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# Catalytic Conversion of *Jatropha curcas* Oil to Biodiesel Using Mussel Shell-Derived Catalyst: Characterization, Stability, and Comparative Study

**Manal E. Shafi [1,\*], Halimah A. Alsabi [2], Suad H. Almasoudi [3], Faten A. M. Mufti [3], Safaa A. Alowaidi [2] and Alaa A. Alaswad [4]**

[1] Sustainable Agriculture Production Research Group, Department of Biological Sciences, Zoology, King Abdulaziz University, Jeddah 21589, Saudi Arabia
[2] Department of Biology, Faculty of Sciences, King Abdulaziz University, Jeddah 21589, Saudi Arabia
[3] Department of Biology, Faculty of Sciences, Umm Al-Qura University, Makkah 96612, Saudi Arabia
[4] Department of Biological Sciences, Faculty of Sciences, University of Jeddah, Jeddah 21589, Saudi Arabia
[\*] Correspondence: meshafi@kau.edu.sa

**Abstract:** Biodiesel represents a promising solution for sustainable energy needs, offering an eco-friendly alternative to conventional fossil fuels. In this research, we investigate the use of a catalyst derived from mussel shells to facilitate biodiesel production from *Jatropha curcas* oil. Our findings from X-ray Fluorescence (XRF) analysis emphasize the importance of carefully selecting calcination temperatures for mussel shell-based catalysts, with 1100 °C identified as optimal for maximizing CaO content. We identify a reaction time of 6 h as potentially optimal, with a reaction temperature of approximately 110 °C yielding the desired methyl ester composition. Notably, a methanol-to-oil ratio of 18:1 is the most favorable condition, and the optimal methyl ester composition is achieved at a calcined catalyst temperature of 900 °C. We also assess the stability of the catalyst, demonstrating its potential for reuse up to five times. Additionally, a thorough analysis of *J. curcas* Methyl Ester (JCME) biodiesel properties confirmed compliance with industry standards, with variations attributed to the unique characteristics of JCME. Comparing homogeneous (NaOH) and heterogeneous (CaO) catalysts highlights the potential of environmentally sourced heterogeneous catalysts to replace their homogeneous counterparts while maintaining efficiency. Our study presents a novel approach to sustainable biodiesel production, outlining optimal conditions and catalyst stability and highlighting additional benefits compared with NaOH catalysts. Therefore, utilizing mussel shell waste for catalyst synthesis can efficiently eliminate waste and produce cost-effective catalysts.

**Keywords:** *Jatropha curcas* oil; sustainable biodiesel production; optimization; stability; NaOH catalysts; CaO catalysts

## 1. Introduction

Biodiesel, a promising and environmentally friendly alternative to conventional fossil fuels, plays a pivotal role in mitigating the environmental and economic challenges associated with petroleum-based fuels [1]. Renewable feedstocks, including vegetable oils and animal fats, undergo a chemical process known as transesterification to produce biodiesel [2]. It offers benefits such as reduced greenhouse gas emissions, enhanced air quality, and a potential reduction in dependence on finite fossil fuel resources [3,4].

The environmental benefits of biodiesel are primarily attributed to its lower carbon footprint, as it is widely considered a carbon-neutral fuel [5]. This is due to the carbon dioxide ($CO_2$) released during its combustion being roughly equivalent to the $CO_2$ absorbed by the plants from which the feedstock was derived [6]. Moreover, biodiesel is biodegradable, less toxic, and produces lower sulphur and particulate matter emissions, enhancing its eco-friendly characteristics [6,7].

Transesterification, a vital step in biodiesel production, involves reacting feedstock materials with alcohol, typically methanol or ethanol, converting triglycerides into fatty acid methyl esters (FAMEs) [8]. The resulting glycerol byproduct holds economic potential, minimizing waste and enhancing production viability [9]. Biodiesel production utilizes acidic, alkaline, and biocatalysts [10], with alkaline catalysts generally preferred despite challenges in post-reaction treatments, glycerol recovery, and energy demands [11]. Acid-catalyzed transesterification, though slower, requires higher alcohol-to-oil ratios. Lipases offer renewable residues and higher thermostability as enzymatic catalysts but are limited by cost and prolonged reaction times [12].

Catalysts play a crucial role in the pursuit of sustainable and cost-effective biodiesel production. They accelerate the transesterification reaction, improve conversion efficiency, and influence biodiesel quality [13]. Biodiesel production commonly uses traditional homogeneous catalysts like sodium hydroxide (NaOH) or potassium hydroxide (KOH) [14]. Despite their effectiveness, these catalysts have drawbacks, including challenges in separating the catalyst from the biodiesel and the requirement for additional processing to remove excess catalyst from the final product [14,15].

Heterogeneous catalysts have emerged as a promising solution to address these issues. Unlike homogeneous catalysts, they remain in a solid phase during the reaction and facilitate easy separation from the biodiesel, thereby simplifying the purification process [16]. When properly designed and selected, heterogeneous catalysts can offer high catalytic activity and reusability [17]. In laboratory settings, various heterogeneous catalysts have been developed for biodiesel synthesis, including alkali and alkaline earth metal oxides, cation-exchange resins, zeolites, and magnesium–aluminum mixed oxides. However, creating highly active heterogeneous catalysts is complex and costly. Thus, finding an optimal, cost-effective solid basic catalyst for biodiesel production is a significant challenge. Utilizing natural calcium sources from waste shells as a cheap catalyst is becoming popular [18]. Exploring alternative solid catalysts, including magnetic ones, has become crucial for green and durable biodiesel production. Magnetic catalysts, known for their easy separation with minimal mass loss, are particularly notable for enhancing efficiency and sustainability in biodiesel production [19,20]. Researchers have explored using waste freshwater mussel shells, eggshells, crab shells, golden apple snail shells, and meretrix venus shells as catalysts for biodiesel production [18,21–23]. These waste shell-derived catalysts show promise due to their affordability and eco-friendly characteristics.

Calcium oxide (CaO) is a promising heterogeneous catalyst for transesterification reactions [24]. It is stable and effective in many chemical processes, such as biodiesel production [25]. Utilizing CaO derived from discarded mussel shells as a catalyst presents an opportunity to convert waste materials into valuable resources [26]. Mussel shells, often considered a waste product, contain calcium carbonate ($CaCO_3$), which can be converted into CaO through calcination. This innovative approach not only offers an environmentally friendly alternative to homogeneous catalysts but also has the potential to enhance the economic sustainability of biodiesel production [27]. A low-cost catalyst derived from waste freshwater mussel shells was synthesized using a calcination–impregnation–activation method and utilized in Chinese tallow oil transesterification. The catalyst features a unique "honeycomb" structure [18].

Rezaei et al. [28] investigated the use of waste mussel shells as a calcium oxide catalyst for biodiesel production. Transesterification reactions were conducted with soybean oil, methanol, and the mussel shell catalyst at 60 °C. Response surface methodology (RSM) was employed to assess the impact of various parameters such as calcination temperature, catalyst concentration, and methanol-to-oil ratio. However, the study found that reusing the mussel shell catalyst negatively affected biodiesel yield after five cycles due to catalyst re-calcination. Khan et al. [29] found high catalytic activity in the transesterification of castor oil and methanol, yielding 87.42% fatty acid methyl ester (FAME) with a 7% Pr-CaO mixed-oxide catalyst under optimal conditions (2.5 wt% catalyst, methanol-to-oil ratio of 8:1, and 65 °C).

The *Jatropha curcas* tree yields approximately 60% oil from its seed kernel. While *J. curcas* oil requires detoxification before consumption, it serves as a valuable source of energy and fuel [30]. Buasri et al. [30] used calcined arcuate mussel shells and dolomitic rock as catalysts for the transesterification of *Jatropha curcas* oil with methanol. The dolomitic rock-derived catalyst, calcined at 900 °C for 2 h, showed smaller CaO crystallites and higher basicity compared to the mussel shell-derived catalyst, likely due to dispersed MgO in the calcined dolomite's CaO matrix. Taufiq-Yap et al. [31] found that clam shells, when calcined at 900 °C for 3 h, can yield active CaO catalysts for converting non-edible *Jatropha curcas* oil into biodiesel. Under optimized conditions (6 h at 65 °C), they achieved a remarkable 93% biodiesel yield.

In our previous study [32], we pioneered the use of mussel shell-derived calcium oxide (CaO) as a novel catalyst to produce biodiesel from *Jatropha curcas* oil, achieving an impressive 99.36% fatty acid methyl ester (FAME) yield. This work laid the groundwork for sustainable biodiesel production by repurposing waste materials, marking a significant contribution to the field. Building on this foundation, the present study seeks to address key questions that emerged from our initial findings, specifically focusing on optimizing catalyst preparation and reaction conditions, assessing catalyst stability over multiple cycles, and comparing the efficacy of mussel shell-derived CaO against traditional NaOH catalysts. The necessity of this new study arises from the imperative to enhance the operational feasibility and environmental sustainability of biodiesel production, aiming to refine and expand the applicability of our innovative catalytic process. Through rigorous experimentation and analysis, we endeavor to provide comprehensive insights into the optimal conditions for biodiesel synthesis, contributing to the ongoing development of cost-effective and eco-friendly energy solutions.

## 2. Results

Our findings build upon the promising results of our prior work [32], presenting a detailed analysis of the catalyst's performance across successive biodiesel production cycles. Unlike the initial study, which primarily focused on optimizing reaction conditions for maximum FAME yield, the current research emphasizes the catalyst's durability and its potential for reuse.

### 2.1. Determination of Fatty Acid Compositions by XRF

Table 1 presents the average weight percentages of elements determined by X-ray Fluorescence (XRF) analysis at different calcination temperatures. The table highlights the elemental composition of the samples post-calcination, showcasing the stability and minor variations in the elemental percentages with increasing temperature. The results reveal the compositions of the catalyst derived from mussel shells, primarily focusing on the percentages of CaO, $Fe_2O_3$, and SrO. At 800 °C, the compositions consist of 98.67% CaO, 0.06% $Fe_2O_3$, and 1.27% SrO. Increasing the calcination temperature to 900 °C leads to a marginal rise in CaO to 98.80%, a slight increase in $Fe_2O_3$ to 0.19%, and a decrease in SrO to 1.01%. The trend continues at 1000 °C, with 98.82% CaO, 0.02% $Fe_2O_3$, and 1.17% SrO. At the highest temperature of 1100 °C, the composition records 98.85% CaO, 0.01% $Fe_2O_3$, and 1.14% SrO.

**Table 1.** The average weight percentages of CaO, $Fe_2O_3$, and SrO at various calcination temperatures determined by XRF analysis.

| Calcination Temperature * | CaO% | $Fe_2O_3$% | SrO% |
| --- | --- | --- | --- |
| 800 °C | 98.67 ± 0.08 | 0.06 ± 0.02 | 1.27 ± 0.05 |
| 900 °C | 98.80 ± 0.07 | 0.19 ± 0.04 | 1.01 ± 0.02 |
| 1000 °C | 98.82 ± 0.04 | 0.02 ± 0.01 | 1.17 ± 0.04 |
| 1100 °C | 98.85 ± 0.06 | 0.01 ± 0.01 | 1.14 ± 0.02 |

* The experiments involved calcining samples at temperatures ranging from 800 °C to 1100 °C. The calcination was carried out in air for a duration of 2 h at each specified temperature. Following calcination, the samples were analyzed using X-ray Fluorescence (XRF) to determine the average weight percentages of CaO, $Fe_2O_3$, and SrO. Standard deviations are provided to indicate the reproducibility of the results.

### 2.2. Composition of Methyl Ester in Biodiesel at Different Reaction Times by GC–MS

Table 2 presents the concentration percentages of various methyl esters in biodiesel, as determined by GC–MS, across four different reaction times (3 h, 4 h, 5 h, and 6 h). These findings illustrate how the composition of biodiesel evolves over time, with a specific focus on the increase in concentration of certain esters, indicative of transesterification process efficiency. Notably, Palmitic Acid ME increased concentration over the reaction time, starting at 6.77% at 3 h and reaching 10.72% at 6 h, as shown in (Table 2, Figure S1). Similarly, both Linoleic Acid ME and Oleic Acid ME showed an upward trend in concentration, with Linoleic Acid ME increasing from 14.69% at 3 h to 21.55% at 6 h and Oleic Acid ME rising from 15.53% to 23.03% over the same period. On the other hand, Stearic Acid ME demonstrated a gradual increase in concentration from 3.89% at 3 h to 6.16% at 6 h. The data underscore the impact of reaction duration on the yield and composition of biodiesel, providing valuable insights for optimizing production parameters.

**Table 2.** Composition of methyl ester in biodiesel at different reaction times by GC–MS.

| Components Methyl Ester * | Concentration (%) | | | |
|---|---|---|---|---|
| | 3 h | 4 h | 5 h | 6 h |
| Palmitoleic ME | – | – | – | $0.53 \pm 0.02$ |
| Palmitic Acid ME | $6.77 \pm 0.14$ | $7.44 \pm 0.03$ | $7.51 \pm 0.05$ | $10.72 \pm 1.82$ |
| Linoleic Acid ME | $14.69 \pm 0.34$ | $16.28 \pm 0.23$ | $15.95 \pm 0.72$ | $21.55 \pm 2.95$ |
| Oleic Acid ME | $15.53 \pm 0.52$ | $18.47 \pm 0.61$ | $16.81 \pm 0.62$ | $23.03 \pm 3.25$ |
| Stearic acid ME | $3.89 \pm 0.02$ | $4.33 \pm 0.02$ | $4.50 \pm 0.15$ | $6.16 \pm 0.90$ |

* The table showcases the composition of methyl esters in biodiesel derived from *Jatropha curcas* oil, analyzed at various reaction times (3, 4, 5, and 6 h) using Gas Chromatography–Mass Spectrometry (GC–MS). These biodiesel samples were prepared through a transesterification process in 3-neck flasks at 110 °C, employing a methanol-to-oil ratio of 18:1 and mussel shell-derived CaO as a catalyst, which was calcined at temperatures ranging from 800 °C to 1100 °C. The transesterification reaction was facilitated by magnetic stirring in a paraffin oil bath with a water-cooled condenser to minimize methanol evaporation, ensuring the integrity of the reaction environment.

The absence of data for palmitoleic ME suggests that it may not be present in the biodiesel under the given conditions or may be present in quantities below the detection limit. Overall, the table provides insights into the dynamic changes in the composition of methyl esters during the biodiesel production process, which can be crucial for optimizing reaction times and product quality.

### 2.3. Composition of Methyl Ester in Biodiesel at Different Reaction Temperatures by GC–MS

Table 3 and Figure S2 illustrate the variations in the composition of methyl esters in biodiesel, as analyzed by Gas Chromatography–Mass Spectrometry (GC–MS), under different reaction temperatures (90 °C, 100 °C, 110 °C, and 120 °C). Notably, Palmitoleic ME is detected only at 110 °C with a concentration of 0.53%. Palmitic Acid ME exhibits fluctuations in concentration with temperature, starting at 10.67% at 90 °C, decreasing to 9.42% at 100 °C, then increasing to 10.72% at 110 °C before declining to 7.31% at 120 °C. Linoleic Acid ME and Oleic Acid ME both show an upward trend in concentration with increasing temperature, peaking at 21.55% and 23.03%, respectively, at 110 °C. In contrast, Stearic Acid ME demonstrates varying concentration with temperature, reaching a maximum of 6.16% at 110 °C. Accordingly, the ideal reaction temperature for biodiesel production appears to be around 110 °C, facilitating the desired methyl ester composition with exclusive Palmitoleic ME detection and increased levels of Linoleic Acid ME and Oleic Acid ME.

**Table 3.** Composition of methyl ester in biodiesel at different reaction temperatures (90 °C, 100 °C, 110 °C, and 120 °C) by GC–MS.

| Components Methyl Ester * | Concentration (%) | | | |
|---|---|---|---|---|
| | 90 °C | 100 °C | 110 °C | 120 °C |
| Palmitoleic ME | – | – | $0.53 \pm 0.03$ | – |
| Palmitic Acid ME | $10.67 \pm 0.90$ | $9.42 \pm 0.80$ | $10.72 \pm 1.30$ | $7.31 \pm 0.50$ |

**Table 3.** *Cont.*

| Components Methyl Ester * | Concentration (%) | | | |
|---|---|---|---|---|
| | **90 °C** | **100 °C** | **110 °C** | **120 °C** |
| Linoleic Acid ME | 12.39 ± 1.10 | 12.15 ± 1.30 | 21.55 ± 2.50 | 9.04 ± 0.90 |
| Oleic Acid ME | 25.93 ± 1.50 | 23.99 ± 2.15 | 23.03 ± 2.70 | 13.52 ± 1.30 |
| Stearic acid ME | 5.12 ± 0.5 | 4.88 ± 0.75 | 6.16 ± 0.80 | 4.034 ± 0.50 |

\* The experiment demonstrates how specific temperatures favor the production of certain methyl esters, with a notable increase in Linoleic Acid ME concentration at 110 °C, emphasizing the critical role of reaction temperature in optimizing biodiesel synthesis.

## 2.4. Composition of Methyl Ester in Biodiesel at Different Methanol-to-Oil Ratios Using GC–MS

Table 4 provides insights into the composition of methyl esters in biodiesel at different methanol-to-oil ratios, analyzed using Gas Chromatography–Mass Spectrometry (GC–MS), as shown in Figure S3. Optimal results appear to be achieved at an 18:1 methanol-to-oil ratio. Palmitoleic ME is notably absent at the lowest (12:1) and highest (21:1) ratios, indicating its optimal presence at the 15:1 and 18:1 ratios. Palmitic Acid ME shows an increasing trend from 10.67% at 12:1 to a peak of 16.12% at 18:1 before decreasing at 21:1. Linoleic Acid ME and Oleic Acid ME exhibit similar patterns, peaking at 33.25% and 34.80%, and 32.36% and 37.69%, respectively, at the 15:1 and 18:1 ratio. Stearic Acid ME follows a similar trend, reaching its maximum of 8.281% at an 18:1 ratio. Crotonic Acid ME is detectable only at the 18:1 ratio, with a concentration of 0.11%. These findings collectively suggest that the 18:1 methanol-to-oil ratio is optimal for achieving the desired methyl ester composition in biodiesel, underscoring the importance of this ratio in optimizing biodiesel production conditions.

**Table 4.** Composition of methyl ester in biodiesel at different methanol-to-oil ratios (12:1, 15:1, 18:1, and 21:1) by GC–MS.

| Components Methyl Ester * | Concentration (%) | | | |
|---|---|---|---|---|
| | **12:1** | **15:1** | **18:1** | **21:1** |
| Palmitoleic ME | – | 1.00 ± 0.02 | 0.97 ± 0.01 | – |
| Palmitic Acid ME | 10.67 ± 0.90 | 12.90 ± 1.30 | 16.12 ± 2.20 | 8.65 ± 0.80 |
| Linoleic Acid ME | 12.39 ± 1.50 | 33.25 ± 3.10 | 32.36 ± 3.30 | 19.22 ± 2.30 |
| Oleic Acid ME | 25.93 ± 2.30 | 34.80 ± 3.50 | 37.69 ± 3.50 | 27.32 ± 3.70 |
| Stearic acid ME | 5.12 ± 0.70 | 8.50 ± 0.95 | 8.281 ± 1.20 | 4.29 ± 0.30 |
| Crotonic acid ME | – | – | 0.11 ± 0.01 | – |

\* This table displays the composition of various methyl esters in biodiesel at differing methanol-to-oil ratios (12:1, 15:1, 18:1, 21:1), analyzed via GC–MS. These measurements were obtained from biodiesel synthesized through transesterification using a heterogeneous catalyst derived from calcined mussel shells, with the reaction conducted at an optimal temperature of 110 °C for a duration of 6 h. The methanol-to-oil ratios were meticulously chosen to investigate their effect on the yield and composition of biodiesel, providing insights into the efficiency of the transesterification process under varying conditions. This experiment underscores the significance of the methanol-to-oil ratio in biodiesel production, demonstrating that a ratio of 18:1 is particularly effective for enhancing the production of key fatty acid methyl esters, thereby optimizing the overall yield and quality of the biodiesel.

## 2.5. Composition of Methyl Ester at Different Calcined Catalyst Temperatures by GC–MS

Examining the methyl ester composition in biodiesel across different calcined catalyst temperatures (800 °C, 900 °C, 1000 °C, 1100 °C), as shown in Table 5 and Figure S4, reveal noteworthy trends. The optimal conditions for Palmitoleic ME detection lie at 800 °C and 900 °C, showcasing concentrations of 0.94% and 0.97%, respectively. In contrast, Palmitic Acid ME decreases from 18.65% at 800 °C to 13.67% at 1100 °C. Linoleic Acid ME and Oleic Acid ME maintain relatively stable concentrations, with Linoleic Acid ME ranging from 32.06% to 28.33% and Oleic Acid ME fluctuating from 35.76% to 36.66%. Stearic Acid ME follows suit, decreasing from 8.06% to 7.21%. Notably, Crotonic Acid ME is detectable only at 900 °C, with a concentration of 0.11%. These findings collectively suggest that the optimal calcined catalyst temperature for achieving the desired methyl ester composition

in biodiesel production may lie within 900 °C, presenting a crucial parameter for further optimization efforts.

**Table 5.** Composition of methyl ester in biodiesel at different calcined catalyst temperatures (800 °C, 900 °C, 1000 °C, and 1100 °C) by GC–MS.

| Components Methyl Ester * | Concentration (%) | | | |
| --- | --- | --- | --- | --- |
| | 800 °C | 900 °C | 1000 °C | 1100 °C |
| Palmitoleic ME | 0.94 ± 0.02 | 0.97 ± 0.03 | – | – |
| Palmitic Acid ME | 18.65 ± 2.40 | 16.12 ± 1.50 | 16.14 ± 1.60 | 13.67 ± 1.50 |
| Linoleic Acid ME | 32.06 ± 3.30 | 32.36 ± 2.30 | 32.39 ± 3.50 | 28.33 ± 2.50 |
| Oleic Acid ME | 35.76 ± 3.50 | 37.69 ± 3.30 | 36.97 ± 3.20 | 36.66 ± 3.90 |
| Stearic acid ME | 8.06 ± 0.90 | 8.281 ± 0.90 | 8.28 ± 0.90 | 7.21 ± 1.10 |
| Crotonic acid ME | – | 0.11 ± 0.01 | – | – |

* Each reaction was conducted under standardized conditions—specifically, a methanol-to-oil ratio of 18:1 at a reaction temperature of 110 °C for 6 h—to ensure that the observed effects on methyl ester composition were attributable solely to changes in catalyst calcination temperature. The presence and concentration of various methyl esters, including Palmitoleic ME, Palmitic Acid ME, Linoleic Acid ME, Oleic Acid ME, Stearic Acid ME, and Crotonic Acid ME, highlight catalytic performance nuances at different temperatures, with 900 °C showing a notably balanced profile for most components.

### 2.6. Compositions of Methyl Ester at Different Calcined Catalyst Concentrations by GC–MS

Table 6 provides a comprehensive overview of methyl ester composition in biodiesel at varying catalyst concentrations, as shown in Figure S5. Notably, Palmitoleic ME is detectable only at a 6 wt% catalyst concentration, showcasing a concentration of 0.97%. Palmitic Acid ME exhibits fluctuations in concentration, reaching its peak at 16.12% with a 6 wt% catalyst. At the same time, Linoleic Acid ME and Oleic Acid ME follow similar patterns, peaking at 32.36%, 37.69%, 19.84% and 23.02%, respectively, at a 6 wt% catalyst. Stearic Acid ME displays a comparable trend, reaching its highest concentration of 8.281% at the 6 wt% catalyst. Crotonic Acid ME is detectable only at a 6 wt% catalyst concentration, with a concentration of 0.11%.

**Table 6.** Composition of methyl ester in biodiesel at different catalyst concentrations (3 wt%, 6 wt%, 9 wt%, and 12 wt%) by GC–MS.

| Components Methyl Ester * | Concentration (%) | | | |
| --- | --- | --- | --- | --- |
| | 3 wt% | 6 wt% | 9 wt% | 12 wt% |
| Palmitoleic ME | – | 0.97 ± 0.02 | – | – |
| Palmitic Acid ME | 8.96 ± 0.60 | 16.12 ± 1.60 | 8.94 ± 0.60 | 9.56 ± 0.80 |
| Linoleic Acid ME | 19.49 ± 1.30 | 32.36 ± 3.30 | 19.84 ± 2.30 | 21.91 ± 2.30 |
| Oleic Acid ME | 25.35 ± 2.20 | 37.69 ± 3.20 | 23.02 ± 2.50 | 24.09 ± 2.90 |
| Stearic acid ME | 4.80 ± 0.70 | 8.281 ± 0.80 | 4.69 ± 0.70 | 4.79 ± 0.50 |
| Crotonic acid ME | – | 0.11 ± 0.01 | – | – |

* This experiment utilized a mussel shell-derived CaO catalyst, prepared through calcination and applied to the transesterification of *Jatropha curcas* oil, to explore the effect of catalyst concentration on the efficiency of biodiesel production. Conducted at an optimal reaction temperature of 110 °C with a methanol-to-oil ratio of 18:1 over a 6 h period, the study aimed to identify the concentration that maximizes biodiesel yield while maintaining the desired methyl ester profile. Notably, a 6 wt% catalyst concentration emerged as particularly effective, enhancing the presence of key fatty acid methyl esters such as Palmitic Acid ME and Oleic Acid ME, thereby underscoring the critical role of catalyst quantity in achieving an optimal biodiesel composition.

### 2.7. Catalyst Stability

The reusability of heterogeneous catalysts is vital both environmentally and economically. To assess this, the catalyst was subjected to multiple cycles, prepared with 6% by weight, calcined at 900 °C, and a methanol-to-oil ratio of 18:1 at 90 °C for 6 h. Figure 1 demonstrates that the calcined mussel shell can be reused up to five times, with FAME yields of 94.13%, 81.53%, 76.56%, and 66.41% for each cycle.
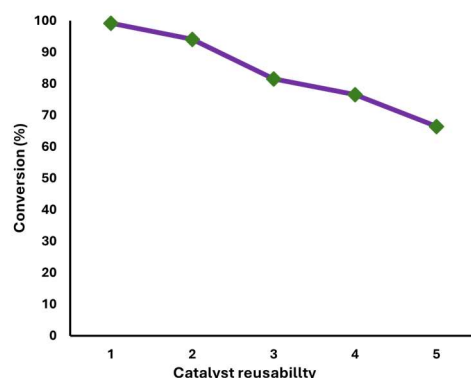
**Figure 1.** Conversion over a number of catalyst cycles, demonstrating catalyst reusability.

### 2.8. Biodiesel Characterization

The results of various physicochemical tests conducted under the optimized conditions for transesterifying *J. curcas* oil were compared with the international standards EN–14214 and ASTM D-6751. Table 7 presents the data, indicating that the produced biodiesel complies with the specifications outlined in ASTM D-6571 and EN 14214. These results are consistent with previous research on *J. curcas* oil methyl ester. The viscosity of the prepared biodiesel falls within the range of 3.5–5 mm$^2$/s, aligning with both standards. The flash point is slightly lower at 110 °C compared to the standards of >120 °C. The cloud point, pour point, cetane number, and density at 15 °C all meet or closely approximate the specified standards. Notably, the calorific value of the prepared biodiesel is 38.140 MJ/kg, surpassing the standards. The distillation profile further illustrates the temperature at which different volumes evaporate, with a residue percentage of 7% and a loss percentage of 1%. Based on the properties listed in Table 7, it is evident that *J. curcas* oil is suitable for biodiesel conversion.

**Table 7.** Chemical and physical properties of JCME.

| Contents | Unit | ASTM D-6751 | EN 14214 | Prepared Biodiesel |
|---|---|---|---|---|
| Viscosity | mm$^2$/s | 1.9–6 | 3.5–5 | 4.89 |
| Flash point | °C | >120 | >120 | 110 |
| Cloud point | °C | –3 to 15 | —— | 0 |
| Pour point | pp | –5 to 10 | —— | –3 |
| Cetane number | | 47–65 | 51 to 120 | 50.5 |
| Density at 15 °C | g /cm$^3$ | 0.82–0.9 | 0.86–0.9 | 0.856 |
| Calorific value | MJ/kg | —— | 32.9 | 38.140 |
| | Volume | Temp. °C | | |
| | 10 mL | 98 | | |
| | 20 mL | 106 | | |
| | 30 mL | 154 | | |
| | 40 mL | 172 | | |
| Distillation profile Initial boiling point | 50 mL | 191 | | |
| | 60 mL | 228 | | |
| | 70 mL | 268 | | |
| | 80 mL | 296 | | |
| | 90 mL | 309 | | |
| | Residue % | 7 | | |
| | Loss % | 1 | | |

### 2.9. Comparison of Homogeneous and Heterogeneous Catalysts

Table 8 delineates the methyl ester composition in biodiesel, employing two distinct catalysts, namely mussel shell-derived CaO and NaOH, as analyzed by GC–MS and indicated in Figure S6. The results illuminate notable variations in methyl ester concentrations based on the catalyst employed. CaO derived from mussel shells predominantly fosters

the formation of Palmitic Acid ME (16.12%), Linoleic Acid ME (32.36%), Stearic Acid ME (8.28%), Crotonic Acid ME (0.11%), Oleic Acid ME (37.69%), Palmitoleic ME (0.97%), Acid ME, Arachidic Acid ME, and Behenic Acid ME. In contrast, the use of NaOH as a homogeneous catalyst induces a distinct composition, marked by higher concentrations of Oleic Acid ME (86.32%), Palmitoleic ME (9.31%), Acid ME (0.75%), Arachidic Acid ME (1.75%), and Behenic Acid ME (0.43%). This comparison underscores the catalyst-dependent nature of methyl ester composition in biodiesel production, shedding light on the distinct profiles facilitated by mussel shell-derived CaO and NaOH. A schematic structure of the JCME oil components is also shown in Figure 2.

**Table 8.** Composition of methyl ester in biodiesel using mussel shells and NaOH by GC–MS.

| Components Methyl Ester * | Concentration of JCME (%) | |
|---|---|---|
| | (CaO) Mussel Shell | NaOH |
| Palmitic acid ME | 16.12 | – |
| Linoleic acid ME | 32.36 | – |
| Stearic acid ME | 8.28 | – |
| Crotonic acid ME | 0.11 | – |
| Oleic acid ME | 37.69 | 86.32 |
| Palmitoleic ME | 0.97 | 9.31 |
| Gondoic acid ME | – | 0.75 |
| Arachidic acid ME | – | 1.75 |
| Behenic acid ME | – | 0.43 |

* This analysis, performed by GC–MS, reveals distinct variations in the fatty acid methyl ester profiles of *Jatropha curcas* Methyl Ester (JCME), depending on the catalyst used. The experiments were carried out under optimized conditions for each catalyst type to fairly compare their efficacy in biodiesel production. Specifically, the reaction with the mussel shell-derived CaO catalyst was conducted at an optimal temperature of 110 °C, with a methanol-to-oil ratio of 18:1, highlighting the catalyst's capability to produce a rich profile of essential methyl esters for quality biodiesel. In contrast, the use of NaOH resulted in a significantly different ester composition, notably a higher concentration of Oleic Acid ME and the presence of esters not detected with the CaO catalyst.
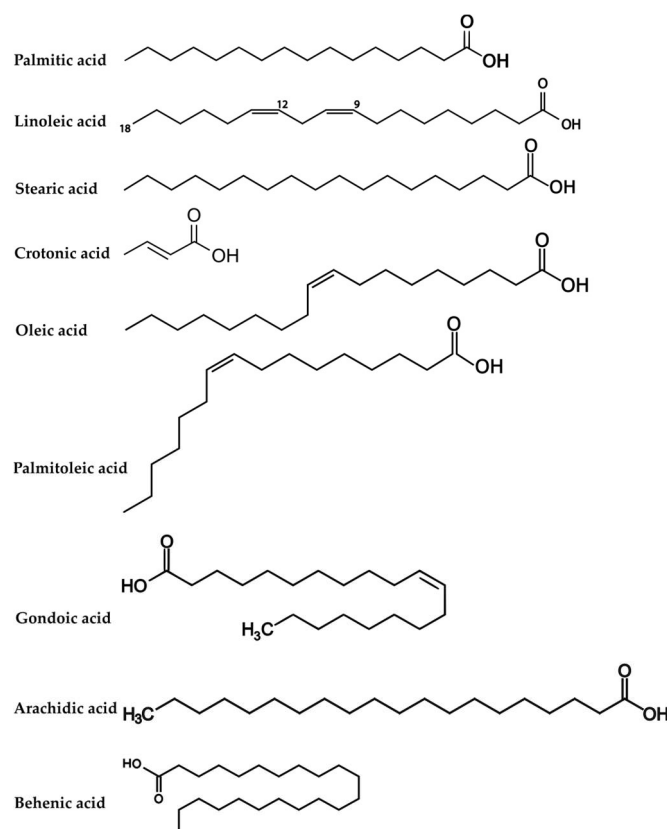


**Figure 2.** A scheme showing the structures of the JCME oil components.

## 3. Discussion

### 3.1. Determination of Fatty Acid Composition

The physicochemical properties of jatropha oil were provided in a previous study by Lee et al. [33]. The oil exhibits specific gravity ranging from 0.860 to 0.933, a flash point of 210–240 °C, and a viscosity of 37.0–54.8 cSt at 40 °C. The fatty acid composition includes palmitic acid, stearic acid, oleic acid, and linoleic acid, with other fatty acids present in trace amounts, including myristic, arachidic, behenic, lignoceric, palmitoleic, erucic, and linolenic acids [33]. Notably, the increasing trend in CaO percentages with rising calcination temperatures (98.67% at 800 °C to 98.85% at 1100 °C) suggests a temperature-dependent synthesis of this key catalytic component [34]. This aligns with the work of Suwannasingha et al. [35], who highlighted the impact of calcination temperature on the phase transformation of $CaCO_3$ to CaO in natural sources like mussel shells. The marginal fluctuations in $Fe_2O_3$ and SrO across the temperatures and the stability of these elements in calcined materials are consistent with the findings of [36].

The observed trend indicates that higher calcination temperatures contribute to the enhanced formation of CaO, known for its catalytic activity in transesterification reactions during biodiesel production [37]. This aligns with the literature, where researchers have highlighted the significance of CaO in facilitating the transesterification process [38]. The optimization of calcination temperature is essential as it directly influences the composition and catalytic effectiveness of the mussel shell-derived catalyst [29]. The substantial concentration of CaO at 1100 °C suggests the potential for improved catalytic performance [39] and the relationship between CaO concentration and catalyst activity [40].

The primary element of vegetable oil is triglyceride, composed of various fatty acids linked to glycerin, making the nature of these fatty acids crucial for the physicochemical properties of fats and oils. The analysis of fatty acid content is vital for evaluating the quality of Jatropha biodiesel [41]. Crude *J. curcas* oils contain fatty acids such as palmitic (C16:0), palmitoleic (C16:1), linoleic (C18:2), stearic (C18:1), and oleic (C18:1) acids. These unsaturated fatty acids, particularly oleic and linoleic acids, contribute to the strong low-temperature properties of *J. curcas* biodiesel [42,43].

### 3.2. Optimal Reaction Times for J. curcas Oil Biodiesel Manufacturing

The results highlight 6 h as a potentially optimal reaction time for biodiesel production based on methyl ester composition. Notably, the substantial elevations in concentrations of Palmitic Acid ME, Linoleic Acid ME, and Oleic Acid ME at the 6 h mark highlight their pivotal implications in biodiesel production. These findings suggest that the dynamics of triglyceride conversion during the reaction process and factors like catalyst efficiency and feedstock variations can influence methyl ester composition [44]. For instance, the elevated concentrations of Palmitic, Linoleic, and Oleic Acid ME may be attributed to the kinetics of triglyceride conversion. At the same time, the moderate increase in Stearic Acid ME hints at a slower conversion rate [45].

Furthermore, the heightened concentrations of Palmitic, Linoleic, and Oleic Acid ME at the 6 h mark align with the kinetics of triglyceride conversion highlighted in these studies [46]. The intricate chemical processes occurring during the reaction period likely contribute to the increased prevalence of these specific methyl esters [47]. Additionally, the moderate increase in Stearic Acid ME implies a slower conversion rate, which resonates with the findings of [47,48], suggesting that specific reaction conditions can influence the pace at which triglycerides are transformed into their respective methyl esters [49].

### 3.3. Optimal Reaction Temperature for Biodiesel Production from J. curcas Oil

The observed temperature-dependent variations in methyl ester concentrations underscore the importance of precise temperature control in achieving desired biodiesel characteristics. The optimum reaction temperature for biodiesel production lies in the vicinity of 110 °C, where the desired methyl ester composition, including the exclusive detection of Palmitoleic ME and elevated levels of Linoleic Acid ME and Oleic Acid ME,

is achieved. Our findings align with established research, confirming the significance of reaction temperature on methyl ester composition, particularly for Palmitic Acid ME, Linoleic Acid ME, and Oleic Acid ME [50]. The exclusive detection of Palmitoleic ME at 110 °C highlights the existence of specific temperature thresholds for certain methyl esters, as supported by [51]. Furthermore, the nuanced relationship between temperature and reaction rates, elucidated by [52], explains the observed fluctuations in Stearic Acid ME concentrations across temperatures [53]. Therefore, optimizing the reaction temperature is critical for tailoring the biodiesel product to meet specific quality and efficiency requirements [54]. The observed variations in methyl ester composition underscore temperature sensitivity in transesterification. Exclusive Palmitoleic ME presence at 110 °C aligns with our composition goals, while fluctuations in other esters result from intricate temperature-dependent kinetics and catalyst activity. Choosing 110 °C as the optimal calcination temperature balances catalyst activity and biodiesel yield, offering clarity on temperature intricacies in biodiesel production.

### 3.4. Ideal Methanol-to-Oil Ratio for J. curcas Oil Biodiesel Production

Notably, the 18:1 methanol-to-oil ratio is the most favorable condition, as it yields the desired composition with optimal concentrations of key methyl esters. This observation aligns with the works of Aboelazayem et al. [55], who similarly emphasized the importance of methanol-to-oil ratios in influencing biodiesel characteristics. The absence of Palmitoleic ME at both the lowest and highest ratios, with its optimal presence at 15:1 and 18:1, echoes the findings of Sakdasri et al. [56], highlighting the sensitivity of this methyl ester to specific reaction conditions. The increasing concentration trends noticed for Palmitic Acid ME, Linoleic Acid ME, and Oleic Acid ME at the 18:1 ratio are consistent with [57], emphasizing the critical role of methanol-to-oil ratios in determining methyl ester outcomes [58]. This correlation suggests that optimal esterification reactions leading to the formation of these key components occur more efficiently at the 18:1 ratio. The detectability of Crotonic Acid ME at the 18:1 ratio aligns with [59], who discussed the impact of specific methanol-to-oil ratios on the formation of certain methyl esters.

The observed trends underscore the complexity of biodiesel production, where even subtle changes in methanol-to-oil ratios can significantly influence methyl ester composition. The 18:1 ratio, identified as optimal in this study, balances methanol availability and efficient triglyceride conversion [60]. These insights pave the way for more precise and efficient biodiesel production processes in alignment with sustainability goals.

### 3.5. Optimal Calcined Catalyst Temperature for J. curcas Oil Biodiesel

Notably, biodiesel production's most favorable methyl ester composition was achieved at a calcined catalyst temperature of 900 °C, underscoring its significance as a crucial parameter for optimal outcomes. The detection of Palmitoleic ME at 800 °C and 900 °C suggests a temperature-sensitive response, as corroborated by the study of Satya et al. [61], who highlighted the role of catalyst temperature in influencing specific methyl esters. The decreasing trend in palmitic acid ME concentration with increasing calcined catalyst temperature underlines the sensitivity of this component to thermal conditions during transesterification [62]. Linoleic Acid ME and Oleic Acid ME exhibit marginal fluctuations across temperatures, reinforcing the findings of [63], who discussed the stability of certain methyl esters under varying reaction conditions. The stability in concentrations of Linoleic Acid ME and Oleic Acid ME suggests that the optimal temperature for their formation lies within the tested range. Stearic Acid ME's decreasing trend aligns with [64], who highlighted the impact of temperature on the saturated fatty acid composition in biodiesel. Furthermore, the detection of Crotonic Acid ME solely at 900 °C suggests the selective formation of certain methyl esters under specific temperature conditions [63,64].

### 3.6. Optimal Calcined Catalyst Concentration for Biodiesel Production from J. curcas Oil

Optimal outcomes are notably achieved at a 6 wt% catalyst concentration, as evidenced by the exclusive detection of Palmitoleic ME, peak concentrations of Palmitic Acid ME, Linoleic Acid ME, Oleic Acid ME, Stearic Acid ME, and the detectability of Crotonic Acid ME. Notably, the detection of Palmitoleic ME solely at a 6 wt% catalyst concentration aligns with the work of [65], emphasizing the catalytic influence on specific methyl ester formation. The concentration trends observed for Palmitic Acid ME, Linoleic Acid ME, Oleic Acid ME, and Stearic Acid ME at a 6 wt% catalyst concentration underscore the pivotal role of catalyst concentration in influencing these key components [66].

The optimal methyl ester composition achieved at a 6 wt% catalyst concentration suggests a delicate balance in the catalytic system [67]. This aligns with Likozar and Levec [68], who discussed the complex interplay between catalyst concentration and reaction kinetics during transesterification. Also, Csernica and Hsu [69] explored the selective formation of certain methyl esters under specific catalytic conditions. These findings collectively highlight the intricate relationship between catalyst concentration and methyl ester composition, emphasizing the need for precise control to tailor biodiesel characteristics. Rezaei et al. [28] investigated the use of waste mussel shells for biodiesel production. Optimal conditions were determined to be a calcination temperature of 1050 °C, 12 wt% catalyst concentration, and 24:1 methanol-to-oil ratio, resulting in maximum biodiesel purity and yield.

### 3.7. Catalyst Stability

The heterogeneous catalysts offer the advantage of reuse in multiple reaction cycles, making catalyst reusability a crucial factor in developing a novel transesterification catalyst. In the first five runs, biodiesel production yielded approximately 94.13%, 81.53%, 76.56%, and 66.41%, respectively. These results indicate a decrease in catalyst activity after each cycle, possibly due to (a) active species leaching into the biodiesel phase, (b) catalyst pore blockage, or (c) dissolution of some bulk calcium oxide in the methanolic solution [70,71]. In the sixth run, the crab shell catalyst produced biodiesel with an 83% yield from Karanja oil, suggesting that mussel shell is a more suitable, cost-effective option for biodiesel production. Additionally, several factors could contribute to this reduction in catalytic activity: (1) sintering of the catalyst at operational temperatures, which could reduce surface area and active sites essential for catalysis [72,73]; (2) accumulation of glycerol or other by-products on the catalyst surface, hindering reactant access to active sites [74,75]; (3) chemical transformation or phase change of the catalyst under reaction conditions [76]; and (4) potential leaching of calcium ions into the solution [77]. The leaching of $Ca(2+)$ from the catalyst surface into the reaction mixture may diminish the catalyst's available active sites, leading to a lower conversion rate over time [78,79].

Rezaei et al. [28] used waste mussel shells for biodiesel production and found that reusing the mussel shell catalyst negatively affected biodiesel yield after five cycles due to catalyst re-calcination. Also, Kouzu et al. [80] found that calcium diglyceroxides were generated after 15% of the reaction time, correlating with heightened glycerin production. The reaction halted upon the removal of the solid catalyst, indicating the transition to a stage where calcium diglyceroxides became the primary catalytic phase. Moreover, De Sousa et al. [81] found that calcium leaching was highest with commercial CaO (219 ppm), followed by eggshell-derived CaO (194 ppm), and lowest with carbothermal route-derived CaO (93 ppm). Higher leaching led to increased rate constants due to prompt reaction with glycerol, forming calcium diglyceroxide.

### 3.8. Biodiesel Characterization

Biodiesel must meet specific standards outlined in ASTM D-6751 and EN 14214 to be used in diesel engines. The Jatropha kernel was found to contain 50% oil based on test sample data, surpassing the oil content of soybean, linseed, and palm kernel oils, which were 18.35%, 33.33%, and 44.6%, respectively [42]. Due to its high oil content, *J. curcas* is an excellent non-edible vegetable oil feedstock for the oleochemical industry.

Viscosity is a critical property of biodiesel that affects engine startup, spray quality, and particle size. *J. curcas* oil exhibited a high kinematic viscosity of 40.02 mm$^2$/s at 40 °C due to its large molecular mass and high FFA content [82]. However, during the transesterification process, viscosity decreased from 40.02 mm$^2$/s to 4.89 mm$^2$/s, primarily due to glycerol removal, which has a high viscosity [83]. This reduced viscosity, falling within the required range, is suitable for combustion, aligning with biodiesel standards. Similar kinematic viscosities have been observed in prior studies on biodiesel produced from *J. curcas* oil [84].

The density of biodiesel is crucial in determining the completeness of the transesterification reaction. As density increases, viscosity also rises, potentially affecting fuel injection system performance due to the larger fuel volume delivered [85]. To meet regulations such as ASTM D 6751 and EN 14214, any biofuel used in burners must have a density between 820 kg/m$^3$ and 845 kg/m$^3$. The tested samples fell within the specified regulatory limits at 0.856 g/cm$^3$, making them suitable for diesel engine injection and combustion. This biodiesel density aligns with values reported in previous studies [86]. The flashpoint of a fuel is the temperature at which a fuel ignites when exposed to a flame. Biodiesel is safer than regular fuel due to its higher flashpoint [87]. Biodiesel standards specify flashpoint temperatures between 100 °C and 170 °C, and the tested sample had a flashpoint of 110 °C, meeting ASTM D6751 and EN 14214 standards. Flashpoint indicates fuel safety during transportation, storage, and handling [88].

The pour point is the temperature at which fuel forms a gel, losing its ability to flow. Biodiesel's pour point is (–3 °C), aligning with the ASTM biodiesel standard. However, due to its properties, biodiesel is less suitable for use at low temperatures. Cloud point, defined as the temperature at which wax particles first appear in cooling fuel, is crucial for low-temperature operability. This investigation obtained a 0 °C cloud point for (JCME), indicating stability and minimal wax formation at low temperatures, consistent with previous studies [89].

The cetane number directly affects combustion quality and engine performance in diesel engines. A minimum cetane number of 47 is required, and the tested diesel sample had a cetane number of 50.5, falling within acceptable limits. Earlier studies have reported cetane number values within the defined reference standard range for biodiesel [87]. JCME is considered suitable for diesel engine use without affecting combustion or performance. JCME possesses a calorific value of 38.140 MJ/kg, similar to diesel. This high calorific value indicates its energy content, making JCME a suitable renewable and alternative fuel [90].

### 3.9. Comparison of Homogeneous and Heterogeneous Catalysts

Using mussel waste as a heterogeneous catalyst in Jatropha oil transesterification shows its potential as a renewable catalyst. Comparative yields with CaO waste shell and NaOH catalyst were similar, suggesting that environmentally sourced heterogeneous catalysts can replace homogeneous catalysts while maintaining productivity. The results showed that CaO predominantly produces Palmitic Acid ME, Linoleic Acid ME, Stearic Acid ME, Crotonic Acid ME, Oleic Acid ME, and others. Conversely, NaOH leads to higher concentrations of Oleic Acid ME, Palmitoleic ME, Acid ME, Arachidic Acid ME, and Behenic Acid ME. This is consistent with the findings of [91] on the impact of heterogeneous catalysts on biodiesel composition.

On the other hand, NaOH as a homogeneous catalyst leads to a distinct profile, with higher concentrations of Oleic Acid ME, Palmitoleic ME, cid ME, Arachidic Acid ME, and Behenic Acid ME. This aligns with the work of Mendow et al. [92], emphasizing the unique impact of alkaline catalysts on fatty acid composition in transesterification. The significant increase in Oleic Acid ME with NaOH may be attributed to its specific catalytic properties, as highlighted by Kumar et al. [93] in their investigation of alkaline-catalyzed transesterification reactions [93,94]. The diversity in the composition of FAMEs observed in Table 8 arises from the intricate and varied fatty acid profile in *Jatropha curcas* oil. This plant oil contains various fatty acids, each contributing differently to the overall FAME composition during transesterification. These findings underscore the importance of catalyst selection

in tailoring biodiesel composition, offering valuable insights for optimizing production processes and meeting specific biodiesel quality standards.

After conducting a comprehensive analysis of reaction optimization, catalyst stability, and comparative analysis with NaOH catalysts, the results presented in this study demonstrate significant advancements in biodiesel production using mussel shell-derived catalysts. Our findings build upon the groundwork laid by our previous research [32], in which we introduced the innovative use of mussel shell-derived (CaO) as a catalyst for sustainable biodiesel production from *J. curcas* oil. While our previous work focused on catalyst characterization and optimizing reaction conditions to achieve high FAME yields, the current study delves deeper into the elemental composition of the catalyst, methyl ester composition analysis, and comparison with homogeneous catalysts, thereby contributing to a more comprehensive understanding of sustainable biodiesel synthesis.

Liu et al. [95] found optimal conditions for soybean oil transesterification using CaO as a catalyst, yielding over 95% biodiesel within 3 h and maintaining consistent activity over 20 cycles. An eggshell-derived CaO catalyst for *Chlorella vulgaris* biodiesel showed promising results, yielding 92.03% biodiesel under the optimal conditions of 70 °C, 10:1 methanol-to-dry biomass ratio, 1.39% catalyst loading, 3 h reaction time, and 140 rpm stirring [96]. Jamil et al. [97] reported CaO as the most effective catalyst, yielding 94.27% biodiesel. Das et al. [98] achieved a high yield of 98.3% using cobalt-doped CaO but experienced a 12% yield loss after three cycles. Todarovic et al. [99] obtained a 99.9% biodiesel yield within 1.5 h using a CaO catalyst with crude biodiesel as a co-solvent. Comparing our results with these studies underscores the efficacy and potential of mussel shell-derived CaO catalysts in biodiesel production.

Overall, the current investigation expands upon the findings of our previous work [32] by delving into the meticulous evaluation of reaction parameters, such as reaction time, temperature, and methanol-to-oil ratio. These insights not only build upon our previous findings but also introduce new dimensions to the optimization of biodiesel production. The ability to reuse the mussel shell-derived CaO catalyst up to five times with minimal loss in efficiency represents a significant advancement in sustainable biodiesel production, offering practical implications for industrial applications and environmental conservation. Furthermore, this research illuminates the superior sustainability and potential cost benefits of CaO catalysts over their NaOH counterparts. By systematically assessing the stability and reusability of the CaO catalyst, we address a critical gap in knowledge regarding the long-term feasibility of biodiesel production processes, thereby underscoring the importance of continuing to explore and optimize renewable energy resources.

However, the observed decline in FAME yield over five cycles from 99.2% to 66.41% raises critical questions about the sustainability and long-term efficacy of mussel shell-derived (CaO) catalysts in biodiesel production. To enhance catalyst reusability, future work should focus on: analyzing recovered catalytic solutions via ICP to quantify Ca(2+) leaching; exploring catalyst modification/coating techniques to mitigate sintering and chemical degradation; investigating post-reaction treatments like calcination or acid washing to regenerate active sites and remove by-products; and employing advanced characterization techniques such as TEM, XPS, and BET analysis to understand catalyst changes. These efforts aim to elucidate activity decrease mechanisms and develop effective regeneration strategies for sustainable biodiesel production. Through such analysis, our study contributes critical insights into the lifecycle management of mussel shell-derived catalysts, marking a significant advancement in the sustainable production of biodiesel.

## 4. Materials and Methods

### 4.1. Mussel Shell-Derived CaO Catalyst Preparation and Oil Extraction

*Jatropha curcas* seeds, sourced from Ahmad Qashash farm in Al-Baha, KSA, underwent a rigorous sorting process to eliminate damaged seeds. The cleaned and de-shelled seeds were then subjected to mechanical press oil extraction, yielding approximately 50% oil. Simultaneously, mussel shells obtained from a local community market in Jeddah, KSA,

underwent a thorough cleaning, drying, crushing, and sieving process to attain a particle size of <250 μm. The prepared mussel shell catalysts underwent calcination at varying temperatures (800, 900, 1000, and 1100 °C) for 4 h, transforming the $CaCO_3$ content into catalytically active CaO. These steps are adapted from established methodologies [70,83]. Biodiesel production ensued through a transesterification process, where the optimized mussel shell-derived CaO catalyst played a pivotal role. *J. curcas* oil, obtained through seed oil extraction, served as the feedstock for transesterification. The experimental conditions included a methanol-to-oil ratio of 18:1, a reaction temperature of 110 °C, and a reaction time of 6 h, as identified through the optimization process.

### 4.2. Evaluation of Reaction Parameters

The effectiveness of mussel shell-derived CaO catalysts in promoting *J. curcas* oil transesterification was assessed by varying reaction parameters. Optimal methyl ester composition in biodiesel production was determined based on factors such as reaction time, temperature, methanol-to-oil ratio, calcined catalyst temperature, and catalyst concentration, aiming to optimize the transesterification process.

The reaction occurred in a 3-neck flask (250 mL) placed in a paraffin oil bath with magnetic stirring and a water-cooled condenser. Various conditions were tested, including different 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, solid catalyst separation was achieved through centrifugation for 20 min at 4000 rpm, isolating methyl ester, glycerol, and the catalyst. Vacuum evaporation removed excess methanol, and the resulting liquid was collected in a glass-separating funnel (adapted from [100,101]).

### 4.3. Determination of Fatty Acid Compositions

The methyl esters of *J. curcas* oil were characterized using GC–MS to determine their composition. Each sample consisted of 0.02 mL of oil and 1.98 mL of hexane. GC analysis was performed with an Rtx5MS column, and peak identification was based on standards (operating conditions are detailed in Table 1). FAME concentrations were calculated in weight percentage (wt%) to ensure uniform and accurate representation. The conversion was determined based on the molar ratio of reacted triglycerides to produced FAMEs. The composition of *J. curcas* oil was determined through Gas Chromatography–Mass Spectrometry (GC–MS). The conversion percentage was calculated using the formula:

$$\text{Conversion (\%)} = [(\text{Moles of FAMEs produced})/(\text{Moles of triglycerides in the oil})] \times 100$$

### 4.4. Catalyst Stability

To evaluate the reusability of the mussel shell CaO catalyst, we recovered it by centrifuging transesterification reactant samples for 20 min at 4000 rpm. The precipitated CaO was washed with n–hexane and dried in a forced-air convection oven, allowing for its reuse in subsequent transesterification reactions [102,103].

### 4.5. Comparison of Homogeneous and Heterogeneous Catalysts

We conducted a transesterification reaction of Jatropha oil into biodiesel using NaOH as a homogeneous catalyst under specific conditions. We then compared the results with the optimal conditions established in this study using CaO derived from mussel shells as a heterogeneous catalyst.

## 5. Conclusions

This research contributes valuable insights into sustainable biodiesel production from *J. curcas* oil, emphasizing the importance of catalyst selection and optimization. The optimal conditions, including calcination temperature, reaction time, temperature, and methanol-to-oil ratio, provide a roadmap for enhancing biodiesel production efficiency. The study's

innovative use of mussel shell-derived CaO as a renewable catalyst showcases its potential for replacing homogeneous catalysts with comparable yields. The demonstrated catalyst stability further supports mussel shell-derived catalysts' economic viability. Additionally, the thorough characterization of JCME biodiesel aligns with industry standards, with variations attributed to the unique properties of *J. curcas* Methyl Ester. Overall, this study advances the understanding of sustainable biodiesel production, offering practical implications for optimizing processes and adopting eco-friendly catalysts to pursue a greener energy future.

**Supplementary Materials:** The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/inorganics12040109/s1, Figure S1. GC–MS spectra of JCME at different reaction times. Figure S2. The GC–MS spectra of JCME in different reaction temperatures. Figure S3. The GC–MS spectra of JCME in different methanol to oil ratio. Figure S4. The GC–MS spectra of JCME at different calcined catalyst temperature. Figure S5. The GC–MS spectra of JCME in different catalyst concentration. Figure S6. The GC–MS spectra of JCME use catalyst (a) CaO from mussel shell (b) NaOH.

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