DOI Link: https://doi.org/10.61586/MfdR5
Vol.50, Issue.8, Part.1, August 2025, PP.45-60

Catalytic Conversion of Waste Cooking Oil to Biodiesel Using Date Palm Frond Ash

Raiedhah A. Alsaiari[1], Moustafa A. Rizk[2,3,*]

1-Department of Chemistry, College of science and art in Sharurah, Najran University, Saudi Arabia
2- Department of Chemistry, College of science and art, Najran University, Saudi Arabia
3- Chemistry Department, Faculty of Science, Suez Canal University, Ismailia, Egypt.
**Email: moustafarizk@yahoo.com*

Received June 2025 Accepted July 2025 Published August 2025

ABSTRACT

The current research examined the catalytic efficiency of ash derived from date palm fronds for the production of biodiesel by the ethyl transesterification of waste cooking oil. The optimal conditions for synthesizing the catalyst were determined by subjecting date palm fronds to various calcination temperatures (ranging from 300 to 700 ◦C). The catalyst produced was subjected to characterization using thermogravimetric analysis (TG-DTG), X-ray diffraction (XRD), BET, and scanning electron microscopy (SEM). The ideal parameters were as follows: a temperature of 85 ◦C, a molar ratio of alcohol to oil of 9:1, a catalyst concentration of 7% (w/w), and a reaction period of 1.5 hour. These conditions led to the formation of biodiesel containing 81% ester content. The reusability of the catalyst was evidenced by its sustained catalytic performance, achieving an ester content exceeding 78% during the initial two reaction cycles. A sustainable and effective catalyst for biodiesel manufacture, the synthesis of a catalyst from date palm fronds has numerous benefits: it is low cost, readily synthesizable, and derived from generally accessible biomass waste.

KEYWIRDS: Biodiesel Synthesis; Waste Cooking Oil (WCO); Ash from date palm fronds; Renewable Energy; Sustainable.

INTRODUCTION

For a significant duration, the global energy composition has mostly relied on fossil fuels. Nevertheless, the rapid use of these fuels has progressively exhausted the available supplies [1]. Furthermore, the increasing apprehension about environmental contamination has prompted various endeavors to alleviate these impacts. Another option is the creation and use of biofuels obtained from sustainable sources. In this particular scenario, biodiesel emerges as a potential alternative to petroleum diesel, either partially or completely [2].

Biodiesel is produced through the combination of alkyl esters derived from fatty acids obtained from renewable sources such as vegetable oils or animal fats. It may be produced via transesterification of triglycerides in the presence of primary alcohols. With certain advantages better lubricity, greater flash point (>130 ◦C) and cetane number (>47), biodiesel characteristics match those of petroleum diesel lack of toxicity, and reduced greenhouse gas emissions [3]

The biodiesel industry currently employs homogeneous catalysts. These catalysts exhibit high catalytic activity, allowing for a brief reaction time and modest reaction conditions. However, they have numerous drawbacks, including the production of significant quantities of effluent during the purification process and the challenges associated with recycling the catalyst.

Solid heterogeneous catalysts address these issues by being readily isolated from the reaction media and capable of being regenerated and reused. Consequently, the products are free from substantial contaminants caused by the catalyst, leading to a decrease in the expenses associated with further processing. Alternative materials have been investigated by numerous authors as potential catalysts for biodiesel production. Several investigations have shown that some calcined waste materials may be directly employed as heterogeneous catalysts without the need for any chemical modification. The biodiesel production process has become more cost-effective and environmentally sustainable due to the widespread availability, abundance of raw materials, and the low cost of catalysts synthesized from various waste sources [4].

The utilization of ash-based heterogeneous catalysts derived from biomass waste in biodiesel synthesis is increasingly attracting attention due to several advantageous factors: they are sustainable and renewable, unlike catalysts made from nonrenewable sources, they are easy to produce at a low cost, they don't require harmful chemicals, and they contribute to less waste. It has also been proposed that ash catalysts produced from biomass Demonstrate high catalytic efficiency in reaction processes, characterized by minimal leaching and outstanding recyclability [5]. This might be because alkali and alkaline earth metals are included in the composition of the catalysts.

A lot of research has been done on biomass-derived ash catalysts that can be used to make biofuel. For example, ash from ripe plantain fruit [6], snail shell [7], banana peel [8], acai seed [9] and hazelnut shell [10] can be used. All of this study has led to the interesting and relatively new idea of using wood ash catalysts with high catalytic activity. This is a hopeful development in the area of green energy. As a result, the abundance, simplicity of collection, and reusability of ash-based catalysts render them more efficient and useful than conventional catalysts. Typically, these biomass ash catalysts are generated through

calcination, burning, and dehydrating. They can be directly applied as catalysts without any modifications and have demonstrated prospective catalytic activities as a result of their fundamental character.

This led to the selection of date palm frond ash (which comes from a lot of farm waste in Saudi Arabia and is seen as a natural, eco-friendly source that is easy to find and doesn't cost much) as a raw material for this research aiming at the possibility of directly utilized it as a basic solid catalyst in the transesterification of waste cooking oil to produce biodiesel.

Materials and methods

The date palm frond and waste cooking oil were obtained from home. For the synthesis of biodiesel, ethanol was used.

Pre-treatment and Characteristics of Waste Cooking Oil (WCO)

The acquired oil was stored under standard atmospheric conditions. It was then filtered using a 110 nm screen to get rid of any residue and major food contaminants. In addition, it was adjusted to 110°C in order to eliminate any residual moisture. Numerous physicochemical characteristics of waste cooking oil (WCO), including density, free fatty acid value, kinematic viscosity, and molecular weight, were measured after the filtering and dehydration process. The results are shown in Table 1 based on the physicochemical characteristics.

Table 1: Physicochemical traits of waste cooking oil (WCO)

Characteristics	Measured value
Density (kg/m^3)	0.9
Free fatty acid %	0.8
Viscosity @40◦C	43
Molecular weight	875

Catalyst preparation

Initially, the fronds of the date palm were cleansed using distilled water to eliminate impurities and dirt. Subsequently, they underwent a drying process in a conventional oven at 100 °C for 24 hours to ensure the removal of residual moisture. Following this, the fronds were crushed and then sorted through a 10 mesh sieve. Subsequently, a quantity of 100 g of the substance underwent calcination in a muffle furnace, with temperatures ranging from 300 to 900 ◦C. The heating process occurred at a rate of 10 ◦C per minute and lasted for a duration of 5 hours. The ash from the date palm frond was crushed using an agate mortar and pestle and then kept in a desiccator for storage.

Transesterification reaction

The transesterification procedures were performed in a 100 cm^3 round-bottomed glass flask reactor that was magnetically agitated and equipped with a reflux condenser, as illustrated in Figure 1a. The catalyst was continuously stirred in an ethanol solution of a predetermined concentration while being maintained at 50 °C for a duration of 20 minutes. Afterwards, using a hot plate, the reaction mixtures were heated and the temperature was controlled within the range of 60 to 95 °C. The catalyst was typically used at a dose ranging from 1% to 9% in a standard reaction. Furthermore, the molar ratio of ethanol to waste cooking oil (WCO) was used according to the required standards. After the specified reaction time, the mixture was further cooled to reach room temperature. After the reaction was finished, the solution was moved to a different funnel. The glycerol and ethyl ester underwent phase separation, forming different layers. The product was left undisturbed overnight to achieve efficient separation. The various phases are readily discernible after an overnight period, as seen in Figure 1b. Upon completion of the transesterification experiment, the catalyst was separated by centrifugation. After the separation, the esters were subjected to a washing process using 500 mL of distilled water at a temperature of 80 ◦C. This was done in order to eliminate contaminants such as alcohol and any remaining glycerol. The biodiesel was subsequently oven-dried at 100 ◦C for 2 h and subsequently stored for further analysis.

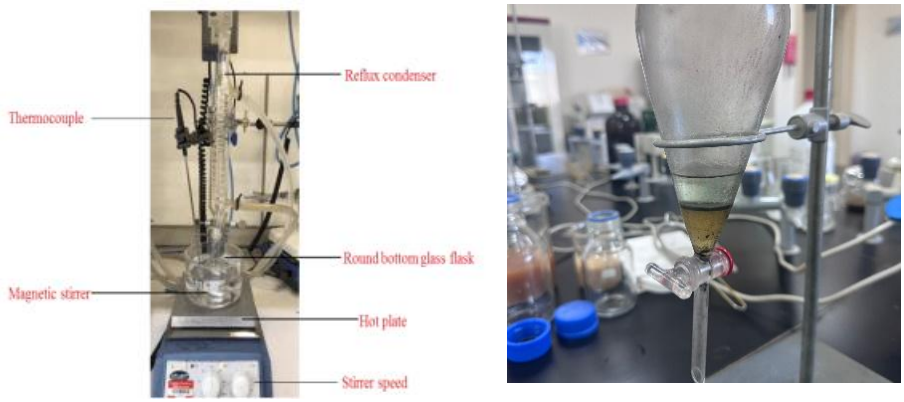


Figure 1: (a) Glass reactor for synthesis of biodiesel (b)Biodiesel separation After one-night stand biodiesel (the top layer) and glycerol.

Catalyst Regeneration

To examine the catalyst's reusability after the transesterification process, the catalyst was retrieved using centrifugation, rinsed with three 50 mL volumes of ethyl alcohol, and then dried in an oven at 115 ◦C for 16 hours. The catalyst that was recovered was used in the transesterification process, following the optimized conditions, for two reaction cycles.

The studies were repeated in order to determine the biodiesel production. The biodiesel yield refers to the percentage of the mass ratio between the biodiesel generated and the total mass of the waste cooking oil used. The calculation was performed with the equation:

$$\text{Yield } \% = \frac{Weight\ of\ biodiesel\ produced}{Weight\ of\ sample\ oil\ used} \text{ x } 100 \qquad (1)$$

Results and discussion

Direct transesterification of Waste Cooking Oil (WCO) was carried out to evaluate the effects of varying catalyst concentration, reaction temperature, ethanol-to-oil molar ratio, and reaction time, while maintaining all other parameters constant. The findings are presented and discussed in the following sections.

Catalyst characterization

BET

The frond ash showed the most favorable adsorption characteristics at 600 °C, with the highest surface area and pore volume. Lower temperatures (300 °C) resulted in incomplete activation, while higher temperatures (900 °C) led to structural degradation and loss of porosity. These observations are consistent with studies on bio-waste-derived adsorbents, where thermal activation must be optimized to balance pore development and structural integrity.

Table 2: Effect of Thermal Treatment on Surface Area, Pore Size, and Pore Volume of Frond Ash.

No.	Symbol	Surface area (m^2/g)	Pore Size (nm)	Pore Volume (cm^3/g)
1	Ash at 300 °C	62.2961	2.6880	0.083726
2	Ash at 600 °C	111.785	2.2874	0.12785
3	Ash at 900 °C	44.2311	1.6720	0.036976

Thermogravimetric analysis (TGA)

Thermogravimetric analysis was performed to determine the thermal properties of the grinded palm fronds. The thermal stability data were collected on a SHIMADZU, DTG 60 thermogravimetric analyzer (KK, SHIMADZU, Kyoto, Kyoto, Japan) under linear temperature conditions. The temperature was changed from 37.5 °C (Room T) to 1000 °C for sample of 26.75 mg placed in an aluminum pan at a heating rate of 10 °C/min under air atmosphere. The initial mass loss begins with the peak at 70 °C is related to moisture content of the sample which was calculated and found to be 6%. The second mass loss,

which is 17.3% from 50 °C to 195 ° C, is attributed to bonded volatile component like crystalline water. The third peak at 280 °C corresponding to mass loss of 20.19 % beginning at 239 °C to 307 °C is due to cellulose decomposition. The two small peaks at 812 °C and 880 °C correspond to mass loss of 3.38% and 4.96% respectively may be related to self-gasification of possibly formed charcoal.

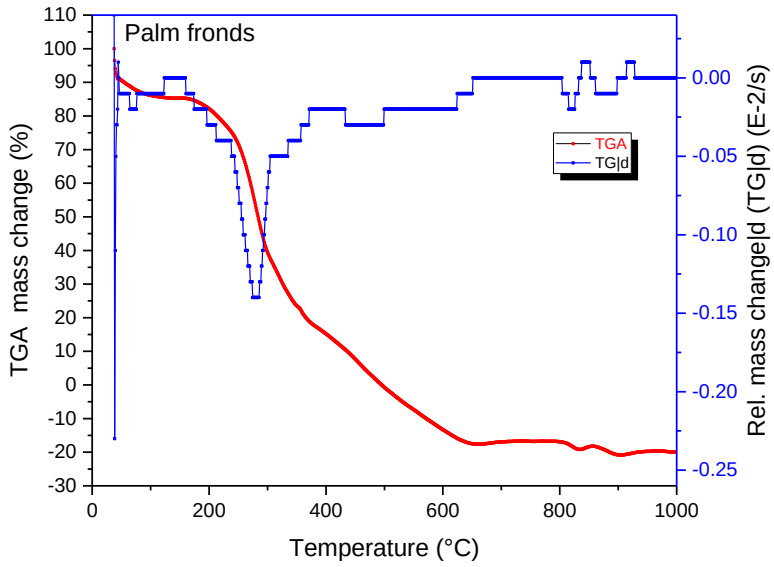


Figure 2: TGA for date fronds material.

SEM/EDAX analysis

The morphology and point element analysis of palm frond ash obtained at 600 °C are shown in Figure 3 (SEM) and Figure 4 (EDX). The Diopside mineral crystals appear in SEM pictures. The amorphous carbon appears which may be due to uncomplete burning. These results are confirmed by element percentage of EDX in table appears in figures 4. The particles' average surface area was calculated from the image to be ranged from 2 to 8 µm. Table in figure 4 displays the average concentration of the various chemical components as determined by EDX. This demonstrates that the Diopside mineral elements shown in XRD Figure 5 are present. All metals are present as oxides and oxysalts, as shown by high atomic percentage of oxygen.

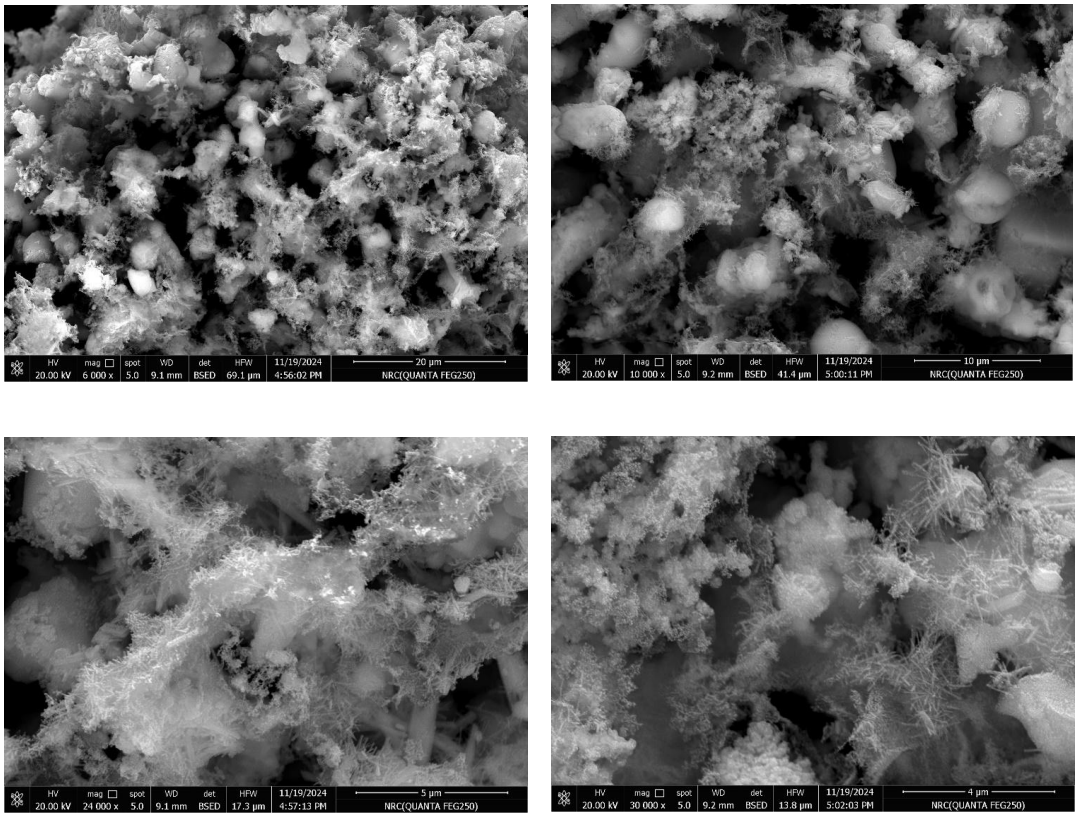
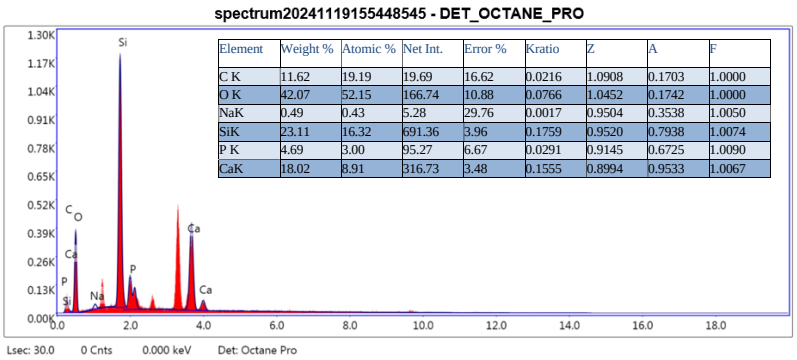


Figure 3: SEM image for ash derived from date palm fronds at different shown magnification.



Element	Weight %	Atomic %	Net Int.	Error %	Kratio	Z	A	F
C K	11.62	19.19	19.69	16.62	0.0216	1.0908	0.1703	1.0000
O K	42.07	52.15	166.74	10.88	0.0766	1.0452	0.1742	1.0000
NaK	0.49	0.43	5.28	29.76	0.0017	0.9504	0.3538	1.0050
SiK	23.11	16.32	691.36	3.96	0.1759	0.9520	0.7938	1.0074
P K	4.69	3.00	95.27	6.67	0.0291	0.9145	0.6725	1.0090
CaK	18.02	8.91	316.73	3.48	0.1555	0.8994	0.9533	1.0067

Figure 4: Element content of date palm frond ash at 600 °C.

XRD Analysis

Crystallographic and amorphous structure of all synthesized catalysts as well as for untreated palm frond particles are investigated by XRD analysis. The d spacing of crystallin forms in different samples are collected from 2Θ of 2 to 60 using Bruker D8 Advance diffractometer, operated at 40kv and 40mA using CU Ka radiation (1.54060). Powder diffraction X-ray analysis Figure 5 shows that all samples contain silicates of varying crystalline grades. The grinded date palm frond sample exhibits amorphous cristobalite. Diopside mineral of varying crystalline grades is also evident in the ash produced at different temperatures. The degree of crystallinity also increases with increasing temperature from 300°C to 900°C, with the diffraction peaks becoming higher and sharper.

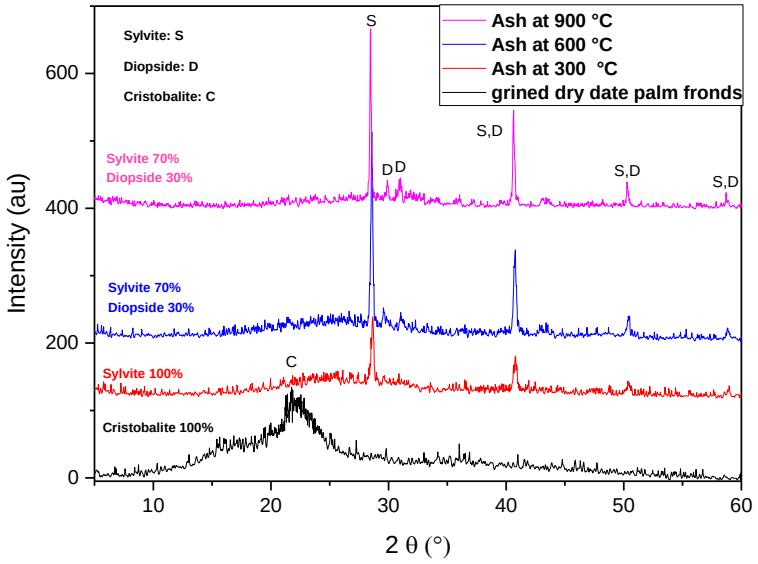


Figure 5: XRD results for synthesized catalysts from waste date palm fronds calcined at different temperatures.

Catalyst temperature and calcination time: effects on the biodiesel conversion

The influence of calcination temperature and duration on catalyst preparation was investigated to optimize its activity in the transesterification reaction. The biodiesel samples exhibited significantly low yield values of 26%, 47%, and 58% when was used catalysts derived from calcination processes at temperatures of 300 ◦C, 400 ◦C, and 500 ◦C, respectively. These findings indicate that when biomass is heated to temperatures as high as 500 ◦C, it undergoes incomplete carbonization. This means that a large quantity of

carbonaceous components is left behind as a consequence of the thermal degradation of hemicellulose, cellulose, and lignin [11]. Table 4 demonstrates that a yield of about 81.0% biodiesel was achieved when the biomass was subjected to calcination at temperatures of 600 and 700 ◦C. The significant rise in biodiesel production is due to more thorough thermal decomposition, leading to the creation of ash that includes carbonates, phosphates, and metal oxides. These findings are consistent with the studies conducted by Luque et al. [12]. According to their findings, the majority of carbon compounds break down between temperatures of 300 to 500 ◦C when exposed to air. At a temperature of 600-800 ◦C, the carbonaceous material is completely converted into ash, mostly metal oxides. The high conversions may be attributed to the ash's inherent nature, which allows it to serve as a catalyst for transesterification process. Biodiesel with ester concentrations of 81.0% for 4 hours at 600◦C. As a result, calcination periods of four hours enhance ash production, which increases the pace at which the transesterification processes convert. As a result, 600◦C and 4 hours were chosen as the calcination parameters for the catalyst production used in the subsequent studies.

Table 4: effect of calcination temperature of the catalyst

Calcination temperature (◦C)	Biodiesel yield (%)
300	26
400	47
500	58
600	81
700	81

Reaction conditions: loading of catalyst 7 wt%, molar ration of date seed oil to ethanol 1:9, 85C, 1.5 h.

Effect of the catalyst dose

Biodiesel synthesis reactions have been determined to be significantly influenced by the quantity of catalyst. The reaction is demonstrated to be highly improbable in the absence of a catalyst. However, the reaction efficiency may be reduced as a result of the reactants' inability to adequately interact with the catalyst as a result of inadequate mixing within the reaction mixture in the event of an excessive quantity of catalyst. Catalysts used insufficiently produces less than ideal reaction efficiency. Therefore, it is important to look into the ideal amount of the catalyst. Under specific conditions including ethanol to oil molar ratio of 9:1, a reaction temperature of 85 ◦C, a stirring speed of 800 rpm, and a reaction length of 90 minutes. the study concentrated on looking at how catalyst concentration affected the optimization process. The authors hypothesized that a catalyst possessing numerous highly reactive basic sites and a large surface area would demonstrate enhanced catalytic activity [13]. Within the scope of this inquiry, the amount of the catalyst

was altered over a range of 1% to 8% in relation to the weight of the oil, as seen in Figure 6. The biodiesel production quantity shown a positive correlation with the increased amount of catalyst. Significantly, the greatest amount of biodiesel was produced by introducing 7% of date palm fronds ash, resulting in an impressive 81% yield of biodiesel under the specified reaction conditions. Furthermore, the reduction in biodiesel production when the loading exceeds 7% may be ascribed to an excessive concentration of heterogeneous catalysts within the reaction system. An excessive amount of catalysts might hinder the proper merging of reactants, resulting in a reduction in the overall generation of biodiesel. Higher catalyst loadings may lead to limits in the transfer of mass for reactants and products, which can both contribute to less than ideal biodiesel synthesis. Furthermore, an increase in the viscosity of the reactant mixture may impede mixing and mass transfer processes, ultimately leading to a decrease in biodiesel yield.

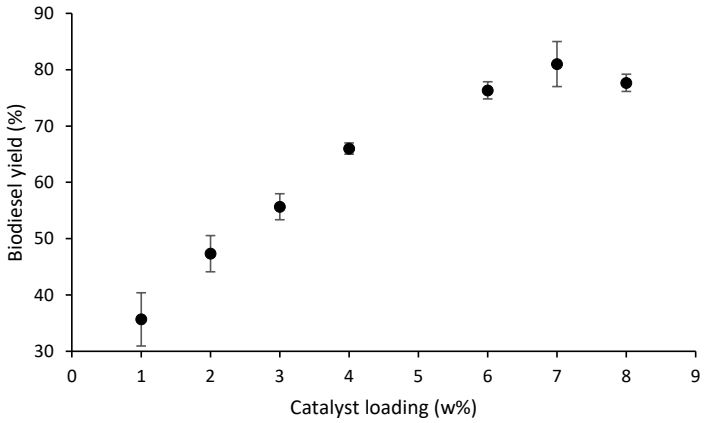


Figure 6: Effect of the concentration of the catalyst on biodiesel yield (%). Reaction conditions: ethanol to date seed oil ratio of 9:1, reaction time 1.5 h, and reaction temperature of 85 ◦C.

Effect of ethanol-to-oil molar ratio

Multiple research investigations have shown that the molar ratio of triglyceride to alcohol is a crucial factor in determining the amount of biodiesel that can be produced [14]. In the transesterification process, three moles of alcohol react with one mole of triglyceride, resulting in the formation of three moles of fatty acid esters and one mole of glycerol, according to theoretical calculations. Nevertheless, since the reaction is reversible, a considerable amount of alcohol is used to facilitate the reaction in the intended direction, resulting in a higher quantity of ester conversion in a somewhat shorter period of time. Initially, a molar ratio of 3:1 was used in this experiment. Later on, the ratio was increased from 3:1 to 12:1. Figure 7 highlights the elements that contribute to the variability in biodiesel production when the molar ratio is changed. Adjusting the molar ratio from 3:1 to 9:1 led to a marked enhancement in biodiesel yield. However, raising the ratio any more

did not result in a growth in the yield of biodiesel that was equivalent. Moreover, it has been shown that an excess of ethanol causes the content of soluble glycerol in biodiesel to rise. The high solute content of glycerol might possibly hinder the biodiesel separation process. This discovery might perhaps be explained by the solution containing an excessive amount of alcohol.

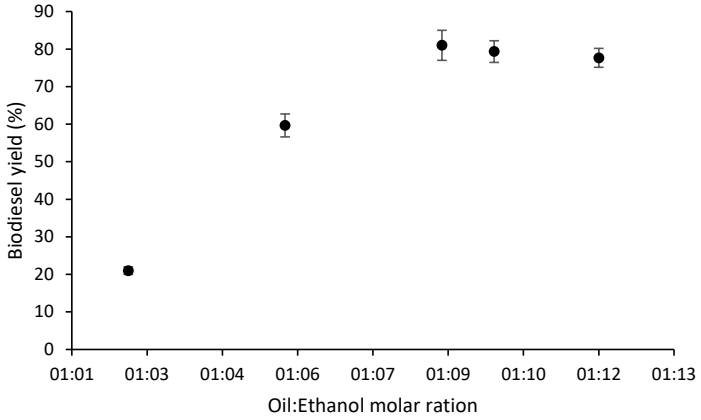


Figure 7: Effect of molar ratio of oil to ethanol on biodiesel yield (%). Reaction conditions: loading of catalyst 7 wt%, time of reaction 1.5 hours, temperature of reaction 85 ◦C.

Effect of reaction temperature

The reaction rate was accelerated due to the generation of more nucleophilic sites resulting from the movement and diffusion of reactant molecules induced by the elevated reaction temperature. Nevertheless, when the temperature of the reaction increased, there was a potential for a significant evaporation of ethanol, this caused a significant decrease in the overall yield of biodiesel. The effect of reaction temperature on the production of ethyl ester biodiesel is seen in Figure 8. The substrate's ascent above the activation energy barrier is facilitated by temperature increase. [15].

Because the oil has less viscosity at higher reaction temperatures, biodiesel production often increases [16]. The generation of biodiesel achieved 81% of its maximum at a temperature reaction of 85 ◦C. the production of biodiesel was decreased when the reaction temperature was lower or higher than 80 °C. The inadequate reaction temperature leads to increased viscosity of the oil at lower temperatures, resulting in insufficient mixing among the reactants. Biodiesel production does not improve when the reaction temperature exceeds 85◦C. According to Ngige and colleague [17], increasing the reaction temperature accelerates saponification rate. Furthermore, mentioned by Baskar et al. [18] was a drop in ethanol's polarity resulting from too heated reactants during a transesterification process. One acknowledges that the reduced polarity is a factor for the limited biodiesel synthesis at high reaction temperatures.

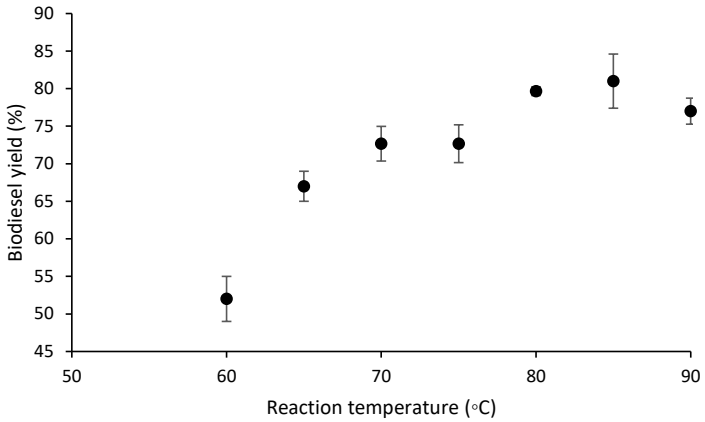


Figure 8: Impact of Reaction Temperature on the Efficiency and Yield of Biodiesel Production (%). Reaction conditions: loading of catalyst 7 wt%, molar ration of oil to ethanol 1:9, 1.5 h.

Effect of reaction time

Due to the direct relationship between energy use and time in the biodiesel synthesis process, it is crucial to determine an adequate reaction time. To examine the influence of reaction time on the proportion of biodiesel production, the length of the reaction was adjusted between 1 and 5 hours, as seen in Figure 9.

The reaction's initial yield experienced a substantial increase after being conducted for 1.5 hours. This phenomenon may be explained by the fact that reactants require a specific minimum duration to conduct a reaction, which is frequently referred to as the activation energy. The yield percentage decreased across all catalyst concentration and ethanol/ratio values when the reaction duration was increased from 2.5 to 4 hours and subsequently to 5 hours. The decrease in value may be ascribed to the reversible nature of the transesterification process. Consequently, the yield % drops after it beyond the ideal level. The observed phenomena may be explained by the steady reduction in the number of active sites that can be accessed over time, which happens due to catalyst deactivation. The highest yield of 81% was reached in 1.5 hours, which could be seen as the best in terms of cost-effectiveness and biofuel production compared to other catalysts. On top of that, using a longer reaction time could cause saponification or the opposite reaction, which would slow down the conversion of waste cooking oil (WCO).

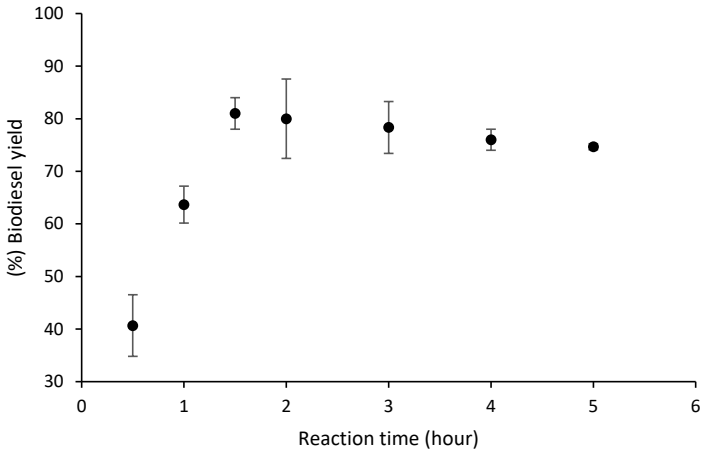


Figure 9: Effect of time of reaction on the yield of biodiesel (%). Reaction conditions: loading of catalyst 7 wt%, molar ratio of oil to ethanol 1:9, and reaction temperature 85 ◦C.

Catalyst reuse

The primary benefit of a heterogeneous catalyst is its reusability, which makes it a desirable option for the biodiesel industry. Using the ideal reaction conditions, the catalyst made from ash date palm fronds was tested for reuse four times over. Table 5 provides an illustration of the findings. The biodiesel yield was calculated after each cycle. The biodiesel samples obtained from the first and second reaction cycles demonstrated elevated ester content, recorded at 79.6% and 78.5%, respectively. However, the third reaction cycle resulted in a 35.0% drop in biodiesel production compared to the first reaction cycle, yielding 46.0%. The pronounced decline may be attributed to the fouling and subsequent deactivation of the catalyst surface by residual triglycerides and glycerol, leading to a reduced number of accessible active sites and, thereby, a diminished conversion rate of triglycerides to biodiesel [19]. Moreover, the decline in the catalytic activity might also be attributed to the loss of catalyst material by leaching during the transesterification process.

Table 5: Investigation of the Reusability Potential of the Ash Catalyst

catalyst	Biodiesel yield (%)
Fresh	81
1st used	79.6
2nd used	78.5
3rd used	46

Reaction conditions: loading of catalyst 7wt%, molar ration of oil to ethanol 1:9, time of reaction 1.5hours, reaction temperature 85 °C.

Biodiesel characterization

The ASTM D6751 standard's limits were compared to the physicochemical properties of the biodiesel synthesized using the established optimal conditions, as illustrated in Table 6. The biodiesel exhibited a kinematic viscosity of 2.7 $mm^2\ s^{-1}$ and a density of 0.869 g cm^{-3}. The values conform to the limitations set by the ASTM standard. The flashpoint defines the categorization of the material's flammability, making it an important criterion for storage and transit safety [20].

With an estimated flashpoint of 122.0 C, the biodiesel produced under ideal circumstances provides for improved safety during fuel handling, storage, and distribution. Elevated acidity levels can exert a corrosive impact on the metallic components of engine systems, hence the biodiesel acid number has to be watched. The investigation yielded a biodiesel acid value of 0.25 mg KOH g^{-1}, which is much less than the ASTM standard. As a result, the physicochemical characterization of the biodiesel reveals conformance with the ASTM D6751 standard and highlights the high quality of biodiesel produced under ideal circumstances.

Table 6: Assessment of Fuel Properties of Optimally Synthesized Biodiesel.

Property	Unit	ASTM	Measured value for prepared biodiesel
Density	kg/m^3	860-894	869
Viscosity @40 ºC	mm^2/s	1.9-6.0	2.7
Acid number	mg KOH/g	≤ 0.5	0.25
Flash point	ºC	>120	122

CONCLUSIONS

In this study, the impact of temperature variation and calcination periods on the synthesis of a novel basic heterogeneous catalyst from date palm fronds were examined. The catalyst that was created by calcining at 800 °C for four hours showed the maximum biodiesel output. Under ideal conditions that is, a temperature reaction of 85 ◦C, molar ration of oil to alcohol of 1:9, loading of catalyst 7.0% (w/w), and reaction duration of 1.5 hour, the biodiesel projected by the regression model was 81.0%. The catalyst was used in consecutive reaction cycles and exhibited appropriate catalytic performance until the third reaction cycle. The catalyst derived from date palm fronds exhibited the excellence and effectiveness of catalysis in transesterification of waste cooking oil, resulting in cost-effective and environmentally-friendly biodiesel generation.

ORCID IDS

Raiedhah A. Alsaiari: https://orcid.org/0000-0002-0735-3162

Moustafa A. Rizk: https://orcid.org/0000-0002-4482-2992

Acknowledgment:
The authors are thankful to the Deanship of Graduate Studies and Scientific Research at Najran University for funding this work under the Growth Funding Program grant code (NU/GP/SERC/13/264-1).

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