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Microwave Assisted Continuous Flow Transesterification of Waste Cooking Oil for Biodiesel Production

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ABSTRACT

Biodiesel is a major renewable energy source derived from vegetable oils. Modern techniques are now required to produce biofuels, particularly biodiesel, to improve the efficiency and sustainability of the process. This study focuses on continuous biodiesel production from waste cooking oil via microwave-assisted transesterification in a flow process. Calcium oxide prepared from willow leaf extract was used as a catalyst at concentrations ranging from 1% to 4%, with a microwave power of 20% of the total 800W and an irradiation time of 0.5-7 min. The oil-to-methanol ratio was 50wt.%-80wt.%, and the reaction temperature was 45-75 °C. The results revealed that the waste cooking oil can be converted to biodiesel with a yield of (93.43%) in 5 min. with a 70wt.% oil/methanol molar ratio and 3wt.% catalyst. Also, the results indicated that the production of high-quality biodiesel, in accordance with ASTM standards, was facilitated under all operational conditions. The results suggest that microwave heating is a viable approach for achieving high biodiesel yields with shorter reaction times, even in continuous reactions. This is attributed to reduced energy activation. This method is promising for industrial-scale production of high-quality biodiesel, with methanol as the preferred alcohol.

Introduction

Environmental and economic issues have drawn increased attention to renewable energy in recent years. The global economy has been significantly impacted by the substantial Fossil fuel depletion and rising pricing [1]. Many nations have developed alternative energy sources, such as wind, water, and biomass, that can be converted into biofuel to reduce fuel use, which is projected to increase by approximately 60% over the next 25 years [2, 3].

The challenge of finding alternative energy sources to replace diesel fuel is a critical concern in the quest for sustainability. Vegetable oils and animal fats serve as significant raw materials for alternative fuels in diesel engines [4]. Short-term engine tests have shown that they can be used directly without significant modifications. However, long-term use has faced many challenges, the most notable being the high viscosity and impurities in these biofuels, which lead to engine performance degradation, increased harmful emissions, and reduced engine lifespan [5].

The transesterification process was used to solve the technical issue of high viscosity in vegetable oils. This procedure depends on the reaction of one form of alcohol, typically methanol or ethanol, with triglycerides, the primary constituent of vegetable oils, in the presence of a chemical catalyst. Triglycerides progressively change into diglycerides,

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monoglycerides, and glycerol during these reactions. The main product used as biodiesel, fatty acid ester, is produced at each stage [6].

Biodiesel derived from renewable, biodegradable sources is an effective option for reducing carbon and sulfur oxide emissions, thereby improving combustion efficiency [7]. Biodiesel receives a lot of attention because it differs from petroleum diesel in that it is non-toxic, degrades quickly, has a higher cetane number, and has a higher flash point [8, 9].

In general, biodiesel is a mono-alkyl ester of long-chain fatty acids [10], so it is called fatty acid methyl ester (FAME). Typically, Biodiesel is derived from oils obtained from algae, animal fats, fungi, vegetable oils, or alternative sources such as used cooking oil [11]. The cost of raw ingredients used in biodiesel production accounts for more than 70% of total costs [12, 13]. Utilizing waste oils and fats in homes and restaurants lowers the cost of producing biodiesel while simultaneously solving the problem of trash disposal because oil is an essential raw material used [14].

Depending on the chemicals involved, transesterification reactions can employ either homogeneous (acid or base) or heterogeneous (acid or base) catalysts. Heterogeneous catalysts are typically solid and, in a phase, different from the reaction mixture, whereas homogeneous catalysts are liquids in the reaction phase. The drawbacks of homogeneous catalysts include their inability to be separated from the product, their tendency to form unstable emulsions, and the production of copious amounts of wastewater. Apart from that, the heterogeneous catalysts are environmentally safe because they are produced via green synthesis and don't corrode. Their main benefit is that they are inexpensive and reusable [15, 16].

Acidic catalysts are appropriate for the reaction if the FFA ratio in the oil and water is high; however, this process requires a long reaction time, a high catalyst concentration, and a high alcohol/oil ratio. Because the catalyst is acidic, it causes corrosion in the equipment [17]. To address these issues, basic heterogeneous catalysts will be used because they are inexpensive, simple to separate from the product, and extremely effective [18]. Oxides, including calcium oxide, are heterogeneous catalysts widely employed in the synthesis of biodiesel due to their unique properties, such as low cost, moderate reaction conditions, and short reaction times, owing to their high effectiveness [19].

A catalyst is also required for the transesterification process to lower the energy activation and drive the reaction [20]. Using fallen willow leaves, which are considered waste, reduces the total cost of producing biodiesel.

Microwaves are a form of electromagnetic radiation, with frequencies between infrared and radio waves, specifically 0.3-300 gigahertz, and wavelengths of 0.01-1 meter. To avoid interference with communication and mobile phone frequencies, most home microwave ovens operate at 2.45 gigahertz. In industrial applications, common frequencies used are 915 and 2450 megahertz. In laboratories and chemical experiments, microwave devices are often operated at 2450 megahertz and a wavelength of 12.24 centimeters [21, 22].

Microwave energy is used in a variety of applications because it speeds up chemical reactions by interacting with polar molecules [23]. Heating in the microwave is fast and homogeneous because microwaves transfer heat via wave absorption, so the sample's temperature is higher than the reactor's surface, and heat transfer in the microwave does not occur through the surface [2, 24]. Unlike traditional methods, Heat moves from the container's exterior to the items inside. via thermal conduction (convention). This method will result in a significant energy loss because the rise in temperature will cause heat to move from the sample's exterior to its interior, and the heating will be inhomogeneous due to thermal conductivity, specific heat, and density [25]. Microwave radiation offers many benefits over traditional methods in the manufacture of biodiesel, including reduced reaction time (fast reactions) due to radiation energy, clean energy that does not produce waste, greater efficiency than traditional methods, which provides the best way to manufacture biodiesel, safety, and a better way to separate products [26].

For batch transesterification to produce biodiesel, you need a large reactor and a high methanol-to-oil ratio. In addition, the process must be water-free, and excess glycerin and catalyst must be removed [27]. Therefore, a system has been developed that can address these problems using continuous reactor technology. This system has proven to be efficient and effective. The continuous-flow reactor has many advantages, including reducing the methanol-to-oil ratio, reducing reaction time, and achieving high biodiesel yields [28, 29]. The most important advantage of this system is that it can process large quantities of reactants while simultaneously withdrawing samples without stopping the process. In addition, it mixes the materials as a result of its flow through the tubes, and it cost is low due to the small reactor size [30]. The continuous flow system also operates under stable conditions, producing stable products; the methyl ester concentration is also stable [31]. The main purpose of using the continuous flow system instead of the batch system in industry is safety. Because a relatively large reactor inside the microwave, under pressure, can lead to many problems that cause malfunctions and disasters. Therefore, to avoid these problems, continuous-flow reactors are used in the microwave because they are relatively safe and compatible with many reagents [30].

The kinetics of the transesterification reaction are crucial for establishing optimal conditions to enhance biofuel (biodiesel) production. Kinetic models offer precise insights into reaction behavior by accounting for mass and heat transfer effects, along with thermodynamic equilibrium, thereby enhancing process efficiency and improving outcome predictions [32].

This research aimed to assess the feasibility of continuous microwave transesterification of waste cooking oil to enhance biodiesel production. Also, the study highlights the impact of catalyst concentration, microwave irradiation reaction time, reaction temperature, and oil-to-methanol molar ratio on biodiesel yield and determines the optimal conditions for biodiesel production. Finally, the reaction kinetics and the activation energy were also investigated.

Materials and Methods

Materials

The experiments used a wide variety of pure chemicals, including sodium hydroxide, calcium nitrate tetrahydrate from India, Thomas Baker, and phenolphthalein reagent from BDH Chemicals Ltd. in England. Thomas Baker and nearly one hundred percent pure methanol with a purity of 99.8 percent, which originated from Chem-lab NV in Belgium. Additionally, ethanol was purchased from RCI Labscan with a minimal analysis of 99.9 percent. Additionally, high-purity deionized water and. The cooking oil used came from local businesses.

Pretreatment of Used Cooking Oil

To eliminate any impurities and food particles, the waste cooking oil is filtered through a strainer. The oil was titrated with 0.1 N KOH to measure free fatty acid (FFA) content. The oil was mixed with 50 ml of ethanol alcohol and a few drops of phenolphthalein indicator. The oil weighed 4g. The FFA% in the oil was determined to be 2.4%, and the mixture was titrated with 0.1 N KOH until a pink color was observed. The water content was determined by placing a 20 g sample of oil in an oven at 110 °C for 2 hours. The water content was determined to be 0.051% by measuring weight losses.

CaO Nano-catalyst Preparation and Characterization

After being cleaned and washed, the willow leaves were dried for six hours at 100 °C. After that, they were ground into a fine powder by crushing them. The powdered leaves were boiled for 30 minutes at 60 °C in deionized water. To obtain an aqueous extraction, it is then filtered and cooled. After heating the aqueous extract to 55 °C, calcium nitrate tetrahydrate and NaOH were added. The formation of a yellow paste indicated the completion of the reaction, and the process was then stopped. A CaO nano catalyst was created by calcining the catalyst at 600°C in a muffle furnace for two hours. Initially, the extract was analyzed using FT-IR and EDX techniques to identify its active components. FT-IR results revealed distinct absorption peaks indicative of calcium-containing compounds, while EDX of the extract showed a high calcium content (10%), indicating that the raw material is rich in calcium and can be converted into efficient calcium oxide (CaO). Next, a CaO nanocatalyst was prepared using the method described in the literature. The catalyst was analyzed by XRF, which revealed a calcium oxide content of 82.542%, indicating a high degree of purity. The catalyst was characterized using BET, FT-IR, and SEM-EDX techniques. EDX results for the CaO catalyst showed that calcium accounted for 49.31% and oxygen for 45.08%, the expected proportions for calcium oxide formation. BET analysis also showed that the catalyst had a specific surface area of 12.097 m^2/g, a pore volume of 0.023711 cm^3/g, and an average pore diameter of 2–10 nm. The crystal structure was confirmed using XRD analysis.

Trans-esterification Reaction in Continuous Reactor

Experiments were carried out continuously in a laboratory microwave of type WB20230745, manufactured by Gongyi Yuhua Instrument Co., Ltd., with a power output ranging from 80 to 800 watts. Fig. 1 illustrates the experimental setup. In this study, a three-neck glass flask was used as the source for preparing the reaction mixture. A mixture of used oil, methanol, and catalyst was placed inside the flask. A condenser was installed on one neck to prevent the methanol from evaporating and returning it to the system. A thermometer was installed in the other neck to monitor the mixture's temperature during preparation. A pump was used to draw the mixture from the flask and pump it into a Teflon tubular reactor constructed from a 2-meter-long tube with an inner diameter of 8 mm. The reaction proceeded as the mixture flowed through the tube under controlled temperature conditions. A sensor was installed at the end of the tube to measure the temperature of the outgoing mixture to ensure stable reaction conditions. The microwave heating device was also inspected before the experiments to ensure no radiation leakage, ensuring safety and accurate results. The Teflon tube was wound into a coil in the microwave and connected to the outflow separating funnel.

Methanol (based on oil weight) was used with (30 g) of waste cooking oil and CaO nano catalyst; the resulting solution was then put into a three-necked glass flask and heated to reaction temperature. To avoid phase separation, the reactants were stirred at 700 rpm using a magnetic stirrer placed in the vessel. The irradiation time was then adjusted by continuously pumping the reactants into the Teflon tubular reactor at varying flow rates. The experiment's independent variables were the amount of catalyst and the methanol-to-oil ratio. The weight ratio of methanol to oil varies between 50%, 60%, 70%, and 80%. The effect of catalyst concentration was also investigated at 1%, 2%, 3%, and 4% of the waste cooking oil weight. The microwave radiation power was set to 20% of the total device power, with different reaction times (0, 0.5, 1, 2, 3, 4, 5, and 7 minutes) and reaction temperatures (45°C, 55°C, 65°C, 75°C). After the reaction was complete, the mixture was left to rest overnight, revealing three distinct layers: a bottom layer with catalyst remnants, a middle layer of glycerin, and a top layer of biodiesel. Centrifugation was used to separate the fuel layer from the catalyst. The mixture was then dried in an oven at 65°C for 24 hours to remove any remaining methanol. Following that, the conversion ratio to biodiesel was calculated using Eq. 1.

$$yield\% = (weight\ of\ biodiesel\ )/(weight\ of\ waste\ cooking\ oil\ ) * 100\% \qquad (1)$$

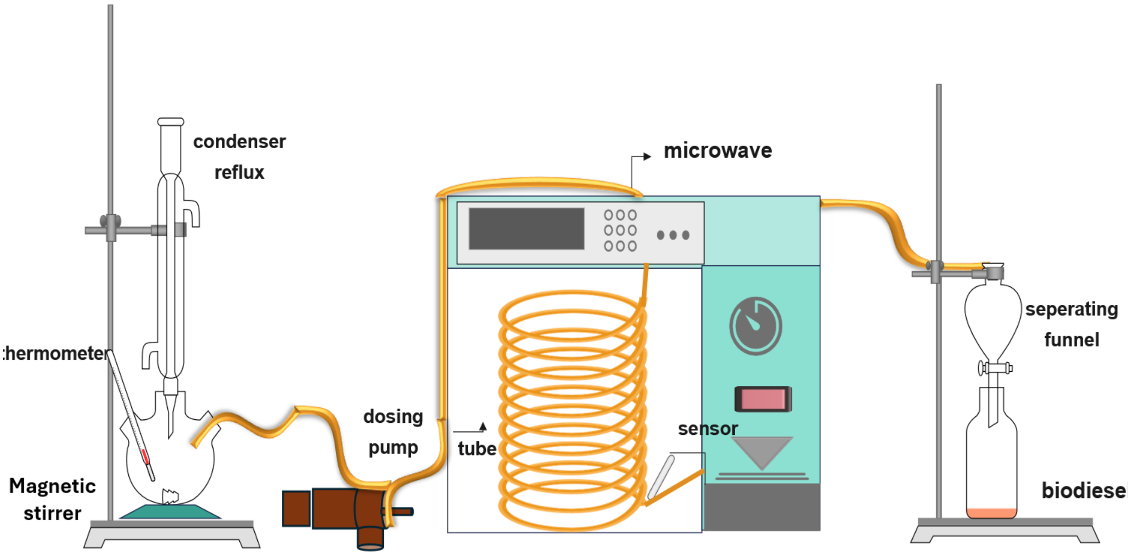


Fig. 1. Scheme diagram of the continuous process in microwave of biodiesel production

Kinetic Study

The total equation of the product fatty acid methyl ester from triglyceride is shown in Eq. 2.

$$TG + 3ROH \overset{\Delta catalyst}{\Longleftrightarrow} GL + 3RCOOR \qquad (2)$$

Due to excess methanol, the transesterification reaction was predicted to have pseudo-first-order kinetics. When the amount of a reactant (methanol) exceeds that of another reactant (TG), the second reaction kinetics become pseudo-first-order, with negligible reversible reactions. The reaction rate (r) can be calculated using Eq. 3 [33].

$$-r_A = \frac{d_{CA}}{dt} = kC_A \qquad (3)$$

In Eq. 3, C_A, k, and t represent TG concentration, rate constant, and reaction time, respectively. Rate constants were calculated using Eq. 4 and observed at various reaction times.

$$-\ln(1 - X_A) = kt \qquad (4)$$

In the above Eq. 4, X represents the product conversion at time t. The activation energy (Ea) was calculated using Arrhenius' equation (Eq. 5).

$$lnk = -\frac{E_a}{RT} + lnA \qquad (5)$$

T represents the reaction temperature, while A and R are the pre-exponential factors $8.314 * 10^{-3} \frac{KJ}{K.mole}$.

To calculate the kinetics, the microwave's temperature increased at 5-minute intervals from 318.15 K (45 °C) to 348.15 K (75 °C).

Results and Discussion

The Parameters' Effect on Biodiesel Product

Methanol to Oil Ratio Effect

The molar ratio of alcohol to WCO is a key factor in determining methyl ester yield. The stoichiometric molar ratio of WCO to methanol is typically 1:3, but this does not always result in a complete reaction. To achieve higher yields of methyl esters, a higher molar ratio is needed to reach equilibrium and complete the reaction [34]. This study examined how different methanol-to-oil ratios (50 wt.%, 60 wt.%, 70 wt.%, and 80 wt.%) affect biodiesel yield. The following parameters were kept constant: temperature at 65°C, catalyst loading of 3 wt.%, microwave power of 20%, and reaction time of 5 minutes. The molar ratio of 70wt.% resulted in the highest biodiesel yield reached 93.43), as shown in Fig. 2a. However, increasing the methanol ratio beyond this limit resulted in no further yield improvement, indicating the achievement of a quasi-stable equilibrium [26].

Catalyst Loading Effect

The WCO conversion and yield are significantly influenced by the catalyst loading. For this set of experiments, the temperature was maintained at 65 °C, the oil-to-methanol ratio was maintained at 70 wt.%, the reaction time was 5 minutes, and the microwave power was 20%. The CaO catalyst was adjusted from 1 to 4 wt.%. As illustrated in Fig. 2b, the catalyst loading increased from 1 to 3wt.%, resulting in a rise in yield from 79.73 to 93.43 wt.%. Nevertheless, the biodiesel percentage was reduced due to increased soap formation, which was caused by an additional increase in catalyst concentration (from 1 to 4 wt.%). Consequently, the highest biodiesel yield was achieved at 3 wt.%, yielding 93.433%.

Temperature Effect

The influence of temperature was investigated by adjusting the reaction temperature between 45 °C and 75 °C while keeping all other variables constant (3 wt.% catalyst concentration, 70 wt.% methanol-to-oil ratio, 20% microwave power, and 5 minutes of reaction time). Temperature influences yield, as shown in Fig. 2d. Yield increased from 65.57% to 94.16% as temperature rose from 45 to 65 °C. Nevertheless, successive increases in temperature led to a drop in yield. The cause is the evaporation of methanol at temperatures above 64.7 °C (its boiling point), resulting in a 75 °C loss in yield. This is consistent with the findings of Gimbun et al (2013) [35]. Methanol generates bubbles in the solution that exceed its boiling point, which can impede the reaction, as per Wang et al. [36]. Additionally, saponification of glycerides is accelerated by higher temperatures, leading to increased glycerol production. The outcome of this investigation is in accordance with the findings of prior investigations [37, 38].

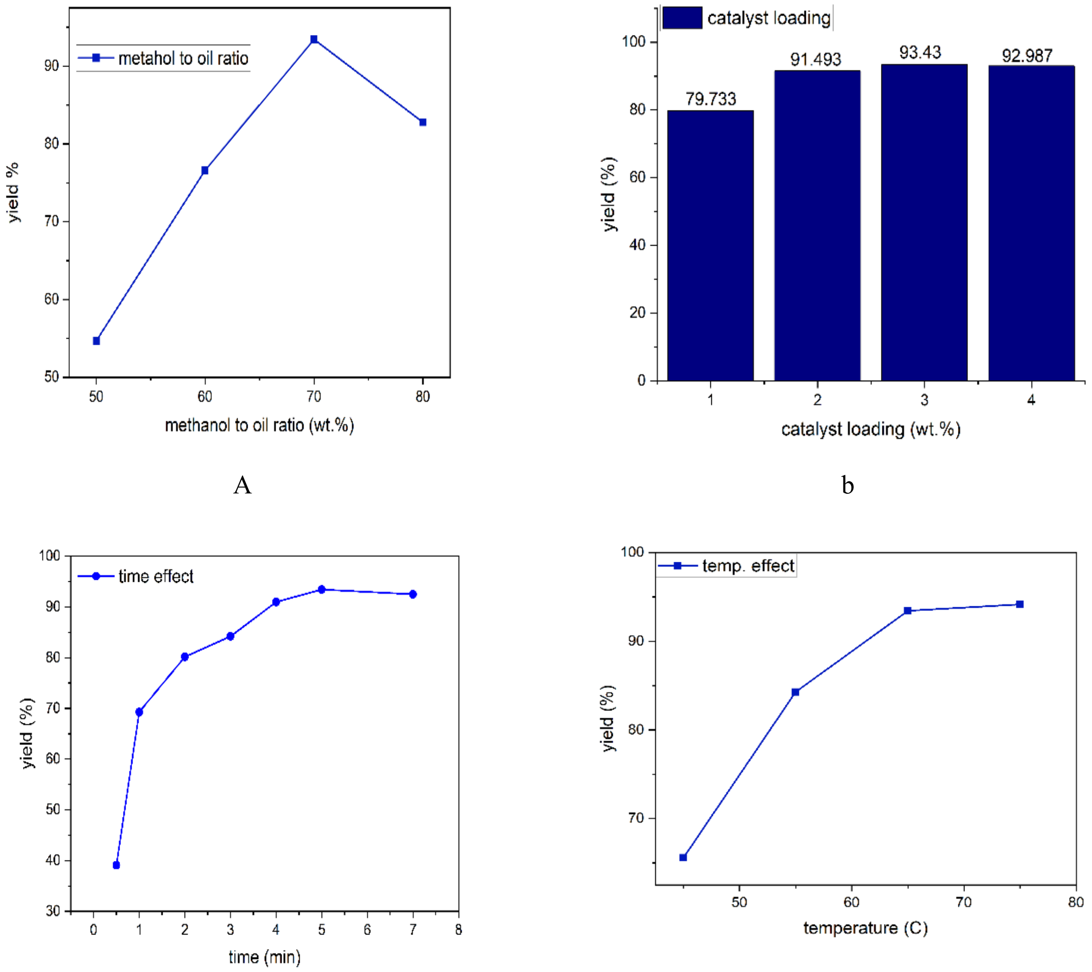
Time Effect

To explore the effect of reaction time on the production of biodiesel, the catalyst concentration was set at 3 wt.%, the methanol-to-oil ratio at 70 wt.%, the temperature at 65 °C, and the microwave irradiation power at 20%, while maintaining the other reaction conditions constant. The reaction time varied from 0.5 to 7 minutes. The results, as illustrated in Fig. 2c, demonstrate that the biodiesel yield improved substantially as the reaction time increased from 0.5 to 5 minutes. The maximum value was 93.433%. Nevertheless, indicators declined when the reaction time exceeded 5 minutes.

This decrease is associated with the potential for esters to hydrolyze, leading to the formation of soap rather than continuing the conversion [39]. This process has a detrimental impact on the reaction's efficiency. The optimal reaction time for achieving the highest yield and conversion rate in subsequent reactions in this study was determined to be 5 minutes.

Improvement Rate of Reaction

In this part of the study, a narrow-channel tubular reactor measuring (2 meters) in length and (5 mm) in internal diameter was used, which is smaller in diameter compared to the reactor used in previous experiments. The experiment was conducted under the same optimal operating conditions that yielded the best results in the larger reactor, including methanol-to-oil ratio, catalyst concentration, temperature, and residence time. Although the narrow-channel reactor offers theoretical advantages in terms of improved diffusion mixing and better control of heat and mass transfer, the results showed a lower yield (86%) compared to the larger reactor (93.43%), even under constant operating conditions, including 3 wt.% catalyst concentration, 70 wt.% methanol to oil ratio, 65 °C reaction temperature, and 20% microwave power [40]. This decrease is attributed to several engineering and behavioral factors associated with the new reactor design, most notably exceeding optimal conditions in the engineering context of a small reactor. Even if the temperature and residence time are identical, the flow distribution within the narrow channel may be irregular, leading to localized stagnation zones and negatively impacting reaction efficiency. Excessive transfer rates: In small, narrow-channel reactors, the mass and heat transfer rates are very high. This can lead to rapid product separation within the reactor before the reaction is complete, especially since the conversion process relies on continuous contact between the oil and methanol. Difference in flow regime: A smaller reactor may tend toward a suboptimal flow regime (e.g., inhomogeneous plug flow or localized mixing) compared to a larger reactor, which provides greater homogeneity, thereby impairing reaction efficiency. Premature product separation: The narrow design contributed to rapid separation of methanol or glycerol from the reaction medium, leading to the reaction being interrupted before reaching maximum conversion. Thus, it is concluded that the optimal conditions achieved in a larger reactor cannot be directly transferred to a smaller reactor without modification, as the reactor's engineering characteristics play a crucial role in determining reaction efficiency.



A b

C d

Fig. 2. The parameters' effect on transesterification reaction: (a) methanol to oil ratio effect, (b) catalyst loading effect, (c) time reaction effect, and (d) temperature reaction effect

Properties of Biodiesel Product

The basic characteristics of the biodiesel produced were studied using a microwave-assisted transesterification process (continuous system), with the results summarized and compared to the ASTM D6751-09 standard and EN 14214, as shown in Table 1.

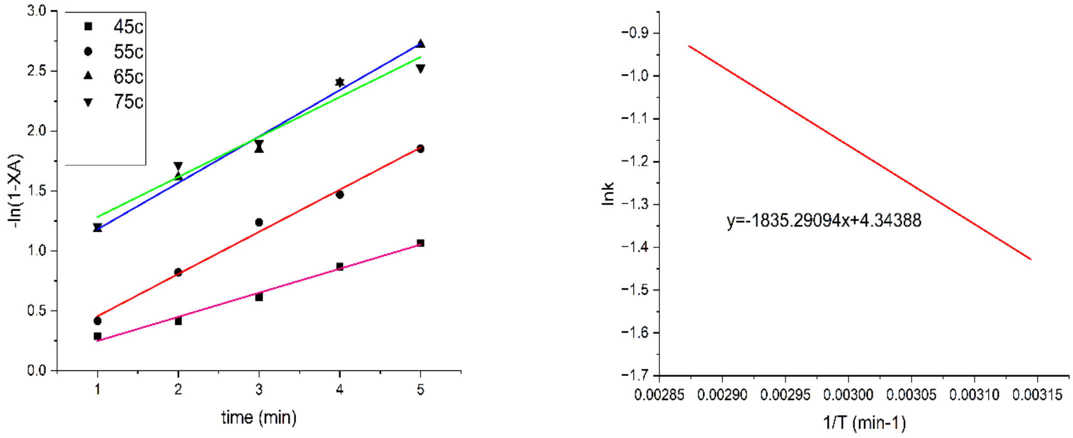
The findings indicated that density, viscosity, and flash point are within the permissible limits set by the standards, indicating the suitability of this biodiesel type for use in diesel engines. The biodiesel produced was determined to have a viscosity of 5.094 mm^2/s. The result obtained agreed with the value of 5.2 mm^2/s reported by [41] in other studies on biodiesel extracted from used cooking oil. The produced biodiesel was estimated to have a flash temperature of 93.5 °C, comparable to diesel, and rendering it safe to handle. Previous studies have reported this flash point, with [41] reporting a flash point of 96 °C, compared to 60 °C for mineral diesel. The density of biodiesel produced from waste cooking oil was 0.908, which is approximately equal to the maximum reported in English studies.

Table 1. Physical properties of the biodiesel product

Property	Biodiesel product	ASTM D 6751	EN 14214
Viscosity (mm^2/s)	5.094	1.9-6	3.5-5
Density (g/cm^3)	0.9128	0.87-0.89	0.86-0.90
Flash point (°C)	93.5	130 minimum	>101

Kinetic Study and Activation Energy

The kinetics of the transesterification reaction were investigated to determine the influence of varying temperatures at different reaction times. The studies were conducted under optimal operating conditions, with temperatures of 45, 55, 65, and 75°C and time intervals of 1, 2, 3, 4, and 25 minutes. Fig. 3a shows a linear relationship between $-\ln(1-X)$ and time, showing the reaction follows pseudo-first-order kinetic models based on Eq. 4. The activation energy (Ea.) was estimated using the rate constants obtained from the experimental data and the Arrhenius equation (Eq. 5). Fig. 3b shows a graph between ln(k) and 1/T. The slope of the line (−Ea./R) was used to compute Ea., and the intercept on the ln(k) axis was used to derive the pre-exponential component (A). The activation energy was 15 kJ/mol, which is less than common for transesterification processes (24.7-84.1 kJ/mol).



A b

Fig. 3. (a) linear relationship between $-\ln(1-X)$ and time, and (b) graph between ln(k) and 1/T

Comparison between Batch and Continuous Processes under the Same Conditions

A comparison was conducted between batch and continuous processes using the same feedstock and identical operating conditions for temperature, catalyst type and quantity, methanol-to-oil ratio, and power, to evaluate the continuous process's performance (Table 2). The results showed that both systems achieved good conversion rates, but the continuous process demonstrated a clear advantage in reducing the reaction time required to achieve maximum conversion. The continuous flow of reactants improved reaction efficiency by enhancing mixing and maintaining temperature stability, thereby accelerating the conversion process. Therefore, the continuous process can be considered a more efficient option for biofuel production compared to the batch process, especially in applications that require reduced operating time without compromising conversion efficiency.

Table 2. Comparative study between batch and continuous processes

Optimum condition	Methanol to oil ratio (wt.%)	Reaction Time (min)	Temperature (°C)	Catalyst loading (wt.%)	Power %	Yield
Batch	70	20	65	3	20	94.606
Continuous	70	5	65	3	20	93.43

Comparative between Other Studies

Although the biodiesel conversion rate in this study was lower than in prior studies, what sets it apart is the use of a new natural catalyst, a CaO nanocatalyst derived from willow leaf extract, which was used for the first time in this context. This catalyst is inexpensive, locally available, and ecologically friendly, making it a more sustainable option than traditional industrial catalysts. Previous experiments, such as those using example: NaOH, achieved conversion rates of 95% under operational settings such as a temperature of 60 °C and a reaction time of 0.5 minutes, using waste soyabean oil as a feedstock [42]. While in other studies that use NaOH as a Catalyst achieved a conversion rate of 99.04% in 21.04 minutes as a result of using coconut oil as feedstock [43]. It was also found that utilizing KOH and NaOH as a catalyst at a similar concentration (1wt.%) and different feed stocks (used vegetable oil and used palm oil) resulted in a high yield in both catalysts, 98.9% and 97%, although using a different alcohol to oil ratio [44, 45]. In a prior study, a $NaOCH_3$ catalyst was employed with Jatropha oil, and under operational conditions including a reaction period of 0.5, a 96.5% conversion rate was attained [5]. In this investigation, CaO was combined under various conditions: 5 minutes reaction time, 65 °C reaction temperature, and a 70 wt.% methanol-to-oil ratio, resulting in a conversion rate of 93.43%. The results have been compared with the previous studies (Table 3).

The efficiency discrepancy can be explained by a variety of factors, including the natural catalyst's features, such as surface area, catalytic activity, and chemical composition. However, the findings represent a promising first step toward developing alternative, more sustainable natural catalysts. This study indicates the potential use of atypical catalysts for biodiesel production, paving the way for further performance improvements through structural modifications or operating conditions.

Table 3. The comparison between different catalysts applied in a continuous process

Process type	Feed stock	Alcohol to oil ratio	Temperature (°C)	Reaction time (min)	Catalyst	Yield %	Ref.
Continuous	Jatropha oil	1:6	-------	0.5	$NaOCH_3$ (1.0 wt.%)	96.5	[5]
Continuous	Used vegetable oil	1:6	--------	1	KOH (1.0wt.%)	98.9	[44]
Continuous	Coconut oil	1:7 methanol	65	21.04	NaOH (1 wt.%)	99.04	[43]
Continuous	Used palm oil	1:12 ethanol	50	0.5	NaOH (1 wt.%)	97	[45]
Continuous	Waste soybean oil	1:6 methanol	60	0.5	NaOH (1 wt.%)	95	[42]
Continuous	Waste cooking oil	70wt.%	65	5	CaO (3 wt%)	93.43	Current study

Conclusion

A continuous process for biodiesel production has been developed using a narrow-channel reactor unit in a microwave to study the effects of Cao nano-catalyst concentration derived from fallen willow extract, temperature, reaction duration, and the methanol-to-oil ratio. The results showed that increasing the catalyst concentration from 1% to 3% by weight significantly increased the biodiesel yield. Additionally, increasing the reaction time improved reaction efficiency. The specialized continuous microwave-assisted reactor developed for this study successfully converts waste cooking oil into biodiesel. The maximum yield (93.43%) was obtained at 65 °C, an oil-to-methanol molar ratio of 70 wt.%, and a catalyst loading of 3 wt.%. CaO catalyst, and a reaction time of 5 minutes. The biodiesel generated in this study meets the specifications outlined in ASTM D6751. Additionally, the design used enhanced the separation of biodiesel from glycerin, thanks to the reliance on gravitational separation within the reaction tube.

Nomenclature

Roman symbols

Symbols	***Description***	***Unit***
A	Pre-exponential factor	mol/g.min
dC_A/dt	Rate of change of concentration	mol/g.min
$Ea.$	Activation energy	KJ/mol
K	Rate constant	1/min
R	Gas constant	J/mol.K
r_A	Rate of reaction	mol/l.min
T	Reaction time	min
T	Reaction temperature	°C
X_A	Conversion of A	%

Abbreviations

Symbol	***Description***
ASTM	American Society of Testing and Materials
BET	Brunauer-Emmett-Teller
CaO	Calcium oxide
EDX	Energy Dispersive X-Ray Spectroscopy
EN	European Norm
FAME	Fatty acid methyl ester
FFA	Free fatty acid
FTIR	Fourier Transform Infrared Spectroscopy
GL	Glycerol
KOH	Potassium hydroxide
$NaCH_3$	Sodium methoxide
NaOH	Sodium hydroxide
RCOOR	Ester (FAME)
ROH	Alcohol
SEM-EDX	Scanning Electron Microscopy–Energy Dispersive X-Ray Spectroscopy
TG	Triglycerides
XRD	X-Ray Diffraction
WCO	Waste cooking oil

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