

Article

Valorization of Banana Peel Waste as a Catalyst for Biodiesel Production: Reaction Kinetics Study

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Abstract

In this work, the use of a banana peel-derived basic heterogeneous catalyst is investigated for producing biodiesel from sunflower oil. The catalyst is produced by treating dried banana peels at 700 °C under normal atmosphere for 4 h. The reaction parameters were optimized by varying the methanol-to-oil molar ratio (MOMR) and the catalyst-to-oil ratio (CTOR). The highest conversion of sunflower oil to biodiesel (99.4%) was attained after a reaction time of 90 min using an MOMR of 9:1, a catalyst loading of 4 wt%, and a stirring rate of 300 rpm. The catalyst demonstrated good stability, with a yield of 99.4% ($\pm 0.28\%$) obtained over three consecutive runs. No active phase loss was observed during the consecutive reactions, as confirmed by the leaching test. The physical and chemical properties of the formed biodiesel were in good accordance with the American Society for Testing and Materials (ASTM) standards. The kinetic study allowed for the determination of the activation energy ($E_a = 58.1$ kJ/mol) and pre-exponential factor ($A = 7.4 \times 10^7$ min⁻¹) of the catalyzed reaction. Additionally, the valorization of waste cooking oil (WCO) (a second-generation feedstock oil) using the waste banana peel-derived catalyst under optimum reaction conditions achieved a yield of 99.1% ($\pm 0.14\%$).

Keywords: biodiesel; banana peel; transesterification; reusability; kinetics

1. Introduction

Growing populations, urbanization, and industrial evolution have rapidly increased the demand for energy [1]. Fossil fuels constitute the main energy source and currently provide more than 80% of the global energy demand. However, given their finite nature, a shortage in natural gas, petroleum and coal is expected because of their extensive consumption [2,3]. Additionally, energy production from burning fuels releases significant amounts of greenhouse gases (GHGs) that contribute to the increase in global average temperatures and toxic emissions, including unburned hydrocarbons, carbon monoxide, sulfur oxides and nitrogen oxides, among others [3]. Accordingly, concerns about fossil fuel exhaustion and environment deterioration have motivated international communities and researchers to find more sustainable and environmentally benign means to power contemporary economies [4]. Potential substitutes should be easy to attain, harmless to the environment, and economically sustainable, and must be compatible with existing engines and vehicles [1].

Biodiesel, in particular, has garnered important attention and has emerged as a noteworthy player in the renewable energy sector owing to its numerous benefits over



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petrodiesel [5]. It is a mixture of fatty acid alkyl esters and is the product of three consecutive reversible transesterification reactions. In these reactions, a catalyst is used to promote the reaction of triglycerides (TGs) with alcohol, usually methanol, to yield fatty acid methyl esters (FAMES) and glycerol [6]. Edible oil feedstock, such as soybean, sunflower, or palm oil, is typically used; however, non-edible oils such as jatropha oil, animal fat, and blends of different oil types can also be used as a source of TGs [2,3,7,8]. In contrast to petroleum diesel, which is rich in alkanes and aromatic hydrocarbons, biodiesel is oxygenated, sulfur free, and non-toxic [9]. Biodiesel can be directly utilized in existing diesel engines without necessitating any adjustments, offers greater qualities including improved lubricity, a greater cetane number, and improved biodegradability, and has little effects on the environment, all while satisfying energy demands. Biodiesel also demonstrates exceptional safety when it comes to its transportation and storage compared to petroleum diesel [5].

The determining parameter in efficient biodiesel production is the catalyst selection. Conventionally, homogeneous catalysts are used in biodiesel synthesis since they are relatively cheap and exhibit high catalytic activities with excellent biodiesel yields. These consist of acidic and basic homogeneous catalysts including sulfuric acid (H_2SO_4), hydrochloric acid (HCl), sodium hydroxide (NaOH) and potassium hydroxide (KOH) [10,11]. However, homogeneous alkaline catalyst usage may lead to soap formation, while acid catalysts are hindered by many complexities such as slowed reaction rates, high temperature and pressure requirements, complications in product separation, and corrosive natures [12,13]. All these complexities are captured in the overall costs of biodiesel production and limit the usage of homogeneous catalysts specifically for commercial biodiesel production [4]. The potential of heterogeneous catalysis in addressing many of these obstacles has made it a focal point in biodiesel synthesis [13]. Heterogeneous base catalysts are capable of producing top-quality biodiesel under mild conditions, are easy to recycle, and prevent soap formation [13,14]. In general, heterogeneous catalysts exhibit good recyclability and reusability potentials, conserving their morphology, active site density, and catalytic activity [4]. Recent advancements have focused on preparing catalysts from easily sourced materials with reduced impact on the environment, greater cost-effectiveness and simpler recovery [15–18]. Several studies have highlighted the application of industrial wastes for catalyzing the synthesis of biodiesel, contributing to a cost-effective and sustainable approach for biofuel production. Due to its rich composition of strong basic sites, red mud has been widely investigated, with high reported biodiesel yields of 99.6%, representing its significant catalytic potential [19]. Similarly, mining industry slags, characterized by their high MgO, CaO, and other basic oxide content, showed notable efficiency in turning both edible and waste cooking oil into biodiesel, reaching yields up to 95% [19,20]. Bio-waste-derived heterogeneous catalysts have also gained a lot of attention due to their low cost, widespread availability, ease of handling, and minimal environmental impact [13,21]. The abundant supply of agricultural residues serves as a perfect source for bio-based catalyst development, as they have a porous structure and show an extended specific surface area, exhibiting robust mechanical properties and improved thermal stability [11]. Numerous works have extensively approached the development of bio-waste-derived catalysts from a wide variety of agricultural wastes such as citrus peels [22], onion peels [23], potato peels [24], corncobs [25], orange peels [26], and cocoa pod husks [27]. All of these works showed biodiesel yields above 95%, highlighting the capacity of agricultural wastes to provide a source of green catalysts for an efficient and environmentally sustainable biodiesel production process. One of the most promising sources of agricultural wastes for catalyst synthesis is banana peels. It is well established that such agricultural wastes are rich in potassium, which constitutes a good source for deriving basic catalysts for biodiesel synthesis [28]. Elemental analysis of waste banana peels shows a high content of potassium

and carbon. Waste banana peel ash (WBPA) is found to be composed of metal oxides (K_2O , CaO) and carbonates (K_2CO_3 , $CaCO_3$), giving it a basic character, an essential property for effective catalytic activity in biodiesel production [29–31]. Banana peel-derived catalysts are therefore valued for their high-mineral matrix, rich in metal oxides and carbonates, as well as trace minerals such as Fe, Si, and P. In addition to a carbon content ranging from 40 to 50% [30,32], carbons are present as different and distinct chemical species and functional groups such as surface functional groups (hydroxyl, carbonyl, and carboxyl carbons), inorganic carbon species (K_2CO_3 and $CaCO_3$), and structural carbon species, creating the backbone of the catalyst [33]. The availability, low cost, ease of processing, non-toxicity, and environmentally benign nature of banana peels make them a compelling alternative for sustainable resource management [34]. Transforming this abundant waste into a heterogeneous catalyst decreases environmental pollution, contributes to the flourishing of green innovations, and encourages a circular economy [34]. Few research papers have investigated the usage of WBPA catalysts for biodiesel synthesis via transesterification.

In this work, a banana peel-derived heterogeneous catalyst was employed to catalyze biodiesel production from sunflower oil. The effects of the methanol-to-oil molar ratio (MOMR) and the catalyst-to-oil ratio (CTOR) were evaluated, and the reusability of the catalyst was assessed. Additionally, a kinetic study of the transesterification process was conducted to determine the pre-exponential factor (A) and the reaction activation energy (E_a).

2. Materials and Methods

Waste banana peels were collected and used for catalyst preparation. Refined sunflower oil was purchased from a store located in Lebanon. The following reagents were all purchased from Sigma-Aldrich (St. Louis, MO, USA): methanol, n-hexane, and methyl heptadecanoate. These reagents were of high analytical purity and were utilized directly without any pretreatment or purification steps.

The properties of the sunflower oil were determined using ASTM and AOCS standard methods: kinematic viscosity (ASTM D445), density (ASTM D4052), saponification value (AOCS Cd 3-25), FFA content (AOCS Ca 5a-40), and water content (AOCS Ca 2c-25). The properties of the waste cooking oil were also determined: density at 25 °C, water content and FFA content. Their corresponding methods are ASTM D4052, AOCS Ca 2c-25 and AOCS Ca 5a-40, respectively.

The waste banana peels were dried in the sun for 4–5 days. They were then pulverized using a blender and calcined for four hours at 700 °C. The collected material was grinded into a finely milled powder and placed in an airtight container for storage until further use.

Simultaneous thermogravimetric and differential scanning calorimetry analyses (TG-DSC) of the uncalcined and calcined banana peels (10 mg) were conducted using Labsys EVO equipment (Setaram Instrumentation, Caluire, France). The samples were heated from ambient temperature to 800 °C at a rate of 5 °C min^{−1} under a 100 mL min^{−1} flow of air (99.99% purity).

CO₂ temperature-programmed desorption (CO₂-TPD) experiments were performed on an AutoChem II 2920 apparatus (Micromeritics Instrument Corporation, Norcross, GA, USA). A ~50 mg sample was first heated in helium (30 mL min^{−1}/600 °C/30 min), then cooled to ambient temperature. The pretreated catalyst was subjected to CO₂ flow for an hour and then purged with helium for 30 min (30 mL min^{−1}). The catalyst was then heated up to 700 °C (10 °C min^{−1}) in order to release the adsorbed CO₂.

All reactions for biodiesel production were performed in a three-neck round bottom flask (250 mL) coupled with a reflux condenser. Continuous stirring ensured a homogeneous mixing of all three reaction components (methanol, oil, and catalyst). Sunflower

oil was first preheated to 65 °C before adding the methanol and the catalyst at a constant mixing rate of 300 rpm. The CTOR was first set at 2 wt% and the reaction temperature at 65 °C. For optimization of the reaction, the MOMR was varied from 6:1 to 12:1 and the CTOR was raised from 2 wt% to 4 wt%. Every reaction was stopped after 90 min, and the excess methanol was evaporated at 70 °C. The remaining mixture was centrifuged at 3000 rpm (5 min) to efficiently separate the obtained biodiesel from the glycerol. The experimental protocol is summarized in Figure 1.

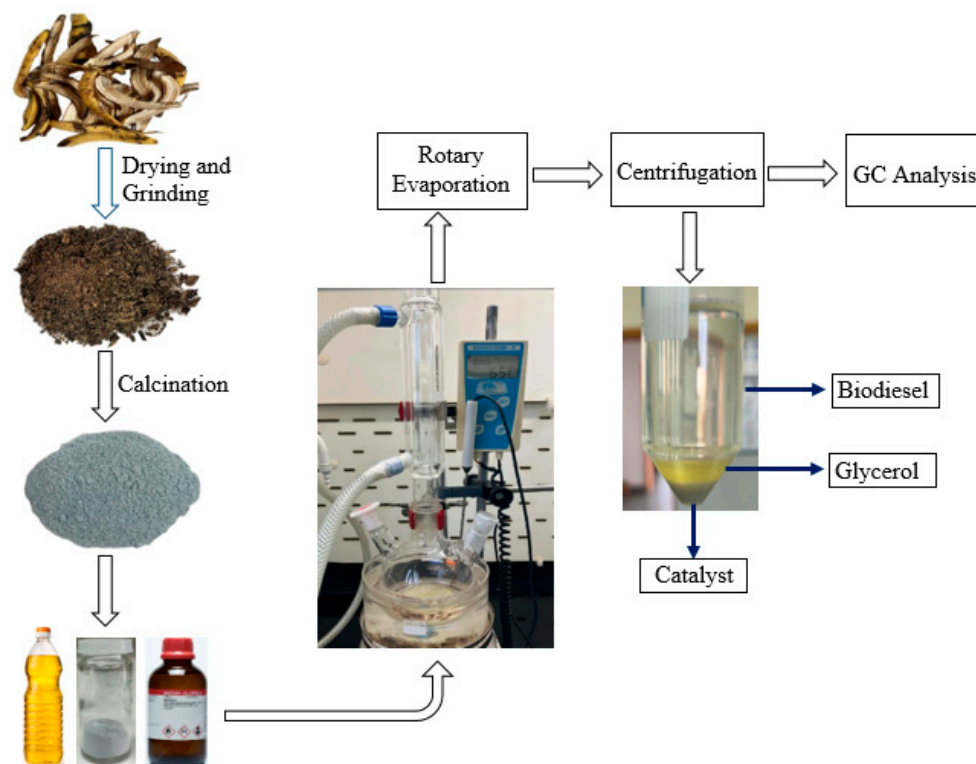


Figure 1. Summary of experimental procedure.

The conversion yield was determined by injecting 50 mg from the biodiesel phase premixed with 1 mL of an internal standard solution (C17, methyl heptadecanoate in hexane, 10 mg mL^{−1}) into an Agilent 7890A series gas chromatography (Agilent Technologies, Santa Clara, CA, USA) (GC: flame ionization detector (FID), HP-5 column (length: 30 m, stationary phase thickness: 0.25 µm, internal diameter: 0.32 mm)).

The catalytic activity in the transesterification reaction was indicated by the resulting FAME yield or content. The FAME yield or content was computed using Equation (1) [35,36].

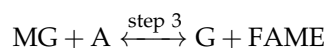
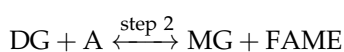
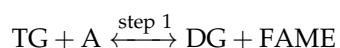
$$\text{FAME content (\%)} = \frac{\sum A}{A_{\text{IS}}} \times \frac{C_{\text{IS}} \times V_{\text{IS}}}{W} \times 100 \quad (1)$$

where $\sum A$ is the sum of the methyl ester peak areas, A_{IS} is the internal standard peak area, C_{IS} is the internal standard concentration (mg mL^{−1}), V_{IS} is the internal standard volume (mL), and W is the biodiesel sample weight (mg).

In the determination of the FAME yields, the total error arising from both analytical procedures and/or experimental variability was found to be less than 5%.

The synthesized biodiesel underwent analysis for its chemical and physical properties in line with ASTM specifications: density (at 15 °C), flash point, kinematic viscosity (at 40 °C) and copper strip corrosion. Their corresponding methods were ASTM D4052, ASTM D93, ASTM D445 and ASTM D130, respectively.

For the kinetics study, the reaction was performed at three separate temperatures spanning from 55 to 65 °C, with an increment of 5 °C. A 1 mL aliquot was sampled at 30 min intervals and placed under the laboratory hood to remove any excess methanol. In order to determine the FAME yield at distinct times and temperatures, vials for gas chromatography (GC) were properly prepared. Performing a kinetic study of the biodiesel production reaction using heterogeneous catalysts offers an understanding of the performance of the biodiesel production process via transesterification, while providing an estimation of the rate of the reaction and the calculation of the reaction speed parameters [37]. Multiple works that focused on developing kinetic modeling for biodiesel production reactions have demonstrated that the reaction follows a pseudo-first-order kinetic approach. This comes after performing a series of assumptions [37–39]. First, the reaction occurs over a set of three steps, described as follows [39]:



where TG, DG, MG, A, G, and FAME represent triglycerides, diglycerides, monoglycerides, alcohol, glycerol, and biodiesel, respectively.

The transesterification reaction takes place in excess alcohol, and it is also assumed that the reverse equations are negligible [39]. Additionally, the reaction between triglycerides and alcohol is slower than the reaction of diglycerides and monoglycerides with alcohol; therefore, the reaction between triglycerides and alcohol is taken as the rate-determining step [37,39]. According to these assumptions, the reaction rate can be written as follows [37]:

$$\frac{-d[\text{TG}]}{dt} = k'[\text{TG}][\text{A}]^3 = k[\text{TG}] \quad (2)$$

where $k = k'[\text{A}]^3$ and $(-d[\text{TG}])/dt$ represent the consumption of triglycerides per unit of time (t), k represents the rate constant, $[\text{TG}]$ denotes the triglyceride concentration, and $[\text{A}]$ corresponds to the concentration of alcohol.

Integrating Equation (2), we obtain

$$\frac{[\text{TG}]}{[\text{TG}_0]} = e^{-kt} \quad (3)$$

where $[\text{TG}_0]$ represents the initial concentration of triglycerides.

Rearranging Equation (3), we get Equation (4):

$$\ln [\text{TG}_0] - \ln [\text{TG}] = kt \quad (4)$$

The oil conversion (X) into biodiesel at any time is given in Equation (5) [39]:

$$X = 1 - \frac{[\text{TG}]}{[\text{TG}_0]} \quad (5)$$

where X is the biodiesel yield.

By substitution into Equation (3), we obtain

$$1 - X = e^{-kt} \quad (6)$$

By rearranging Equation (6), we obtain Equation (7):

$$-\ln(1 - X) = kt \quad (7)$$

A plot of $-\ln(1 - X)$ versus the reaction time (t) allows us to determine the reaction rate constant (k) values at various temperatures. The values of the rate constant (k) obtained can be used to calculate the activation energy of the reaction (Ea) as well as the pre-exponential factor (A) using the Arrhenius Equation (8) [35,38]:

$$\ln(k) = \ln(A) - \frac{1}{T} \frac{E_a}{R} \quad (8)$$

The Arrhenius pre-exponential factor and the activation energy can be correspondingly determined from the y-intercept and the slope of the equation [35].

3. Results and Discussion

3.1. Characterization of Edible Sunflower Oil

The properties of the utilized sunflower oil are displayed in Table 1. The values follow the AOCS and ASTM requirements and match the desired specifications for biodiesel production. Similar results were reported in other works [35,40].

Table 1. Physico-chemical properties of refined sunflower oil.

Property	Value	Test Method	Requirements
Density at 25 °C (Kg/m ³)	920	ASTM D4052	918.00–921.00
Kinematic viscosity at 40 °C (mm ² /s)	41.34	ASTM D445	-
FFA content (wt%)	0.06	AOCS Ca-5a-40	≤0.5
Saponification value (mg KOH/g)	193	AOCS Cd-3-25	188–194
Water content (wt%)	0	AOCS Ca-2c-25	≤0.2

3.2. Catalyst Characterization

The thermal behavior of both dried banana peels and calcined banana peels was explored under an oxidative atmosphere using TG-DSC analyses. Figure 2a shows the TG curves obtained for both samples, whereas Figure 2b shows their corresponding DSC curves. The TG curve of dried banana peels shows significant weight losses at different temperature intervals.

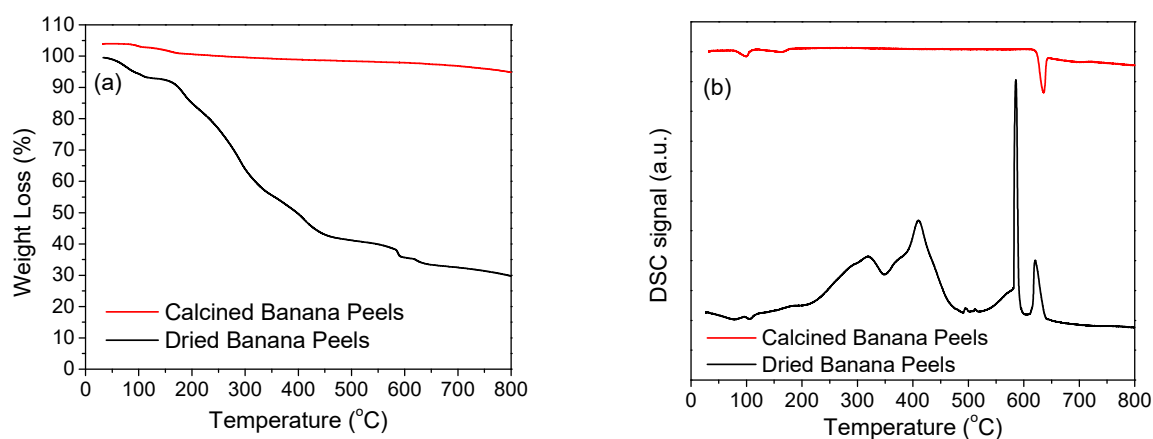


Figure 2. (a) TG and (b) DSC curves of dried banana peels and calcined banana peels.

Initially, a weight loss due to moisture removal from the dried peels can be seen at 100 °C. Although the banana peels were dried at a high temperature, complete removal of moisture was not possible due to their hygroscopic nature [41]. This explains the weight reduction caused by the expulsion of adsorbed moisture at 100 °C seen in the TG curve of the calcined peels. The second phase of weight loss takes place at temperatures between 150 and 400 °C. In this phase, thermal decomposition of the organic constituents of banana peels, namely cellulose and hemicellulose, caused a decrease in the sample weight, leading to char formation [41]. The third phase occurs between 500 and 650 °C. At such temperatures, carbonaceous compound decomposition and char and metal carbonate oxidation/decomposition occur, releasing CO and CO₂ and leaving behind metal oxide compounds [13,31,42]. Banana peel-derived catalysts can be calcined at high temperatures, while alkaline carbonates such as K₂CO₃ remain present given their high thermal stability [43]. No further weight loss was observed at higher temperatures, leading to the choice of a temperature of 700 °C for banana peel calcination. The DSC curve of the dried banana peels showed several endothermic peaks, all in good agreement with the weight losses observed in the corresponding TG curve. The endothermic peak occurring at around 100 °C is caused by the evaporation of moisture. The endothermic process taking place between 150 and 400 °C is related to the decomposition of cellulose and hemicellulose. At a temperature higher than 500 °C, heat is absorbed and associated with the degradation or oxidation of carbonaceous components [41]. Apart from the desorption of re-adsorbed moisture around 100 °C, the TG curve of calcined banana peels shows no significant weight loss, confirming its thermal stability in the considered temperature range. An exothermic peak observed at around 650 °C in the calcined banana peel DSC curve could be associated with the crystallization of inorganic mineral phases present in the calcined sample [30].

The fundamental properties of the calcined banana peels were studied using CO₂-TPD. The results are shown in Figure 3. Three main desorption peaks (I, II and III) were detected at 100, 700 and 750 °C, caused by the presence of alkaline sites of composite basic metal oxides. The temperature of the CO₂ desorption peak correlates with the strength of the base site. Stronger basic sites are linked to higher CO₂ desorption temperatures. At higher temperatures, the observed CO₂ desorption peaks could be due to the decomposition of surface carbonate species with highly alkaline sites [44].

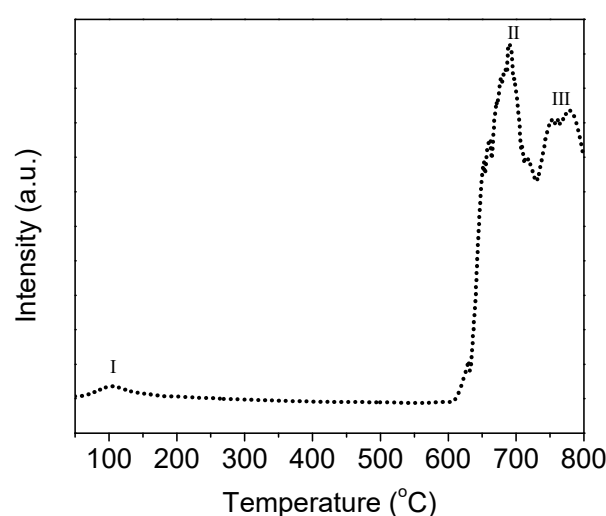


Figure 3. CO₂-TPD profile of calcined banana peels.

3.3. Catalytic Activity of Calcined Banana Peels Catalyst: Process Optimization

To investigate the catalytic effectiveness of calcined banana peel catalysts for the conversion of sunflower oil into biodiesel, the effect of different reaction parameters such

as the methanol-to-oil molar ratio (MOMR) and the catalyst-to-oil molar ratio (CTOR) on the transesterification reaction was studied.

3.3.1. Effect of the Methanol-to-Oil Molar Ratio (MOMR) on FAME Yield

Calcined banana peels (700 °C/4 h) were used to catalyze the transesterification reaction. The reaction was performed with various MOMR values (6:1, 9:1, and 12:1), while other parameters were kept constant (CTOR of 2 wt%, reaction temperature of 65 °C, stirring rate set at 300 rpm, and reaction time of 90 min).

The FAME yield increased from 12.0% ($\pm 0.28\%$) to 27.0% ($\pm 0.35\%$) when the MOMR was increased from 6:1 to 9:1 (Figure 4). This is due to excess methanol creating a large number of methoxy ions on the surface of the catalyst, speeding up the reaction [45]. In addition, excess methanol removes the product molecules present on the surface of the catalyst, regenerating the active catalytic sites [45]. A further increase in the MOMR to 12:1 led to a decrease in the biodiesel yield to 16.0% ($\pm 0.63\%$). Poor glycerol separation from the produced biodiesel at a high MOMR hinders the reaction, thereby decreasing biodiesel yields. In addition, the highly dispersed catalyst causes a reduced interaction with the reactants [23,45,46]. Hence, an MOMR of 9:1 was considered for performing additional experiments. These findings align with observations reported from other researchers, who noted a progressive bell-shaped curve for the biodiesel yield when optimizing the MOMR. Excessive use of methanol decreased the biodiesel yield by diluting the system and increasing the glycerol solubility, interfering with the biodiesel conversion, while insufficient alcohol levels had negative effects on the biodiesel yield [47].

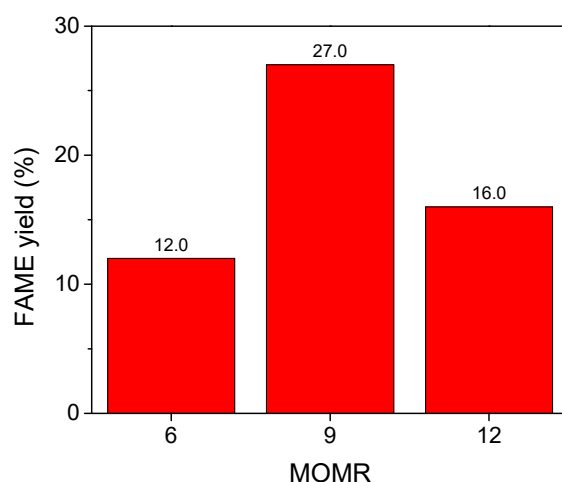


Figure 4. Effect of MOMR on FAME yield (reaction temperature = 65 °C, CTOR = 2 wt%, reaction time = 90 min, stirring rate = 300 rpm and calcination temperature = 700 °C).

3.3.2. Effect of Catalyst-to-Oil Molar Ratio (CTOR) on FAME Yield

The effect of various catalyst loadings (2, 3, and 4 wt%) on the biodiesel yield was investigated. The following parameters were kept unchanged throughout all experiments: MOMR (9:1), reaction temperature (65 °C), reaction time (90 min), and stirring rate (300 rpm). Figure 5 shows that a FAME yield of 27% ($\pm 0.35\%$) was recorded for a 2 wt% catalyst loading. This yield improved after increasing the catalyst loading, reaching a maximum of 99.4% ($\pm 0.28\%$) for a CTOR = 4 wt%. The better availability of basic active sites yielded more biodiesel from the reaction [25,46]. In addition, reduced methanol mass transfer limitations and an increased catalyst amount are additional reasons for the increased biodiesel production yield [48]. Hence, a 4 wt% catalyst loading was chosen for the remaining transesterification reaction.

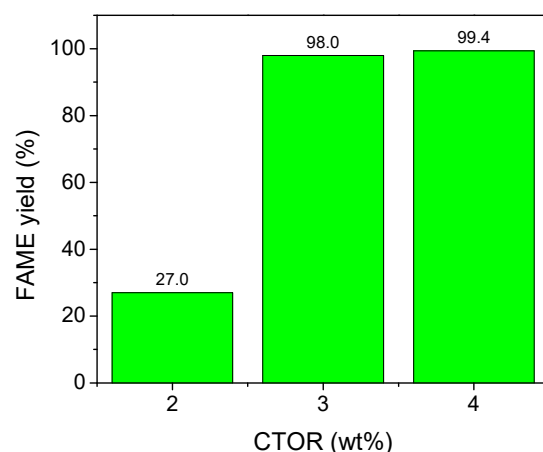


Figure 5. Effect of CTOR on FAME yield (reaction temperature = 65 °C, MOMR = 9:1, reaction time = 90 min, stirring rate = 300 rpm and calcination temperature = 700 °C).

3.3.3. Mechanism and Comparison to Literature

The catalytic pathway for the transesterification reaction using an alkaline solid catalyst synthesized from calcined banana peels is documented in the literature and is shown in Figure 6. The fatty acid carbon chains are denoted as R1, R2, and R3, while M-O is the surface basic site of the catalyst [30,49,50]. The availability of basic sites on the catalyst surface was verified through the CO₂-TPD analysis shown in Figure 3. The mechanism involves a cycle of four consecutive steps that result in the regeneration of the alkaline active sites of the catalyst at the end of the last step. In the first step, the adsorption of alcohol molecules on the basic sites of the catalyst results in the creation of an active methoxide ion. The second step consists of the methoxide ion attacking the carbonyl carbon of the triglyceride molecule, giving rise to a tetrahedral intermediate. In the third step, the alkoxide group in the triglyceride molecule is kicked off, liberating a FAME molecule and forming a diglyceride anion. Lastly, the diglyceride anion picks up the H⁺ from the basic site, regenerating the active sites of the catalyst that will be ready for another catalytic cycle [49].

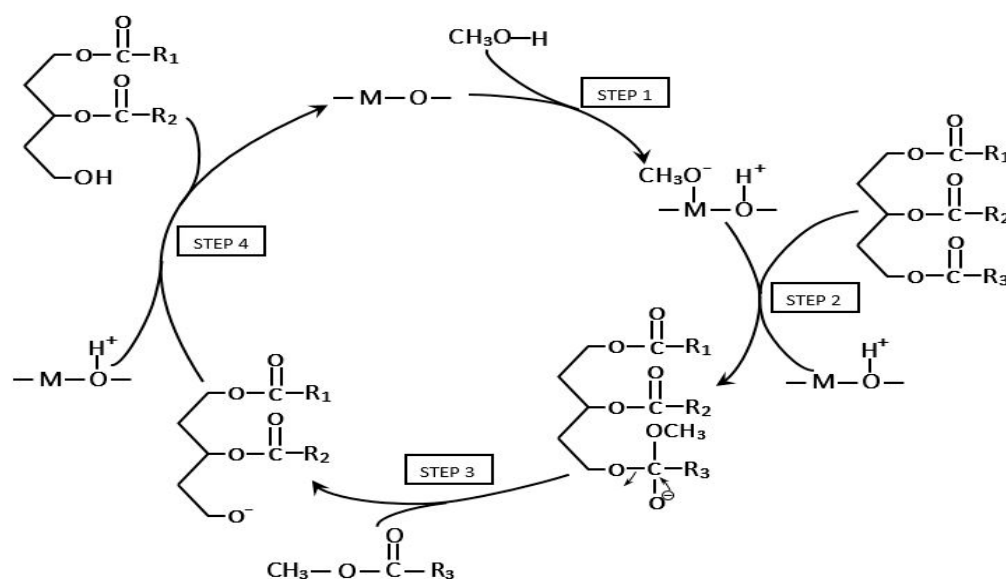


Figure 6. Suggested mechanism for the transesterification reaction under metal oxide catalyst. The schematic is based on the works of others [11,49].

Table 2 lists the FAME yields obtained with different commercial potassium-based catalysts and banana peel-derived catalysts reported in the literature [13,28,51,52]. The banana peel-derived catalysts used for biodiesel synthesis offer similar results to those obtained using commercial ones such as potassium carbonate supported on alumina. However, the use of banana peel-derived catalysts offers superior economic and environmental properties, as it not only aligns with the sustainability trends in renewable energy and waste valorization but also contributes to a circular economy and offers an economical approach for biodiesel synthesis, which is not the case for commercial catalysts [28]. As shown in Table 2, the calcination of the banana peels for 2 h at 600 °C gave a biodiesel yield of 95.1%. In this work, the choice of 700 °C as a calcination temperature increased the FAME yield to 99.4% ($\pm 0.28\%$). Moreover, the catalyst synthesized in this work was prepared through the direct calcination of the dried peels, which is not the case for other similar catalysts, where the dried peels are usually burned in open air and then calcined. These results present a simple and new method for catalyst preparation and demonstrate its higher efficiency as a sustainable and economically efficient substitute for traditional potassium-based catalysts.

Table 2. Comparison between commercial and banana peel-derived catalysts.

Catalyst	CTOR (wt%)	MOMR	Temperature (°C)	Time (min)	Yield (%)	Ref
K ₂ CO ₃ /Al ₂ O ₃	5	12:1	80	240	99.3	[52]
Activated Banana Peel Biochar	0.5	15:2	90	90	85	[51]
Calcined BPA at 600 °C for 2 h	1.5	15:1	65	60	95.1	[28]
Calcined BPA at 600 °C for 4 h	1	15:1 (ethanol to oil)	70	240	92.3	[13]
Calcined BPA at 700 °C for 4 h	4	9:1	65	90	99.4	This work

3.4. Catalyst Reusability

Heterogeneous catalyst stability and reusability represent an important advantage for low-cost biodiesel synthesis. The reusability performance of the catalyst was investigated for three consecutive runs using calcined (700 °C) banana peel ashes under optimum reaction conditions (MOMR = 9:1, CTOR = 4 wt%, stirring rate = 300 rpm, reaction temperature = 65 °C, and time = 90 min). At the end of each run, the catalyst was recovered and dried in an oven (at 110 °C for 4 h), without any further treatment before reusing it in the subsequent run. The obtained biodiesel yield following each reaction cycle is shown in Figure 7, where run 1 corresponds to the reaction performed with the fresh catalyst. The results show that the catalyst is stable, maintaining a biodiesel yield in the 99.4% ($\pm 0.28\%$) range. Figure 7 also compares the stability of the catalyst used in this study with other catalysts derived from banana peels [13,42,46]. Unlike the catalyst used in this study, the catalysts used in previous works showed a decrease in the biodiesel yield after the first run. These catalysts were regenerated using methanol, petroleum ether, or acetone washing and dried before being used in the next run to remove any biodiesel, glycerol, or unwanted molecules present on the catalyst surface [13,46]. The catalysts in these works all showed a decline in the FAME yield with each run, along with a decrease in the amount of available active sites and changes in the catalyst composition and properties, including the possible leaching of active sites [46]. These works also demonstrated a decrease in the concentration of potassium species, especially given that potassium shows a higher tendency for leaching compared to other elements, which directly affects the catalyst activity [42]. The constant high yields in our case indicate that the catalyst's alkaline active sites are not prone to

leaching and that catalyst poisoning or active phase blockage are unlikely. The findings highlight the good stability of the catalyst in biodiesel production and demonstrate its ability to be reused without intermediate washing between runs, which contributes to reduced energy consumption and lower overall production costs.

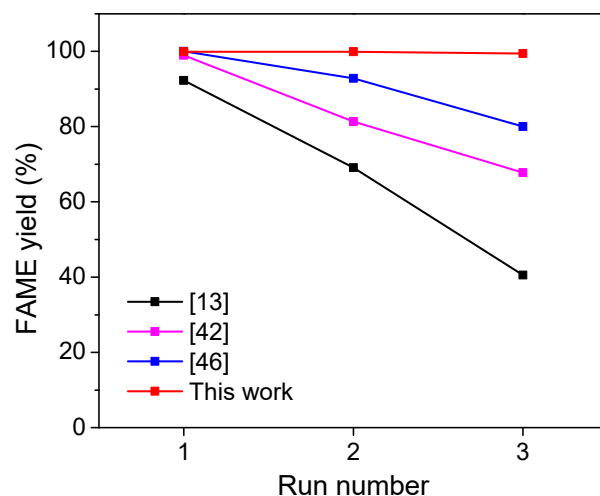


Figure 7. FAME yield versus run number during the transesterification of vegetable oil with banana peel-derived catalysts [13,42,46].

3.5. Active Phase Leaching Assessment

To assess active phase leaching during the reaction, the homogeneous contribution was evaluated by removing the catalyst from the reaction medium before reaction completion. Figure 8 plots the biodiesel yield versus time, with and without the catalyst. A FAME yield of 99.4% ($\pm 0.28\%$) was achieved after 90 min with presence of the banana peel catalyst. In a different experiment, the FAME yield was 91% ($\pm 0.98\%$) after 30 min. The catalyst was immediately removed at this time (30 min), and the reaction kept going for an additional hour. The FAME yields recorded at 60 and 90 min from the start remained constant at 91% ($\pm 0.98\%$). These results prove that the reaction occurs at the solid–liquid interface, and therefore the existence of a catalyst having a solid nature is essential for the reaction. Moreover, the reaction stopped when the catalyst was removed, indicating negligible active phase leaching and therefore stable catalytic activity over three consecutive runs.

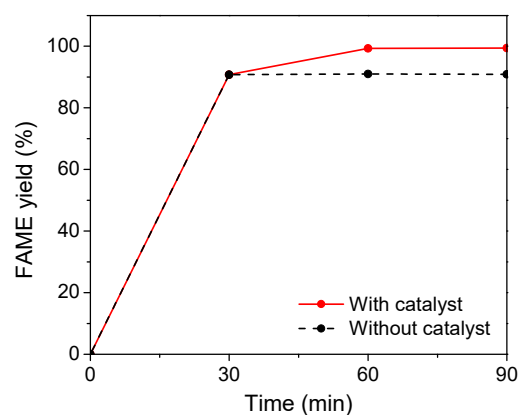


Figure 8. FAME yield (%) versus reaction time (min) in the presence of the waste banana peel catalyst and after its removal (MOMR = 9:1, CTOR = 4 wt%, stirring rate = 300 rpm, and T = 65 °C).

3.6. Kinetic Study

For the kinetic study, the transesterification reaction was performed at three separate temperatures (55, 60, and 65 °C). Following Equation (7), a plot of $-\ln(1 - X)$ versus time is presented in Figure 9. The increase in the reaction temperature causes a rise in the FAME yield. The slope corresponds to the reaction rate constant (k) that also increases with increasing temperature due to lower oil viscosity and improved mixing of reactants [31]. Figure 10 shows the Arrhenius plot of $\ln(k)$ versus $1/T$ according to Equation (8). The calculated activation energy (E_a) and Arrhenius frequency factor (A) are 58.1 kJ/mol and $7.4 \times 10^7 \text{ min}^{-1}$, respectively, and are comparable to results found in the literature [31,53]. The obtained value for E_a falls within the range (21–84 kJ/mol), explaining the rapid conversion of the reactants and showing the effectiveness of the catalyst for the transesterification reaction [4,29]. Studies where waste banana peels in particular were used as catalysts for biodiesel production proceeded with activation energies between 33 and 57.2 kJ/mol, allowing high biodiesel conversion at moderate reaction temperatures [29,53]. The activation energy is a thermodynamic constant defined by the catalyst active sites and reaction pathway. Increasing the temperature alters the viscosity of the feed and allows molecules to more easily overcome this E_a barrier, even though the activation energy remains constant. For highly viscous feeds, the reaction rate would be lower because of mass transfer limitations, but the activation energy would still remain unchanged [54].

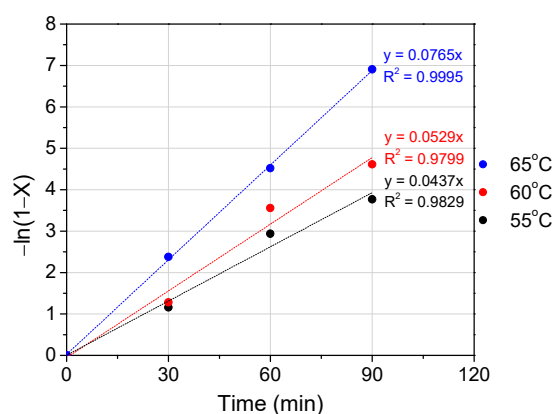


Figure 9. Plot of $-\ln(1 - X)$ versus reaction time at various temperatures (55 °C, 60 °C and 65 °C) (MOMR = 9:1, CTOR = 4 wt%, stirring rate = 300 rpm).

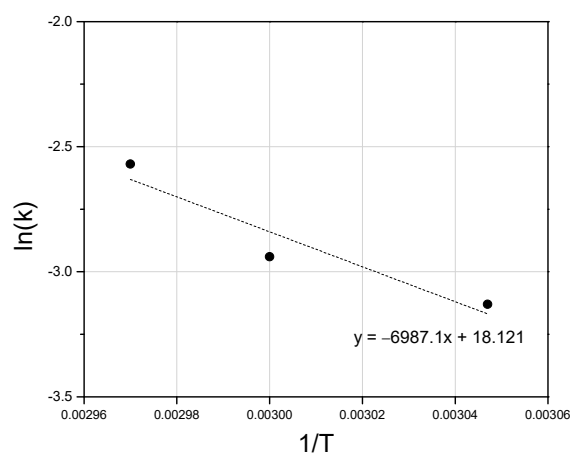


Figure 10. Arrhenius plot of $\ln(k)$ versus $1/T$ for the transesterification of sunflower oil using waste banana peel catalyst (MOMR = 9:1, CTOR = 4 wt%, stirring rate = 300 rpm).

3.7. Biodiesel Properties

The physical and chemical properties of the obtained biodiesel, such as density, flash point, and copper strip corrosion, were tested and checked against their ASTM requirements in Table 3. The acceptable density value ensures the easy and efficient delivery of a sufficient amount of fuel to the engine, while also decreasing dirt deposition [55]. Additionally, the high flash point value obtained confirms the safety of the storage and transportation of the biodiesel [55]. The low level of copper strip corrosion offers information about the level of corrosiveness inside the engine and fuel tank and ensures that the synthesized biodiesel does not present corrosion-related issues following its application. Based on ASTM standards, the maximum acceptable level of corrosion using the copper strip corrosion test is N°. 3. This work reported a very low corrosion level rating of 1a, corresponding to a slight tarnish almost similar to that of a freshly polished strip. This low corrosiveness level is associated with very low levels and even near-total absence of sulfur in the synthesized biodiesel molecules [56]. While biodiesel demonstrates better characteristics when compared with petroleum-based diesel, it differs in many properties, particularly in its stability and increased NOx emissions [57]. Due to the high oxygen content, biodiesel shows higher NOx levels upon combustion, raising environmental concerns [58]. Their high double bond content also causes reduced degradation resistance when exposed to heat, humidity, and light, as well as increased NOx emissions [57,58]. In order to meet environmental emissions standards and prevent potential operational problems, additives are being used to promote biodiesel properties [59]. These additives provide a wide range of benefits, such as corrosion protection, enhanced biodiesel stability for extended storage life, and better emissions cleanliness [59]. In terms of the obtained by-products, glycerine's purity was not quantified in this work. Previous works have shown that the glycerine produced during transesterification reactions using banana peel-derived catalysts was exceptionally clean and of high quality. The biochars may act as an in situ purification medium or a sorbent that traps and removes impurities that are produced during the transesterification reaction [60].

Table 3. Physical and chemical properties of the produced biodiesel.

Property	Value	ASTM Requirement
Density at 15 °C (Kg/m ³)	880	860–900
Flash point (°C)	135	>93
Copper strip corrosion	1A	<N°. 3

3.8. Catalytic Activity of Waste Banana Peel Ash Catalysts for WCO

The transesterification reaction of waste cooking oils using the synthesized banana peel-derived catalyst was also tested. Due to their negative impact on food security, low cost, and high availability, waste cooking oils have emerged as important candidates, known as second-generation feedstocks, for biodiesel synthesis [59]. Waste cooking oils were collected from households and restaurants and filtered to remove remaining solid particles prior to their usage in the reaction. Table 4 displays the properties of the collected WCO, with the obtained results demonstrating that the tested properties are consistent with the ASTM requirements. The transesterification reaction was performed under the previously obtained optimum conditions (CTOR = 4 wt%, MOMR = 9:1, T = 65 °C, stirring rate = 300 rpm, time = 90 min). A FAME yield of 99.1% ($\pm 0.14\%$) was achieved. This proves the suitability of the synthesized catalyst for biodiesel synthesis using waste cooking oils, offering a cost-effective and environmentally sustainable route for the biodiesel production process.

Table 4. Physical and chemical properties of waste cooking oil (WCO).

Property	Value	ASTM Requirement
Density at 25 °C (Kg/m ³)	918.4	918–921
FFA Content (wt%)	0.084	<0.5
Water Content (wt%)	0.07	<0.2

4. Conclusions

Being derived from agricultural residues, biomass-derived catalysts offer an economical and sustainable alternative for traditional commercial catalysts used for biodiesel synthesis. The application of such catalysts for biodiesel synthesis not only shows their ability in catalyzing transesterification reactions, but also promotes a circular economy by valorizing waste into valuable products, reducing the carbon footprint and cost of the process. Their biodegradable and environmentally benign nature make them a safe and viable option for the commercial-scale production of biodiesel. The present work showed the effectiveness of a heterogeneous base catalyst prepared from waste banana peels calcined at 700 °C for biodiesel production. The results revealed that an MOMR of 9:1, CTOR of 4 wt%, mixing rate of 300 rpm, temperature of 65 °C, and reaction time of 90 min were optimal for achieving a 99.4% ($\pm 0.28\%$) biodiesel yield. The catalyst also showed good stability, maintaining a yield of 99.4% ($\pm 0.28\%$) over three consecutive cycles with no observable active phase loss. The synthesized biodiesel showed good physico-chemical properties that were in accordance with the ASTM requirements. An activation energy of 58.1 kJ/mol and a pre-exponential factor of $7.4 \times 10^7 \text{ min}^{-1}$ were determined. Moreover, the banana peel catalyst was efficient for the transesterification of a second-generation WCO feedstock, as it showed a yield of 99.1% ($\pm 0.14\%$). These results show the high potential of banana peel-derived catalysts as sustainable, low-cost, and highly effective catalysts for large-scale biodiesel production.

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Abbreviations

The following abbreviations are used in this manuscript:

MOMR	Methanol-to-oil molar ratio
CTOR	Catalyst-to-oil ratio
FAME	Fatty acid methyl ester
WCO	Waste cooking oil

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