Global NEST Journal, Vol 27, No 5, 06871

Activated carbon from waste date palm fronds: A sustainable green heterogeneous catalyst for biodiesel synthesis

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Received: 07/10/2024, Accepted: 23/04/2025, Available online: 24/04/2025
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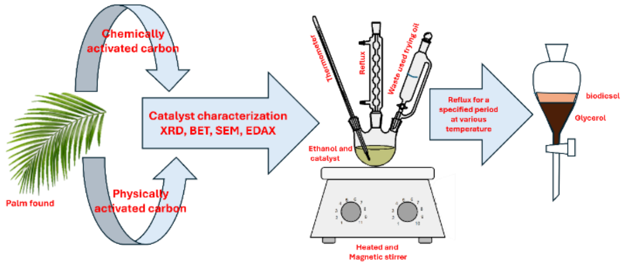
https://doi.org/10.30955/gnj.06871

Graphical abstract



Abstract

Biodiesel is a well-established, environmentally friendly alternative to fossil fuels. As well as less expensive and able to provide greener energy. All of which have favorably impacted bioeconomy. Physically activated and chemically activated carbon catalysts were synthesized from clean and dry date palm fronds at varying conditions and temperatures. Brunauer–Emmett–Teller (BET), and thermal gravimetric analysis (TGA) were utilized to characterize this catalyst. The results demonstrated that as the calcination temperature increased, the pore size of the chemically activated carbon catalyst decreased. The catalysts were used to produce eco-friendly biodiesel from examined waste cooking oil by optimal yield of (88 mass%). Optimal reaction conditions utilized included a catalyst concentration of 5 mass%, an oil to ethanol molar ratio of 1:10, and a temperature of 80°C for a duration of 3 hours. The potential of fatty acid ethyl ester to function as a suitable substitute fuel is indicated by its compliance with the fuel properties outlined in ASTM D 6751. Consequently, it is commendable to create and execute an environmentally friendly and energy-efficient strategy that employs biodiesel derived from abundant agricultural waste.

Keywords: biodiesel, waste cooking oil, calcination, activated carbon, environmentally friendly.

1. Introduction

Activated carbon is an exceptional adsorbent due to its superior adsorption characteristics. Carbon may be synthesized from a variety of sources, including biomasses from agricultural by-products, garbage, wood, and certain polymers, to create activated carbon (AC). Activated carbons are widely used in many fields to remove color, clean, separate, and remove smells from fats and veggie oils, clean water and treat pollution, clean petrol and air, and in the food and drug industries (Lua and Yang 2004, Gurten *et al.* 2012, Al-Swaidan *et al.* 2011). The increasing significance of low-cost agricultural byproduct biomasses and associated wastes is evident. Activated carbons may be derived from many carbonaceous materials, including coal, wood, lignin, and various types of shells (such as coconut, walnut, palm, pecan, almond, and physic nut). Other sources include cotton stalks, maize cob, chickpea husk, tobacco stems, sugar cane bagasse, date stones, and cherry stones (Wu *et al.* 2005, Ould-Idriss *et al.* 2011, Arami-Niya *et al.* 2012).

There are two distinct approaches to producing activated carbons: chemical activation and physical activation. The chemical activation method involves initial precursor activation using carbonaceous materials, which is subsequently carbonized at varying temperatures. Charcoal is physically activated by first carbonizing it at a high temperature and then using activating chemicals such steam, oxygen, and carbon dioxide to activate it. When compared to physical activation, chemical activation is often carried out at lower temperatures. Chemical activation results in greater carbon yields and pore formation than physical activation (Ahmadpour and Do 1997, Fierro *et al.* 2007, Yaman *et al.* 2006).

In Saudi Arabia, where more than 20 million trees are grown, the date palm is one of the most important and widely grown plants. It is thought that 75,000 tonnes of date frond are cut every year because three million date palm trees are pruned (Al-Juruf, 2002). The fronds of the date palm tree are often burned or thrown away as garbage, which has a negative impact on the environment.

Fatty acid methyl esters (FAMEs) have extensive uses in biosurfactants, biolubricants, environmentally friendly

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solvents, hydraulic fluids, dispersion agents, as well as cosmetic (Encinar *et al.* 2020, Esipovich *et al*. 2019, Salimon *et al.* 2012). These substances are not harmful and can be replenished, and they can easily break down naturally (Radzi *et al.* 2006). Fatty acid methyl esters (FAMEs) have comparable characteristics with diesel oil, making them suitable for use as a biofuel. In addition, they possess low toxicity, high fat solubility, and exceptional wetting properties at surfaces. Additionally, this characteristic renders them appealing constituents in cosmetic compositions (Radzi *et al.* 2006). Various sources of TAGs (triacylglycerols), including vegetable oil, animal fats, and algal oil, may be used in the process of transesterification. The composition and purity of fatty acid esters of alcohols derived from triglycerides are determined (Singh *et al*. 2020).

The transesterification process of vegetable oils in commercial settings mostly employs a homogeneous basic catalyst as the preferred method. The catalysts, such as KOH, NaOH, or CH_3ONa, are the most typical examples. Advantages of this transesterification method include a high reaction rate under mild reaction conditions (atmospheric pressure and temperature below the boiling point of methanol or ethanol), as well as a catalyst that is both inexpensive and widely available.

An alkaline catalyst is ineffective for converting oils with large levels of FFAs (free fatty acids), and the transesterification process must be conducted in conditions without water (Terigar *et al*. 2010, Gog *et al.* 2012). This is because the alkali catalyst's efficacy is enhanced by its inclusion of free fatty acids (FFAs). Furthermore, the formation of soaps adds complexity to the process of separating the ester phase from the glycerin phase and purifying the ester phase (Gog *et al.* 2012). As an alternative to alkaline catalysts, acid homogeneous catalysts may convert oils with a high concentration of free fatty acids. Organic sulfonic acid, HCl, H_2SO_4, and $Fe_2(SO_4)_3$ are among the most prevalent. On the contrary, acid-catalyzed transesterification reactions are not advised owing to their relatively sluggish reaction rate, which necessitates alcohol in greater quantities and temperatures exceeding 100°C (Talha *et al*. 2016).

Heterogeneous catalysts have recently attracted a lot of attention due to their reduced susceptibility to FFA contamination during transesterification. Transesterification expenses could be reduced through the application of inexpensive catalyst substitutes. Heterogeneous catalysts have many benefits in the manufacturing process. Firstly, they can be readily isolated from the reaction products. Secondly, they are reusable and exhibit high catalytic activity. Lastly, they may be used several times for transesterification. Specific stages in the process of transesterification, such as the purifying of fatty acid methyl ester and glycerol, might be excluded.

A promising and inexpensive solid carrier for chemical catalysis is activated carbon. Activated carbon is essentially produced by chemically or physically activating a variety of carbon materials, including biomass, coal, and wood. Previously, anthracite and bituminous coal have been the primary sources of activated carbon. In the present day, any substance that has a significant amount of carbon and a little amount of inorganic matter may be used to produce activated carbon (Sajjadi *et al.* 2019). There are many different kinds of micropores and macropores that are properly separated in activated carbon. Because activated carbon has tiny holes in it, it can be used to speed up chemical processes. Due to their affordability and ability to withstand high temperatures and radiation, they are often used in the transesterification of triglycerides (Gupta *et al*. 2014). The adsorption surface area of the biomass generated material depends on the activation technique used, which may be either physical or chemical. The range of surface area is between 500 m^2/g and 3000 m^2/g, as reported in references (Vargas *et al*. 2010).

Most frequently, activated carbon is employed as a carrier for CaO (Zu *et al*. 2010), KOH (Narowska *et al*., 2019), and lipase (Giraldo *et al*. 2012) during the transesterification process. In contrast, the efficacy of transesterification is 91.3% for KOH/AC catalysts and 87.0% for CaO catalysts (Babel *et al*. 2016). Biodiesel production from oil of bitter almond was produced with a yield of 93.2% by using acetate potassium as a heterogeneous catalyst on AC (Fadhil *et al.* 2016). Prior research has indicated that the utilization of heterogeneous catalysts in the transesterification process necessitates either a substantial surplus of alcohol or a considerable quantity of catalyst [Raiedhah *et al*. 2023).

This research aims to produce activated carbon from date palm fronds waste, which is a plentiful agricultural by-product in Saudi Arabia and it is considered a natural, environmentally friendly source, widely available, and financially inexpensive, via the method of physical and chemical activation as a catalyst for the biodiesel production using WCO. Activated carbons are characterized to ascertain the morphological and chemical alterations.

2. Experimental

2.1. Materials and methods

2.1.1. Pre-treatment and characteristics of waste cooking oil

The oil obtained was stored at standard atmospheric conditions. Subsequently, it underwent filtration using a 110 nm screen to effectively remove any notable food impurities and remnants. Moreover, the temperature was adjusted to 110°C in order to eliminate any remaining moisture. Various physicochemical parameters of waste cooking oil (WCO), including density, free fatty acid value, kinematic viscosity, and molecular weight, were ascertained subsequent to the filtration and dehydration procedures. The results are presented in **Table 1** in accordance with the physicochemical parameters. The outcomes of the gas chromatography analysis to ascertain the fatty acid content are presented in **Table 2**.

2.1.2. Preparation of activated carbon

The process of producing activated carbons included the collection of waste fronds from date plants in Sharurah, located in the south of Saudi Arabia. The fronds of the gathered date were first chopped into pieces of 1-5 cm. Subsequently, they were washed several times using tap water and then distilled water to remove any foreign substances like as dust, fibres, and contaminants. The cleaned leaves are subjected to a drying process at a temperature of 100°C for a duration of 8 hours in order to eliminate any remaining moisture.

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

Characteristics	Measured value
Density (kg/m^3)	0.9
Free fatty acid (mass %)	0.8
Viscosity at 40°C (cP)	43
Molecular weight (g/mol)	875

Table 2: Fatty acid composition of waste cooking oil

Fatty acid	Composition (mass %)
Methyl myristate (C14:0)	0.11
Palmitic acid (C16:0)	2.11
Oleic acid (C18:1)	3.32
Linoleic acid (C18:2)	58.32
Arachidic acid (C20:0)	0.48

2.1.3. physical activation

After drying and grind the sample, the process was carried out at different calcination temperatures (300◦C, 600◦C, 900◦C) for 3 hours under a nitrogen atmosphere. The sample was pressed into a stainless steel container to isolate oxygen. After the carbonation process: samples were taken and weighed. They were then ground, weighed again, and the samples were divided into containers as shown in **Figure 1.**

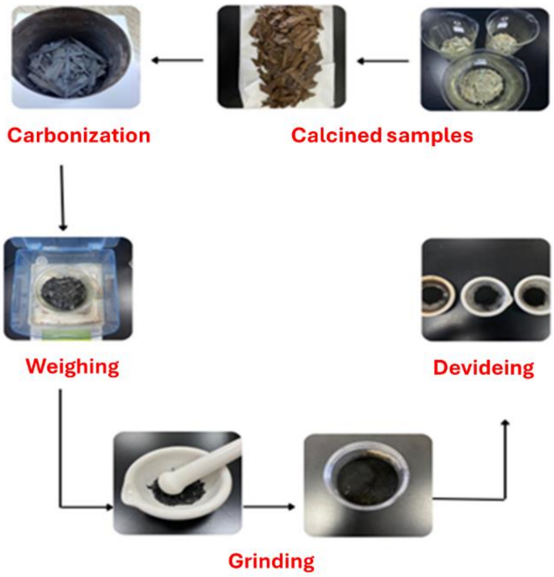


Figure 1. Physical activation of raw materials

2.1.4. Chemical activation

The primary process used for the production of activated carbons from date palm fronds waste involves activation and pyrolysis. KOH was used as an activating agent to chemically activate the precursor, which is the waste from the fronds of the date. A dried sample of date palm fronds, weighing 20 g, was immersed in a solution containing 200 mL of KOH at concentrations of 15% for a duration of 24 hours at a temperature of 24°C. The soaked fronds were subjected to a Muffle furnace at a temperature of 100°C for a duration of 3 hours until they were completely dried. Subsequently, the frond of the date was subjected to carbonization by heating it in a furnace at an activation temperature of 300°C for a duration of 3 hours. The activated carbons were expanded. The activated carbons are rinsed with a hydrochloric acid (HCl) solution in order to lower the pH. Excess activating agent was removed from the washing effluent until it reached a pH of 7 using double distilled water. Ultimately, the samples were subjected to a drying process at a temperature of 110°C in an oven in order to eliminate the moisture present.

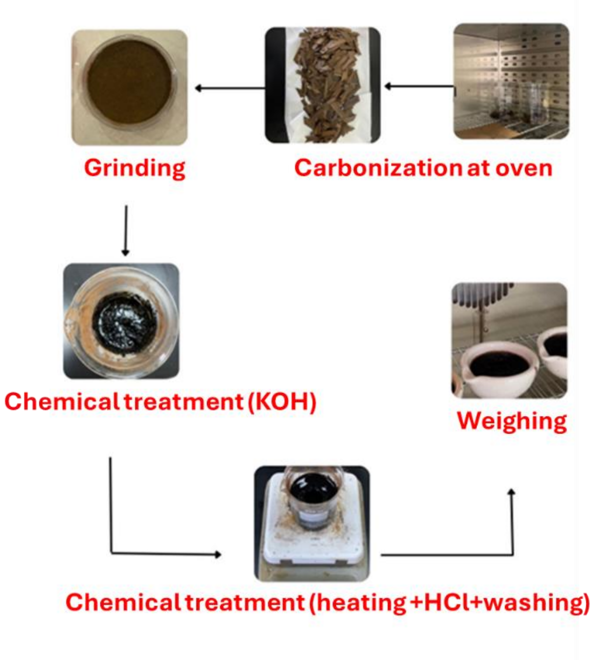


Figure 2. Chemical activation of date palm fronds.

2.1.5. Experimental Procedures for transesterification reaction

Activated carbon catalysts were assessed for their catalytic capabilities during the transesterification of used heating oil and ethanol in order to generate biodiesel. A 100-ml round-bottom flask was utilized to conduct the transesterification; it was also equipped with a thermometer, magnetic stirrer, and a reflux condenser as shown in **Figure 3**. In general, the process involved preheating a mixture comprising ethanol and catalyst at 60°C for 30 minutes while agitating with a magnetic stir. Subsequently, the waste used frying oil was added. Following vigorous agitation at 800 rpm, the reaction mixture is refluxed for a specified period of time at various temperatures. After the completion of the reaction, the ester phase was isolated from the glycerol phase using a separation funnel and further purified by washing it with distilled water to eliminate the catalyst (**Figure 3b**). Subsequently, the biodiesel was subjected to dehydration using anhydrous Na2SO4, followed by filtration and subsequent determination of its weight.

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:

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

From our trans-esterification expert in several of our previously published articles, we find that ethanol is faster than methanol. Ethanol is also less toxic and safer for environmental sustainability because it is biodegradable, reduces emissions and is more energy efficient. Ethanol reduces the agglomeration of the heterogeneous catalyst. This leads to more dispersion of the catalyst in the reaction medium, larger catalyst exposer surface and a faster reaction.

3. Result and discussion

3.1. Characterization of the Activated Carbon

3.1.1. Catalysts surface analysis

Brunauer–Emmett–Teller (BET) analysis (Quantachrome, Boynton, Florida, USA) used to investigate the potential of prepared catalysts to produce biodiesel. As shown on **Table 3** the surface area of chemically activated samples thermally treated at 600°C and 900°C are 35.7 m^2/g and 48.64 m^2/g. These areas are the smallest of all samples so these are not included for further check as catalyst for biodiesel production. The highest biodiesel yield of 88% is obtained from the highest surface area catalyst, which produced by physical activation at 900°C. It is clear from the data of biodiesel yield (Table 4) and surface properties (**Table 3**) generally that, as the surface is increasing, the biodiesel yield increases. For physically activated catalysts, increasing carbonation temperature from 300 to 900°C generally increase carbon content as well as surface area and pore size.

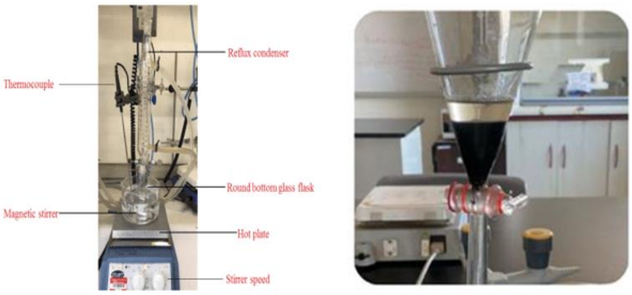


Figure 3. (a) Glass reactor for synthesis of biodiesel at different parameters (amount of catalyst from 1 to 10 mass%, reaction time range 1-5h, reaction temperature from 60 to 90°C and ethanol to oil molar ratio from 3:1 to 12:1) (b)Biodiesel separation After one-night stand biodiesel (the top layer) and glycerol.

Table 3. BET for date frond material

Date frond material	Surface area (m^2/g)	Pore Size (nm)	Pore Volume (cm^3/g)
Without treatment	72.8345	1.8755	0.068302
Physical activation at 300◦C	136.612	2.1146	0.14444
Physical activation at 600◦C	105.983	2.0380	0.10800
Physical activation at 900◦C	212.708	2.9637	0.31520
Chemical activation at 300◦C	164.854	1.9964	0.16456
Chemical activation at 600◦C	35.777	2.1614	0.038665
Chemical activation at 900◦C	48.643	2.6005	0.063248

3.1.2. 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 % begin 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 char.

3.1.3. SEM/EDAX analysis

The morphology and point element analysis of obtained particles from treating palm frond sample chemically then heated at 600°C for 4 h are obtained from SEM/EDAX analysis shown in **Figure 5**. Like wise for particles just physically treated at 600°C are shown in **Figure 6**. The Diopside mineral crystals appears in SEM picture of chemically treated palm frond sample shown in **Figure 5-a** as will as physically treated sample shown in **Figure 6-a**. The amorphous carbon appears in physically treated sample shown in **Figure 5-a**. These results are confirmed by element percentage of EDAX in **Figures 6-b, 5-b**, **Tables 4** and **5.** Comparing the EDAX analysis of chemically activated carbon **Figure 5-b** and **Table 4** with physically activated carbon **Figure 6-b** and **Table 5**, it is clear that the content of silicon element is lower in chemically treated catalyst due to dissolution and removal by strong alkaline KOH solution.

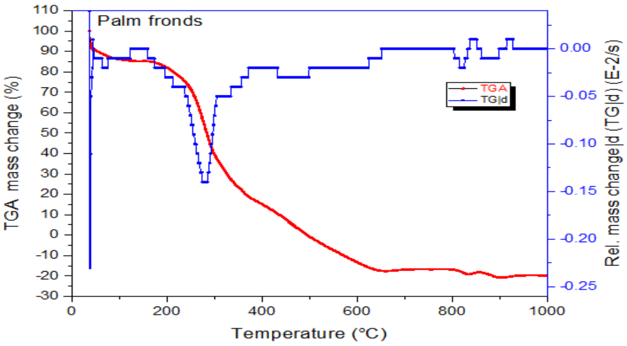
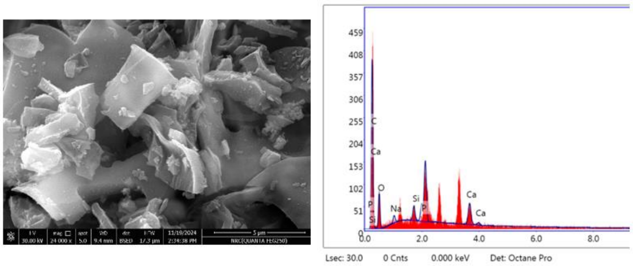
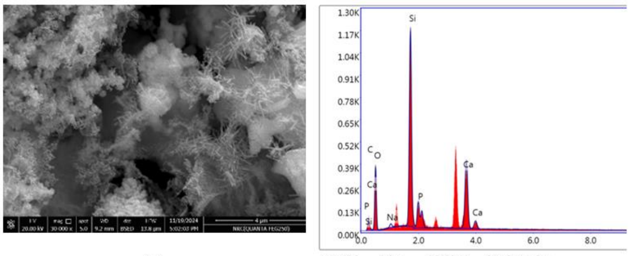


Figure 4: TGA for date palm fronds material.



(a) (b)

Figure 5. SEM(a)/EDAX(b) Analysis of chemically activated palm frond sample thermally treated at 600°C.



(a) (b)

Figure 6. SEM(a)/EDAX(b) Analysis of physically activated palm frond sample thermally treated at 600°C.

Table 4: Element percentage of chemically activated palm frond sample thermally treated at 600°C.

Element	Weight %	Atomic %	Net Int.	Error %	Kratio	Z	A	F
C K	63.42	74.14	102.68	12.48	0.2066	1.0294	0.3165	1.0000
O K	21.00	18.43	33.22	14.45	0.0280	0.9841	0.1355	1.0000
NaK	0.23	0.14	1.58	75.94	0.0009	0.8929	0.4428	1.0042
SiK	12.09	6.04	204.94	4.18	0.0957	0.8931	0.8827	1.0042
P K	1.02	0.46	12.87	7.64	0.0072	0.8576	0.8179	1.0057
CaK	2.24	0.79	21.60	17.39	0.0195	0.8423	1.0070	1.0223

Table 5. Element percentage of physically activated palm frond sample thermally treated at 600 °C.

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

3.1.4. XRD Analysis

Crystallographic and amorphous structure of all synthesized catalysts as will 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). The presence of sylvite mineral as presented in XRD of chemically activated catalyst **Figure 7**, may be due to the difficulty of complete washing after chemical treatment with KOH and subsequently HCl. The silicate diopside phase appears in both chemically treated catalyst **Figure 7** and physically treated palm frond **Figure 8**. This result is evidenced by SEM/EDAX analysis **Figures 5,6** and **Tables 6,7**.

3.1.5. Influence of different parameters

There is a constant relationship between the reaction parameters and the transesterification reaction. Finding the best transesterification process parameters is critical for optimizing biodiesel production. In this study, four separate variables pertaining to transesterification were investigated. These variables included the following: the ethanol-to-waste cooking oil molar ratio, which could be anywhere from 1:3 to 1:12, the catalyst concentration, which could be anywhere from 1 to 7 weight percent (wt.%) after physical activation at 900◦C, the reaction temperature, which could be anywhere from 60 to 90°C, and the duration of the reaction, which could be anywhere from one to five hours.

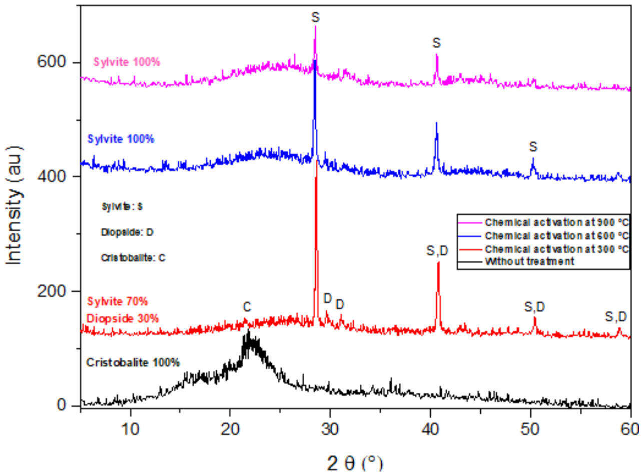


Figure 7: XRD analysis of chemically activated palm frond samples, without treatment and thermally treated at 300, 600 and 900°C.

3.1.6. Effect of reaction temperature on the biodiesel production

The acceleration of the reaction rate may be attributed to the augmented quantity of nucleophilic sites that arise from the movement and diffusion of reactant molecules caused by the elevation in reaction temperature. In order to get the ideal reaction temperature, the reactions were carried out within the temperature range of 60–90°C (**Figure 9**). A minimal yield of biodiesel was observed at lesser temperatures, while an increase in reaction temperature resulted in a greater yield. Due to the oil's decreased viscosity, an increase in reaction temperature typically results in an enhancement in biodiesel output. At a reaction temperature of 80◦C, 88% of the maximal biodiesel production occurred. The presence of mass transfer restrictions in the heterogeneous catalysts system has been confirmed. These constraints may be resolved by either using a co-solvent or conducting the reaction at elevated temperatures. Increasing the reaction temperature enhances the possibility of collisions between the reactants and the catalyst, hence reducing mass transfer restrictions and leading to maximal conversion.

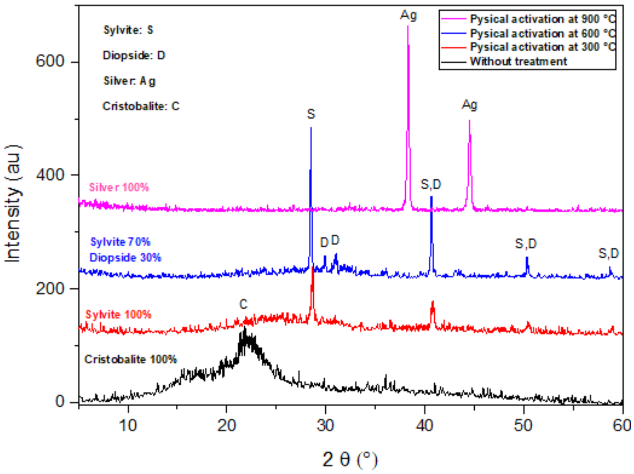


Figure 8: XRD analysis of physically activated palm frond samples, without treatment and thermally treated at 300, 600 and 900°C.

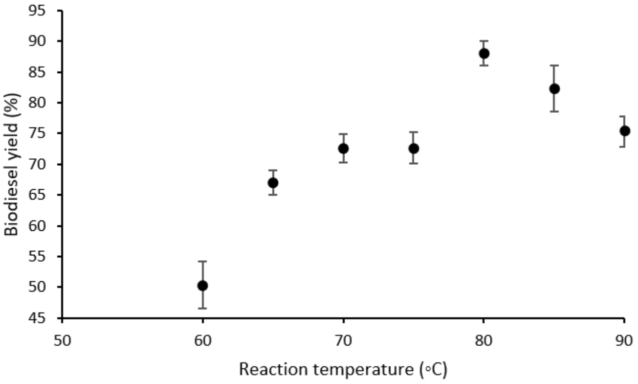


Figure 9. Effect of reaction temperature on biodiesel yield (%). Reaction conditions: catalyst loading 5 wt%, ethanol to date seed oil molar ratio 10:1, 3 h.

3.1.7. *Effect of ethanol-to-oil molar ratio*

Multiple research investigations have shown that the molar ratio between triglyceride and alcohol plays a crucial role in determining the amount of biodiesel that can be produced (Raiedhah *et al.* 2023). The transesterification process theoretically necessitates the combination of one mole of triglyceride with three moles of alcohol to yield one mole of glycerol and three moles of fatty acid ester. Therefore, reactions were conducted using ethanol to oil ratios ranging from 3:1 to 12:1 in order to determine the ideal ratio for obtaining the maximum quantity of ethanol as shown in **Figure 10**. The study revealed that the production of biodiesel had a positive correlation with the ethanol to oil ratio, with the yield steadily growing until reaching a ratio of 10:1 to reach the heist yield 88%. The reason for this is that the elevated ethanol to oil ratio enhances the production of ethoxy species, which actively participate in the reaction, resulting in a substantial conversion rate and a significant yield of biodiesel. Beyond this point, the output remained relatively constant as an overabundance of ethanol results in a rise in the soluble glycerol concentration within biodiesel. Furthermore, the elevated solute concentration of glycerol has the potential to hinder the biodiesel separation procedure.

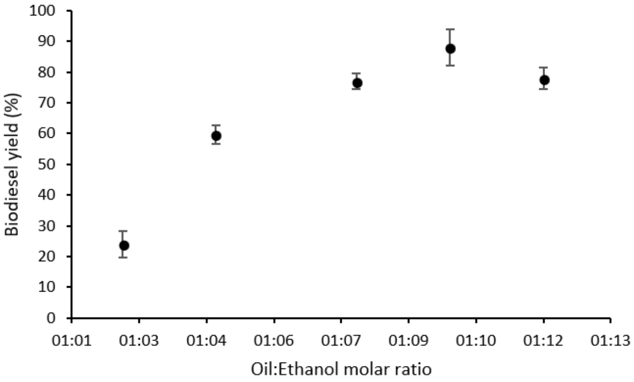


Figure 10. Effect of ethanol to waste cooking oil molar ratio on biodiesel yield (%). Reaction conditions: catalyst loading 5 wt%, reaction time 3 h, reaction temperature 80°C.

3.1.8. *Effect of the concentration of the catalyst*

The quantity of catalyst employed in the process also affects the production of biodiesel. The study primarily aimed to analyze the influence of catalyst concentration on the optimization process, given certain parameters such as an ethanol-to-oil molar ratio of 10:1, a reaction temperature of 80°C, a stirring speed of 800 rpm, and a reaction length of 180 minutes. The catalyst quantity in this work is systematically altered from 1 to 10 wt%. The findings for various catalyst levels are shown in **Figure 11**. Observations revealed that a poor yield was achieved at a low catalyst quantity, which subsequently rose as the catalyst amount grew. Therefore, the ideal catalyst quantity is found to be 5 wt%. Optimal biodiesel production is achieved when the catalyst concentration reaches 5 wt%, after which there is no further increase in yield. Remarkably, the use of a 5% activated carbon catalyst resulted in the greatest production of biodiesel, reaching an impressive 88% under the specified reaction conditions. Moreover, the reduction in biodiesel production when the loading exceeds 5% might perhaps be attributed to an excessive amount of heterogeneous catalysts present in the reaction vessel. 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. Hence, an enough quantity of catalyst is necessary to overcome the energy barrier necessary for the reaction to take place. Thus, in cases of poor yield, it is possible that the quantity of catalyst used was insufficient to start the mechanism. Conversely, when the catalyst quantity is increased, there is an adequately large number of active species that participate in the reaction, leading to a high production of biodiesel.

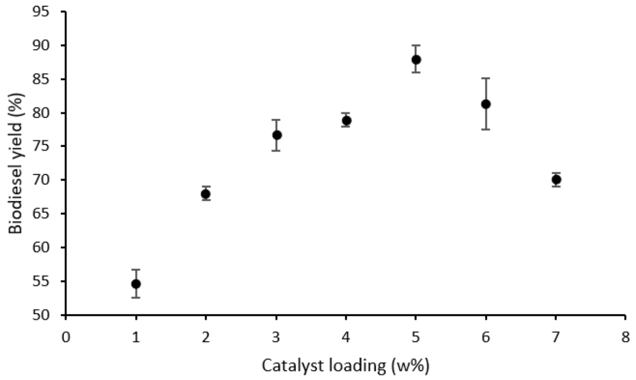


Figure 11. Effect of catalyst loading on biodiesel yield (%). Reaction conditions: ethanol to date seed oil ratio of 10:1, reaction time 3 h, and reaction temperature of 80°C.

3.1.9. Effect of reaction time

Considering the strong relationship between energy use and time in the biodiesel production process, it is crucial to determine a suitable reaction time. To examine the influence of reaction time on the percentage of biodiesel output, the length of the reaction was varied between 1 and 5 hours. The data in **Figure 12** showed a positive correlation between response time and biodiesel yield, with the highest 88% yield found at 3 hours. After this point, the yield rate remained constant. The increased yield seen with extended reaction times may be due to the enhanced interaction between the reactants, resulting in the formation of the final product. Conversely, when the reaction time is short, there is not enough time for the reactants to interact with one other. Further increase in the reaction time (more than 4 hours) result in slight decrease in the biodiesel yield, the decline in value may be ascribed to the reversible characteristic of the transesterification chemical reaction. Consequently, the percentage of yield drops above the ideal level. The observed phenomena may be ascribed to the progressive decrease in the number of active sites that are available over time, which happens due to catalyst deactivation.

3.1.10. Compare activity for all synthesized catalysts

Here it was compared the activity of all catalysts prepared by chemical and physical activation under the ideal reaction conditions chosen in previous experiments. Catalysts physically activated at 800°C are the most effective in producing biodiesel, and perhaps the main reason for this is the increase in its surface area and the stability of the catalyst during the reaction. For physically activated catalysts, the biodiesel yield is corelated to the active sites on the surface, through pore size or pore volume. Therefore, as the pore size of the catalyst particles increases, the yield increases. On the other hand, for chemically activated catalysts, surface basicity appears to be the Key factor in determining the catalytic process and yield.

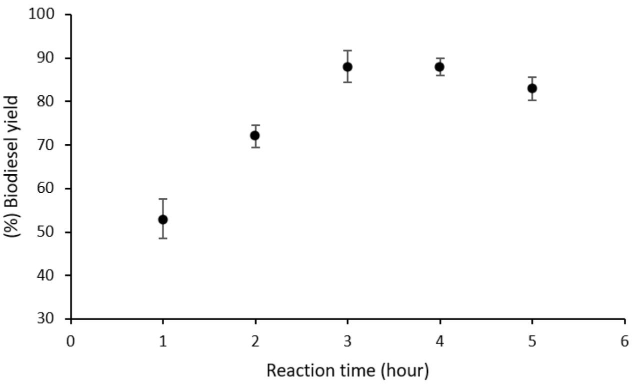


Figure 12. Effect of reaction time on yield of the biodiesel (%). Reaction conditions: catalyst loading 5 wt%, ethanol to date seed oil molar ratio 10:1, and reaction temperature 80°C.

Table 6. Compare activity for all synthesized catalysts

Catalyst	Biodiesel yield (%)
Catalyst physically activated at 300◦C	68
Catalyst physically activated at 600◦C	77
Catalyst physically activated at 900◦C	88
Catalyst chemically activated at 300◦C	71

Reaction conditions: catalyst loading 5 wt%, ethanol to date seed oil molar ratio 10:1, and reaction temperature 80°C, reaction time 3 hours.

Table 7. Reusability test of catalyst

Catalyst	Biodiesel yield (%)
Fresh	88
1st used	86
2nd used	84
3rd used	70

Reaction conditions: catalyst loading 5 wt%, ethanol to date seed oil molar ratio 10:1, and reaction temperature 80°C, reaction time 3 hours.

Table 8. Biodiesel's fuel qualities at ideal reaction circumstances.

Property	Unit	ASTM	Measured value for prepared biodiesel
Density	kg/m^3	860-894	890
Viscosity @40°C	mm^2/s	1.8-5.0	4.2
Acid number	mg KOH/g	≤ 0.45	0.6
Flash point	◦C	>120	126

3.1.11. Reusability of catalyst

Another notable advantage when employing a solid catalyst is its capacity to undergo regeneration and subsequent reuse. The catalyst produced in this study was recovered by filtering it using vacuum pressure and then washing it with methanol to evaluate its reusability. Following drying at 100°C, it was utilized in the subsequent cycle. The investigations pertaining to reusability are illustrated in **Table 7**. A marginal decline in activity was noted during the two runs of catalyst reuse. Therefore, it can be deduced that the catalyst possessed reusability in addition to its exceptional catalytic activity.

3.1.12. Characteristics of synthesized biodiesel

The American Society for Testing and Materials (ASTM) method was used to assess the fuel characteristics of the biodiesel produced after calcination under ideal reaction conditions to create activated carbon catalyst. The high-quality fuel characteristics of the produced biodiesel were found to be within the acceptable range as specified in the biodiesel standard (**Table 8**).

4. Conclusion

The generation of biodiesel from waste cooking oils may have environmental benefits by mitigating waste and offering an alternate energy source to petroleum fuels. Scientists are investigating renewable energy sources like biodiesel due to the present energy crisis and the rapid depletion of fossil fuel reserves. Our research aimed to examine the potential of waste cooking oil as a valuable resource for producing biodiesel using a sustainable catalyst generated from waste date palm fronds. The activated carbon catalyst derived from date palm fronds has great potential as a catalyst, yielding 88% biodiesel and maintaining its activity over several cycles without degradation. The optimal reaction conditions were determined as follows: temperature, 80°C; time, 3 hours; molar ratio of ethanol to oil, 10:1; and catalyst loading, 5 wt%. These conditions resulted in a biodiesel yield of 88 wt%. Waste cooking oil is an excellent source of biomass that may be used for renewable energy, contributing to the global goal of achieving zero pollution. Research has been conducted to examine the fuel properties of synthetic FAME and compare them to the specifications outlined in ASTM D 6571. Further investigation is necessary to explore cost estimations, engine efficiency, and strategies to reduce smoke emissions in the manufacturing of biofuels.

Acknowledgment

The authors are thankful to the Deanship of Graduate Studies and Scientific Research at Najran University for funding this work under Scholars Funding Program grant code (NU/FSP/SERC/13/254-1).

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