, as revealed by XRF analysis, is CaO . The waste mussel shell catalysts contain high CaO concentrations, ranging from 98.67 to 98.85 wt.%. This substantial presence of calcium suggests that the initial material was composed of CaCO_3 , which becomes CaO after calcination.

The methyl ester composition in biodiesel was analyzed at different calcined catalyst temperatures using gas chromatography–mass spectrometry (GC–MS). The components of methyl ester examined included Palmitoleic ME ($\text{C}_{17}\text{H}_{32}\text{O}_2$, C16:1), Palmitic Acid ME ($\text{C}_{17}\text{H}_{34}\text{O}_2$, C16:0), Linoleic Acid ME ($\text{C}_{19}\text{H}_{34}\text{O}_2$, C18:2), Oleic Acid ME ($\text{C}_{19}\text{H}_{36}\text{O}_2$, C18:1), Stearic Acid ME ($\text{C}_{19}\text{H}_{38}\text{O}_2$, C18:0), and Crotonic Acid ME ($\text{C}_5\text{H}_8\text{O}_2$, C4:0). All the above components were found at 900 °C, as shown in Figure S1.

2.2.4. Sample Structures

Figure 5 presents the XRD patterns of natural and calcined mussel shells. The natural mussel shells show a predominant composition of CaCO_3 , as indicated by the XRD results, with no discernible CaO peak. However, with increased calcination temperature, CaCO_3 completely transforms into CaO , accompanied by the release of (CO_2). The main component of calcined catalysts at or above 800 °C is the active ingredient (CaO). The XRD pattern exhibits sharp, high-intensity peaks, signifying the well-defined crystalline structure of the CaO catalyst. The catalyst peak is observed at 2θ values of 32.28°, 37.45°, 54°, 64.33°, 67.56°, and 79.89°, indicating reflections from planes with (1 1 1), (2 0 0), (2 2 0), (3 1 1), (2 2 2), and (4 0 0) orientations, respectively (JCPDS card 01-075-0264). These peaks align with the CaO standard pattern and the XRF investigation, which revealed a substantial concentration of CaO .

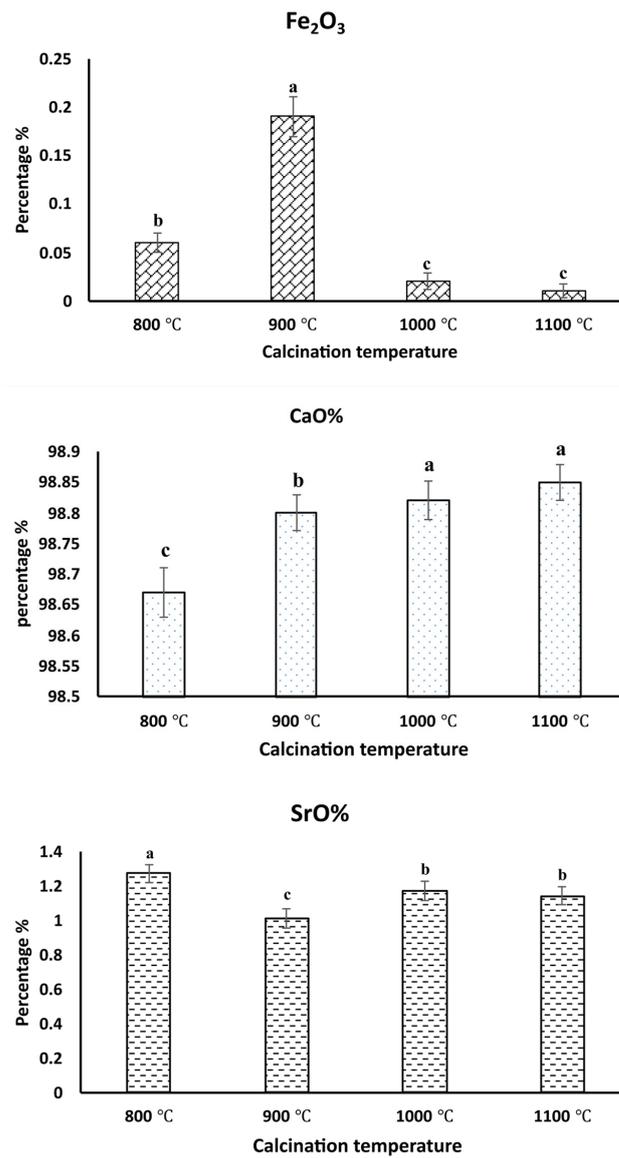


Figure 4. The average weight % of elements by XRF. (a–c) different letters means significant differences.

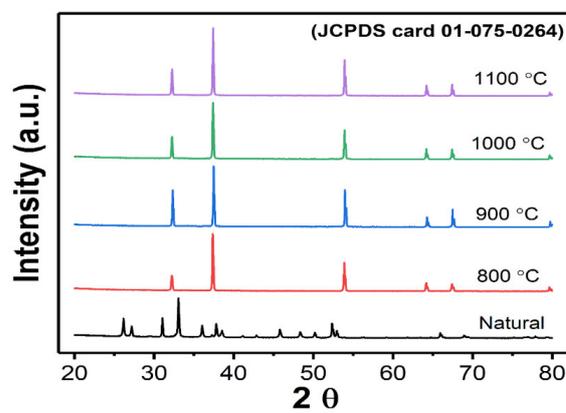


Figure 5. XRD patterns of natural and calcined mussel shells.

2.2.5. Morphological Properties

Scanning electron microscopy images at various magnifications are presented for mussel shells calcined at 800 °C (Figure 6), 900 °C (Figure 7), 1000 °C (Figure 8), and

1100 °C (Figure 9). In the SEM image of the sample calcined at 800 °C, the particles exhibit a relatively smooth and slightly cracked surface. However, in the sample calcined at 900 °C, the surface becomes rougher and more cracked; suggesting increased surface area and enhanced contact between reactants and the catalyst surface. Furthermore, the samples calcined at 1000 °C and 1100 °C display a similar morphology with a roughly textured surface, albeit less cracked than those calcined at 900 °C.

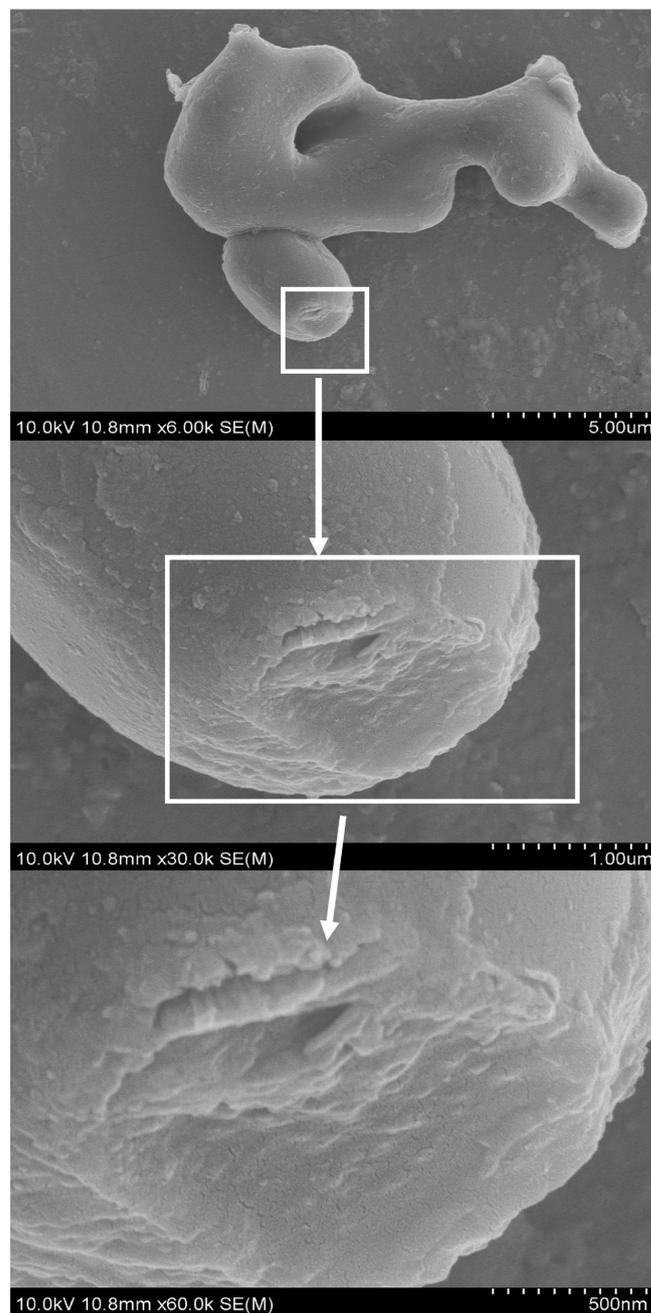


Figure 6. SEM image of a mussel shell subjected to a temperature of 800 °C for 4 h.

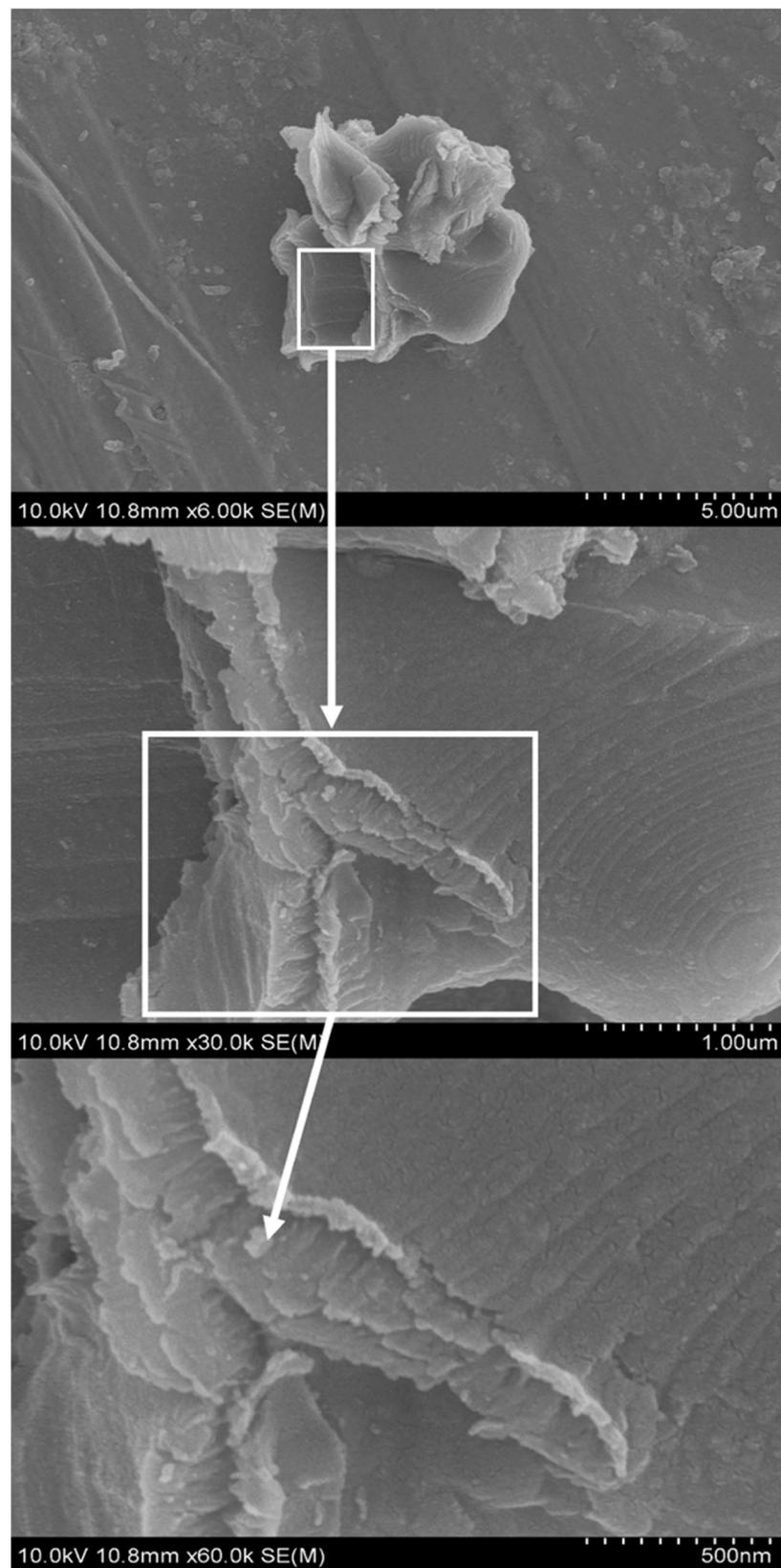


Figure 7. SEM image of a mussel shell subjected to a temperature of $900\text{ }^{\circ}\text{C}$ for 4 h.

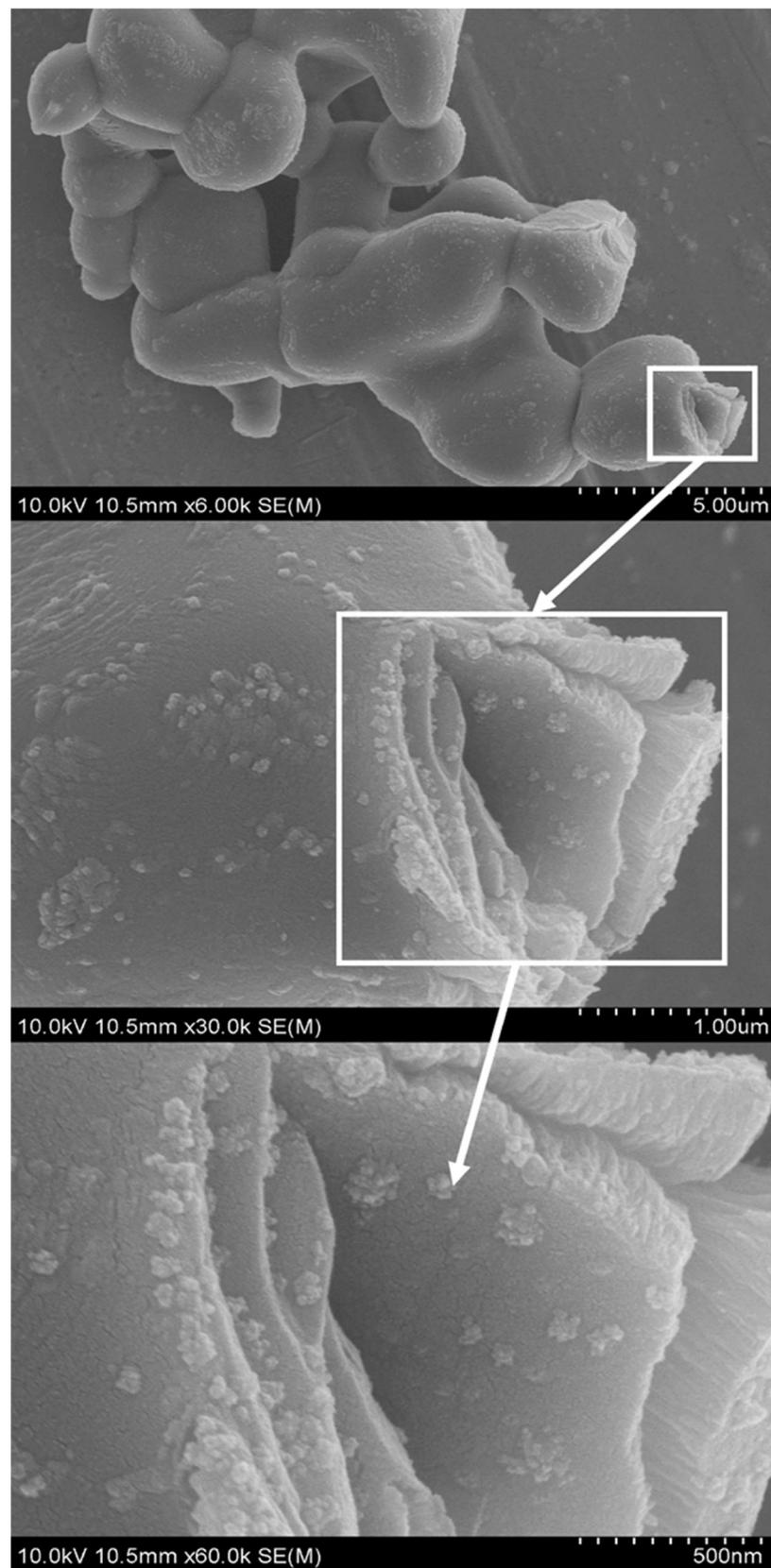


Figure 8. SEM image of a mussel shell subjected to a temperature of 1000 °C for 4 h.

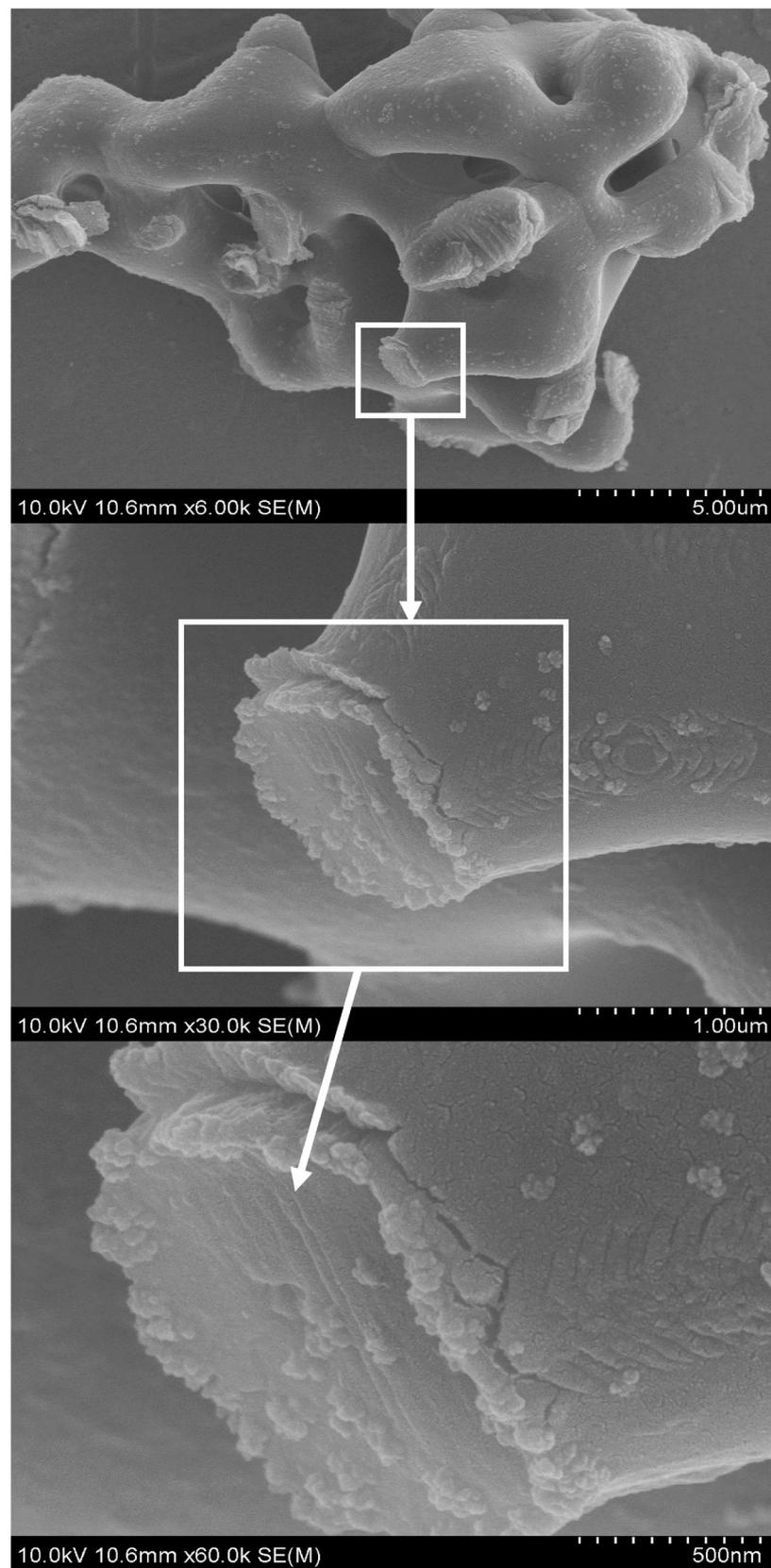


Figure 9. SEM image of a mussel shell subjected to a temperature of 1100 °C for 4 h.

2.2.6. Chemical and Physical Properties of *J. curcas* Methyl Ester and Number of Reusability

The results in Table 1 indicate that the prepared biodiesel meets most standards for diesel fuels, although it slightly exceeds the cloud point limit. However, its density (0.856 g/cm^3) and calorific value (38.140 MJ/kg) are within acceptable limits. The distillation profile ($98\text{--}309 \text{ }^\circ\text{C}$) suggests suitability for diesel engines. The flash point (110°C) exceeds the minimum requirement, and viscosity ($4.89 \text{ mm}^2/\text{s}$) falls within specified ranges for both standards.

Table 1. Chemical and Physical Properties of *J. curcas* Methyl Ester.

Contents	ASTM D-6751	Prepared Biodiesel	EN 14214
Cloud point	-3 to $15 \text{ }^\circ\text{C}$	0	-----
Pour point	-5 to 10 pp	-3 pp	-----
Calorific value	---	38.140 MJ/kg	32.9
Viscosity	$1.9\text{--}6 \text{ mm}^2/\text{s}$	$4.89 \text{ mm}^2/\text{s}$	$3.5\text{--}5 \text{ mm}^2/\text{s}$
Cetane number	47–65	50.5	51 to 120
Density at $15 \text{ }^\circ\text{C}$	$0.82\text{--}0.9 \text{ g/cm}^3$	0.856 g/cm^3	$0.86\text{--}0.9 \text{ g/cm}^3$
Flash point	$>120 \text{ }^\circ\text{C}$	$110 \text{ }^\circ\text{C}$	$>120 \text{ }^\circ\text{C}$
Oxidation stability	Max. 3 h	4.5 h	Min. 6 h
Sulfur content	$\leq 15 \text{ ppm}$	8 ppm	$\leq 10 \text{ ppm}$
Free glycerol content	$\leq 0.020\% \text{ wt.}\%$	0.011% wt.%	$\leq 0.020\% \text{ wt.}\%$
Total glycerol content	---	0.032%	Max. 0.25%
Iodine value	$\leq 120 \text{ I}_2/100 \text{ g}$	$99.2 \text{ I}_2/100 \text{ g}$	$\leq 120 \text{ I}_2/100 \text{ g}$
Phosphorus content	Max. 10 ppm	5 ppm	Max. 10 ppm
Water content	Max. 500 ppm	350 ppm	Max. 500 ppm
Ash content	Max. 0.01%	0.005%	Max. 0.02%
Carbon residue	Max. 0.050%	0.035%	Max. 0.20%
Acid value	$\leq 0.050 \text{ mg KOH/g}$	0.030 mg KOH/g	$\leq 0.050 \text{ mg KOH/g}$
Distillation profile Initial	Vol.	Temp. $^\circ\text{C}$	
boiling point	50 mL	191	
	80 mL	296	
	90 mL	309	

The catalyst, prepared with 6% by weight, calcined at 900°C , and a methanol-to-oil ratio of 18:1 at $90 \text{ }^\circ\text{C}$ for 6 h, was tested through multiple cycles. Results demonstrate that the calcined mussel shell can be reused up to five times, with FAME yields of 99.2%, 94.13%, 81.53%, 76.56%, and 66.41% for each cycle, as illustrated in Figure 10.

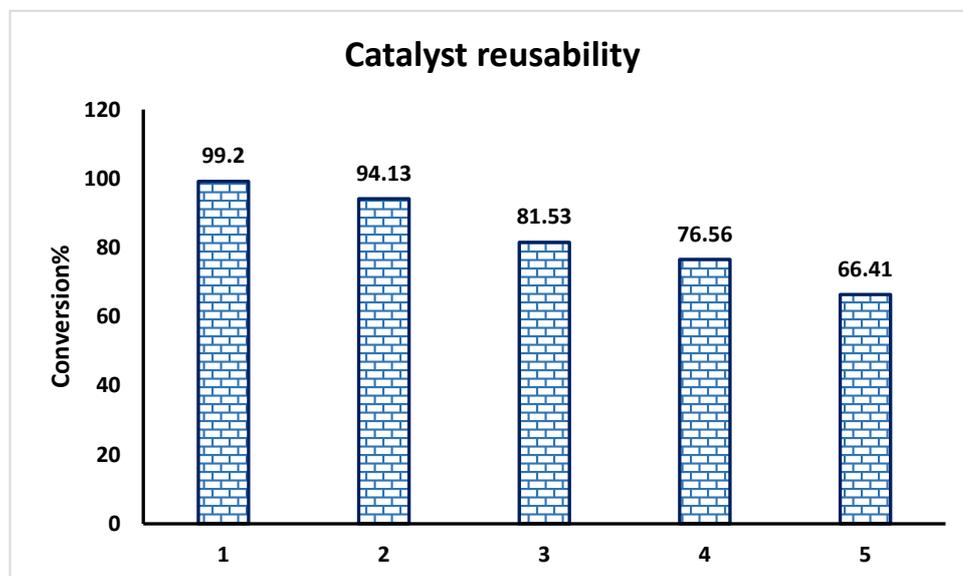


Figure 10. Number of reusability for the *Jatropha curcas* catalyst.

3. Discussion

Over the past decade, the world's energy demand has seen a significant increase, driven by rapid industrialization. Traditional energy sources not only lack sustainability but also pose environmental challenges. The expanding global industries have hastened the depletion of conventional resources [26]. Projections indicate a 40% increase in worldwide energy demand from 2010 to 2030. Biodiesel, a biofuel capable of substituting petroleum-based diesel, comprises a chemically intricate mixture of fatty acid mono-alkyl esters, primarily derived from plant oils or animal fats. Non-edible sources are favored for biodiesel production due to their cost-effectiveness and lack of competition with food supplies [27].

Jatropha curcas, an agricultural plant with abundant oil content, can thrive in marginal soils in tropical and subtropical climates. This plant offers various economic benefits while aiding soil improvement and erosion prevention. Its latex leaves and fruit can be used to make medicine, manure, and insect repellent. Additionally, press cake from *Jatropha* can be used to generate biogas. Over the last decade, numerous *Jatropha* projects worldwide have capitalized on the potential value chain it offers [28].

Catalysts play a crucial role in the transesterification process [29]. When transforming vegetable oils into biodiesel, heterogeneous catalysts show potential. Unlike homogeneous catalysts, heterogeneous ones can be reused, regenerated, and employed continuously [30]. Our study aimed to create biodiesel from crude *J. curcas* oil using mussel waste shells as a sustainable source for a heterogeneous catalyst. We conducted various tests to comprehend the catalyst's properties, including TGA, XRD, XRF, SEM, and FTIR.

Various factors impact the transesterification process, and their optimization is crucial to achieving high-quality biodiesel meeting regulatory standards. Each of these factors plays an equally vital role in the process.

Thermal stability (TGA) is a common method to determine the calcination temperature of biomass precursors, assessing compound loss at different temperatures [31]. Previous studies have reported the conversion of CaCO_3 into CaO at different calcination temperatures. Research by Rahman et al. [32] investigated the influence of calcination temperature (ranging from 800°C to 1100°C) on the transesterification process. It was found that calcining oyster shells at 800°C significantly enhances catalyst performance. TG/DTA research by Lin et al. [33] indicated that full conversion of CaCO_3 to CaO can be achieved by calcination at temperatures between 800°C and 1000°C . All calculations in our study were performed between 800°C and 1100°C for four hours. As a result, the sample's weight remained constant at 53.31 wt.% at 800°C , consistent with the expected weight change (44 wt.%) during the CaCO_3 to CaO conversion [34].

The fundamental properties of the CaO catalyst in both mussel shells and calcined mussel shells were evaluated through FTIR spectra conducted at temperatures of 800, 900, 1000, and 1100°C . CaCO_3 displays well-defined infrared bands at 705, 858, 1099, and 1457 cm^{-1} , corresponding to C–O's bending and stretching modes [35]. These bands show variations in intensity and characteristics during the calcination process at temperatures ranging from 800 to 1100°C , attributed to the thermal breakdown of CaCO_3 and the formation of $\text{Ca}(\text{OH})_2$ and CaO . An additional distinctive band at 3639 cm^{-1} arises from creating basic OH groups associated with Ca atoms [32]. Previous research studies [36] have reported similar findings, reinforcing the results of the DSC/TGA, XRD, and XRF tests.

X-ray fluorescence (XRF) results confirmed that the mussel shell catalysts contained over 98.60% CaO , consistent with DSC/TGA and FTIR data findings. The high calcium content indicates that the waste material was primarily composed of CaCO_3 , successfully transformed into CaO through calcination [37,38].

In this study, waste mussel shells underwent X-ray diffraction (XRD) examination to determine their chemical composition before and after calcination. Initially, the mussel shells had a CaCO_3 concentration ranging from 95% to 99%. After calcination, the shells appeared entirely white, indicating a complete conversion of CaCO_3 to CaO [37]. These

results align with the CaO signals observed in previous research [33,39]. It can be inferred that samples calcined at 800, 900, 1000, and 1100 °C for 4 h predominantly contained CaO, with no detectable CaCO₃ remaining, consistent with TGA and XRF analyses indicating a high CaO content.

The SEM images of CaO derived from mussel shells reveal surface morphology and area variations among different catalysts. When calcined at 900 °C, the particles exhibited rough surfaces with significant porosity due to the thermal modification of organic components within the mussel shell catalysts [37]. This phenomenon is in line with research trends exemplified in [38], which contribute valuable insights into the nuanced relationship between surface characteristics and overall catalyst efficiency since the diversity of catalysts and reaction systems discussed in these references broadens the context of our findings, reinforcing the significance of surface modifications in enhancing catalytic properties.

Regarding the impact of reaction time on biodiesel conversion, the data indicates that extending the transesterification time from 3 to 4 h increased the conversion percentage (from 40.90% to 46.54%). However, at 5 h, there was a decrease in yield (44.79%), possibly because the liquid product that had been deoxygenated broke down into lighter portions. Similarly, selectivity increased with a reaction time of 6 h (yielding 62.51%). This suggests that an extended reaction duration is required to improve the effectiveness of the interaction between the reactant's molecules and the catalyst's surface, promoting a more extensive reaction [40]. This is in line with the results of Nurdin et al. [41] and Mohammed et al. [42], which indicated that *Jatropha* oil achieved the highest conversion rate in 6 h. It is worth noting that Kamel et al. [43] also observed the effects of reaction duration on the transesterification process using *J. curcas* oil.

Temperature is another crucial factor that must be controlled to enhance biodiesel production. It has been observed that as the temperature increases, the reaction rate decreases, leading to lower yields, especially beyond 120 °C. The highest methyl ester yield, 77.78%, was achieved at 90 °C, but this yield decreased to 71.03%, 62.51%, and 50.53% at 100 °C, 110 °C, and 120 °C, respectively. This decrease is primarily due to higher temperatures causing methanol to evaporate, resulting in reduced yields [44]. Additionally, when the reaction temperature surpasses the optimal range, biodiesel yields drop due to triglycerides undergoing saponification [45–47]. The highest methyl ester production reached 86% under operational conditions after 6 h, highlighting the substantial impact of temperature on oil transesterification [48]. The initial increase in conversion from 3 to 6 h signifies the importance of extended transesterification time for biodiesel yield. This can be attributed to the completion of the transesterification reaction, allowing for a more thorough conversion of triglycerides to (FAMEs). However, the slight dip in conversion at 5 h may indicate a transitional phase where certain reactants are in the process of depletion or by-products start to influence the overall conversion rate. The subsequent rise to the highest conversion at 6 h suggests a continued positive effect of prolonged reaction time, allowing for further reaction completion and FAME formation.

One important aspect affecting the generation of biodiesel is the molar ratio of methanol to oil. The stoichiometric molar ratio for methanol to oil in transesterification is 3:1 [30,49]. A higher molar ratio is preferred to expedite the reaction and increase product generation [50,51]. The biodiesel output rose with an increase in the methanol-to-oil molar ratio, peaking at 99.36% at 18:1. However, as the ratio was further elevated to 21:1, the proportion of biodiesel output began to decline. A larger alcohol-to-oil molar ratio enhances glycerol solubility in biodiesel, with free glycerol forming droplets or dissolving in biodiesel. This occurs because alcohols can serve as co-solvents, enhancing the solubility of glycerol in biodiesel [52]. Several other studies also support the optimal molar ratio of 18:1 for achieving the highest biodiesel yield [53]. Concerning the methanol-to-oil molar ratio, the initial increase in yield up to a 1:18 molar ratio aligns with the conventional understanding that higher methanol concentrations facilitate faster reaction kinetics and enhance overall biodiesel production. The peak yield at this ratio could be attributed to

an optimal balance between reactants, ensuring sufficient methanol for transesterification without an excess that may lead to undesirable side reactions or hinder the reaction equilibrium. The subsequent decline in yield at a 1:21 molar ratio may be attributed to an excess of methanol, leading to diminishing returns as an excessively high concentration may disrupt the equilibrium of the transesterification reaction or promote unwanted side reactions, ultimately reducing overall biodiesel yield.

As scientists emphasize, optimizing the calcination temperature in catalyst production is crucial for achieving effective catalytic performance. The calcination process influences the structural and catalytic characteristics of catalysts, leading researchers to explore various calcination temperatures [54]. The XRD analysis confirmed that the high CaO concentration in the samples resulted in high yields across all tests. The maximum conversion rate of 98.52% was observed at 900 °C, aligning with SEM data showing a rougher, more fractured surface that increases the reaction's surface area. Due to less surface cracking, the yields for samples calcined at 1000 °C and 1100 °C were 92.93% and 87.73%, respectively. The temperature of 900 °C for the mussel shell catalyst, as per the existing literature [55], was selected as the optimal choice.

The catalyst's concentration plays a pivotal role in improving the yield of methyl ester synthesis [29]. The highest yield of 92.78% was achieved at a catalyst loading of 6 wt.%, indicating increased active catalytic sites with a higher quantity [48,56]. However, as the catalyst quantity was increased to 9–12%, the conversion efficiency dropped to 65.89% and 64.88%. This decrease can be attributed to excessive surface-active sites on the catalyst for reactant adsorption, leading to no improvement [40]. Several studies have reported that increasing the catalyst amount adversely affects yield [46,57].

4. Materials and Methods

4.1. Seed Material

We obtained local *Jatropha curcas* seeds from Ahmad Qashash farm in Al-Baha, KSA, and procured mussel shells from a local community market in Jeddah, KSA. We used high-purity methanol (99.9%) for the experiments. The *J. curcas* seeds were carefully sorted, with damaged seeds discarded. The selected, undamaged seeds were cleaned, de-shelled, and dried at 100–105 °C for 35 min.

4.2. Oil Extraction

We extracted oil from the seeds using a mechanical press technique [43,58]. Following extraction, the obtained oil was filtered, resulting in an approximate oil yield of 50% (as shown in Figure S1).

4.3. Catalyst Preparation

Mussel shells were collected from a local community market in Jeddah, KSA. The mussel shell waste underwent thorough cleaning with warm water, followed by drying at 100 °C in an oven for 4 h. Subsequently, the dried shell waste was crushed and sieved to achieve a particle size of 250 µm using a laboratory sieve shaker [56].

4.4. Catalyst Calcination

The cleaned and crushed mussel shell particles were divided into four groups and then calcined in a muffle furnace for 4 h at temperatures 800, 900, 1000, and 1100 °C. This calcination process transformed the mussel shells from CaCO₃ to CaO, resulting in a white powder that was stored in a silica gel desiccator [56].

4.5. Transesterification

J. curcas oil was converted into biodiesel in 3-neck flasks (250 mL) utilizing CaO obtained from mussel shells as a heterogeneous catalyst. This reaction occurred in a paraffin oil bath with magnetic stirring and a water-cooled condenser. Various conditions were explored, including catalyst loadings (3, 6, 9, 12 wt.%), catalyst calcination temperatures

(800, 900, 1000, 1100 °C), conversion time intervals (3, 4, 5, 6 h), transesterification temperatures (90, 100, 110, 120 °C), and oil/methanol ratios (12:1, 15:1, 18:1, 21:1). After each test, the solid catalyst was separated through centrifugation for 20 min at 4000 rpm, allowing for the isolation of methyl ester, glycerol, and the catalyst. Excess methanol was evaporated under a vacuum, and the filtrate liquid was collected in a glass-separating funnel [47,59–63].

Notably, to minimize methanol evaporation, a water-cooled condenser was used to control the vaporization of methanol and ensure a closed system. Additionally, efficient stirring techniques were employed to reduce the likelihood of methanol escaping further. Moreover, we preferred not to use transesterification temperatures exceeding 120 °C since beyond 120 °C, we anticipated potential complications such as increased energy consumption and undesired side reactions, which could affect the overall efficiency of the process. By limiting the temperature range, we aimed to balance maximizing biodiesel production and avoiding potential drawbacks associated with higher temperatures.

4.6. Catalyst Characterization

The resulting catalyst's physicochemical properties were assessed through several techniques:

- DSC/TGA Analysis: Thermal stability was assessed using differential scanning calorimetry thermogravimetric analysis in a compressed air flow, ramping from 35 to 1100 °C at 10 °C per minute.
- FTIR Analysis: Fourier-transform infrared spectroscopy with attenuated total reflection (ATR-FTIR) was employed to investigate surface structure properties and identify functional groups. This analysis covered a wave number range of 400–4000 cm^{-1} at a resolution of 4 cm^{-1} . The related data were recorded using Spectrum 10TM software (version 142) with a LiTaO₃ detector (New York, NY, USA), which was included with the device.
- XRF Analysis: The elemental compositions of both mussel shells and the resulting catalysts were determined using an X-ray fluorescence spectrophotometer (Horiba 7000 model, Montpellier, France).
- XRD Analysis: X-ray diffraction (XRD) was employed to determine the materials' crystalline phases. Using a Shimadzu Model XRD 6000 (Kyoto, Tapan), diffraction patterns were detected with 1.5406 of Cu-K generated at 40 kV and 40 mA tube voltage, radiating in a range of 20°–80°. The crystallographic phases of catalysts were determined using the Powder Diffraction File (PDF) database maintained by the Joint Committee on Powder Diffraction Standards (JCPDS).
- SEM Analysis: Scanning electron microscopy (SEM), facilitated by an SEM TM3030 instrument from Hitachi, Tokyo, Japan, was employed to examine the surface morphology of the CaO catalyst within the mussel shell.

4.7. Statistical Analysis

The statistical analysis was conducted using the least significant difference (LSD) test, and the interpretation of significant differences among the calcined catalyst temperatures was determined using letters from Duncan's multiple range test.

5. Conclusions

This study highlights the promising potential of mussel shell-derived CaO as a sustainable catalyst for converting *J. curcas* oil into biodiesel. Our eco-friendly and cost-effective approach aligns seamlessly with global initiatives for cleaner and economically viable energy sources. We have delved into the surface properties of the mussel shell-derived CaO catalyst using advanced characterization techniques such as X-ray photoelectron spectroscopy (XPS), Fourier-transform infrared spectroscopy (FTIR), and scanning electron microscopy (SEM). The catalyst exhibits outstanding performance in transesterification, yielding an impressive 99.36% FAME, and demonstrates exceptional reusability, surpassing traditional homogeneous catalysts in five cycles. The resulting biodiesel meets stringent

global standards, emphasizing its status as a cleaner and economically viable energy source. This research addresses environmental concerns and offers a compelling and cost-effective solution, marking a significant advancement toward a greener energy landscape. Repurposing waste mussel shells for CaO catalysts aligns with the global pursuit of sustainability in biodiesel production, contributing substantially to a more responsible and efficient energy future, bridging the gap between waste management, catalyst development, and sustainable energy production.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/catal14010059/s1>: Figure S1: Composition of methyl ester in biodiesel at different calcined catalyst temperature by GC–MS.

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