

## Research Article

# Biodiesel Yield and Conversion Percentage from Waste Frying Oil Using Fish Shell at Elmina as a Heterogeneous Catalyst and the Kinetics of the Reaction

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In this study, biodiesel was produced from waste frying oil as feedstock with a calcined fish shell under a heterogeneous solid base catalyst. The process of transesterification was done by varying methanol-to-oil molar ratio, catalyst amount, reaction temperature, and reaction time. The heterogeneous catalyst was prepared stepwise as follows: washing and drying the fish shell for 24 hours at 120°C in an oven, then crushing to form powder having been pound for 2-3 minutes in an agate mortar cleaned with nitric acid (6 N). The powdered fish shell was then calcined at 950°C for 4 hours using a muffle furnace. The calcined catalyst was subsequently kept in a desiccator to avoid encountering moisture. The prepared catalyst was then characterized using XRD and FT-IR. The optimum biodiesel yield of 99.58% was obtained under methanol-to-oil ratio of 10:1, catalyst amount of 3.0 wt%, reaction temperature of 60°C, and reaction time of 8 hours with mass transfer control of 600 rpm. The optimum rate of constant of 0.164 L/mol·S<sup>-1</sup> was determined using the second-order kinetics model. The constant rate of reaction indicated that the forward reaction is crucial for the reaction. The properties of biodiesel produced conformed with those of the international standard using ASTM D6751.

## 1. Introduction

Biodiesel is produced by transesterifying long-chain fatty acids sourced from animal fats and vegetable oils with aliphatic alcohols (methanol or ethanol) in the presence of a suitable catalyst to make esters of long-chain fatty acids and glycerol [1, 2]. Transesterification is the most prevalent way of synthesizing biodiesel. The type of catalyst to be employed is determined by the amount of free fatty acid (FFA) contained in the feedstock, which is either acid or base. In the case where values of free fatty acid are more than 2 mg-KOH/g, the recommended process is a two-stage process [3, 4] or neutralization of oil using an alkali before transesterification. The method of two-stage biodiesel production begins with acid-catalyzed esterification, in which free fatty acid is changed to esters by acid, followed by base-catalyzed transesterification, comparatively. The commonly used mineral acids are hydrochloric or sulphonic and sulphuric

acids. Sodium hydroxide is the base catalyst that is widely used due to it being cheap and safe in handling as compared to potassium hydroxide with methanol as the ideal alcohol. Methanol and ethanol are the most commonly used alcohols for biodiesel production, with methanol producing a fatty acid methyl ester mixture (FAME) and ethanol producing a fatty acid ethyl ester mixture (FAEE) [4]. Methanol is affordable compared to other alcohols and produces fuel with a short chain with required qualities [5, 6].

Feedstock has a role in the high cost of biodiesel manufacturing [7]. Waste frying oil (WFO) used as an alternative to virgin oil for producing biodiesel is one method of reducing the cost, considering the estimate to be half the price of pure oil [3]. This is because waste frying oil, non-edible oil [8], insects [9], and waste animal fat (cow, pig, and sheep) are all inexpensive and readily available at restaurants. After each usage, unused cooking oil is disposed of. Palm oil, soybean oil, sesame oil, and sunflower oil are

among the waste frying oils available [3, 10]. The use of waste cooking oil for biodiesel production provides an alternative to disposing of waste cooking oil into the environment which brings significant environmental benefits [11].

Designing a suitable reactor that can be used for biodiesel production requires the study of the kinetics and reaction rate constant. Several studies have been carried out on transesterification and its kinetics. Typically, Darnoko and Cherryan [12] looked at the kinetics that involved transesterification of palm oil through the application of potassium hydroxide as the base catalyst. The conversion process in this case was described as second-order. Leevijit et al. [13] also used sodium hydroxide as a catalyst to transesterify palm oil with methanol. Both of their studies used thin layer chromatography/flame ionization detectors to determine the % weight of the reaction mixture and reported second-order kinetics. Similar works on the kinetics of *Brassica carinata* and sunflower oils methanolysis by Vicente et al. [14] found that initial mass transfer affected the kinetics with second-order, indicating that the rate is kinetically regulated. The initial mass conversion kinetics was accompanied by a chemical reaction that held second-order rates, according to Nouredini and Zhu [15] in the investigation of the kinetics of soybean oil transesterification with methanol. Stamenković et al. [16] used basic kinetics models to investigate the kinetics of sunflower oil methanolysis at low temperatures, reporting sigmoidal kinetics in which mass transfer initially drove the kinetics and chemical reaction controlled the region. According to Bambase et al. [17], an agitation speed of 400–600 rpm is necessary to successfully decrease the mass transfer area in hydroxide-catalyzed methanolysis of crude sunflower oil for biodiesel.

The current work employed a heterogeneous catalyst prepared from the fish shell for transesterification of waste frying oil. The reaction conditions were optimized by varying the methanol/oil molar ratio, reaction time, catalyst quantity, and temperature. The reaction rate constant ( $k$ ) and second-order kinetics transesterification were measured as part of the kinetics study. Using EN 14214 and ASTM D 6751 standards, the characteristics of the fuel were determined and compared.

## 2. Methodology

Waste frying oil was gathered from a small restaurant in Cape Coast. The crude oil obtained was degummed using water at 70°C–80°C for 15 minutes. To minimize the free fatty acids, the degummed oil was neutralized using analytical-grade sodium hydroxide. The School of Biological Science Laboratory provided gas, chromatography, grade chemicals, and biodiesel standards. The remainder of the chemicals used was of analytical grade. Elmina fish market in Cape Coast was used to collect the fish shells.

**2.1. Catalyst Preparation.** Washing was done initially on the fish shell with tap water to aid in the removal of sand and the flesh that adhered to the shells. The clean and dry shells were then crushed for 2–3 minutes in an agate mortar that had

been cleaned with nitric acid (6 N) and within intervals of 3 times for the removal of the organic impurities. Rinsing was then done using distilled water. After that, drying was done at 120°C for 24 hours in an oven. The crushed dry shells were calcined in a muffle furnace at 950°C for 4 hours. The white fluffy and porous materials were next pulverized using an agate mortar and kept in a desiccator.

**2.2. Catalyst Characterization.** The base strength of the catalyst was evaluated using several Hammett indicators. Approximately, 25 mg of the catalyst was agitated in a solution containing 5 ml of methanol solution of Hammett indicators and permitted to equilibrate for 2 hours. The basic strength is said to be more than the weakest signal that creates a color change but less than the strongest indicator that causes no change. Phenolphthalein ( $H^- = 9.3$ ), bromothymol blue ( $H^- = 7.2$ ), 2,4-dinitro aniline ( $H^- = 15$ ), Nile blue sulfate ( $H^- = 10.1$ ), p-chloroaniline ( $H^- = 26.5$ ), and 4-nitro aniline ( $H^- = 18.4$ ), were utilized as Hammett indicators. Differential thermal and thermogravimetric (DTATGA) analyses were performed in the temperature range of 35–1100°C utilizing a Netzsch-Geratebau GmbH (Germany) apparatus (Model STA 409) under nitrogen flow. Sun et al. [18] used a Brunauer-Emmett-Teller (BET) surface area study to estimate parameters such as surface area, mean pore diameter, and pore volume. The Barrett-Joyner-Hallenda (BJH) technique was used to determine the distribution of pore size in the samples [18]. Nitrogen adsorption-desorption isotherms were measured using the Micromeritics ASAP 2020 device at a temperature of  $-195.562^\circ\text{C}$ .

An X-ray diffractometer (Scifert and Co. model 3000) was used to examine the catalyst's XRD pattern at an angle of  $2^\circ$  and a scan range of  $10$ – $90^\circ$ .

Fourier transformed infrared (FT-IR) spectroscopy using a Thermo Electron Co. Model Nicolet 5700 fitted with a Hg-Cd-Te cryodetector at wave numbers of  $4000$ – $650\text{ cm}^{-1}$  at a resolution of  $4\text{ cm}^{-1}$  was used to determine functional groups bound to the catalyst surface. An infrared vacuum cell having greaseless stopcocks and KBr windows were used to record the spectra [19].

**2.3. Refining of the Waste Frying Oil.** The free fatty acid value was determined by calculating the concentration of sodium hydroxide solution that was made in order to neutralize the oil. At room temperature ( $30^\circ\text{C}$ ), the alkali solution was added to the oil, stirred at 300 rpm, and allowed the reaction to continue for 10 minutes, and thereafter, the mixture was subjected to heating at  $70^\circ\text{C}$  in order to breakdown the formation of soap. Following that, a centrifuge at 4500 rpm for 20 minutes was used to separate the samples. The oil was then washed three times with warm water ( $50^\circ\text{C}$ ) and dried with a rotatory evaporator.

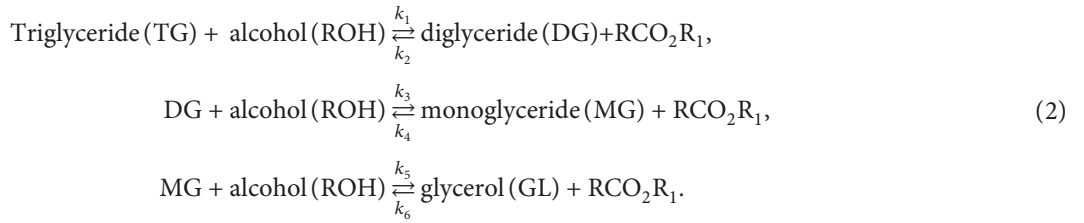
**2.4. Reaction Procedure.** A 500 mL round bottom flask was used as a reactor for the transesterification process. A reflux condenser, thermometer, mechanical stirrer, and sample port were all included in the reactor's design. Before being

put into the reactor, the oil was preheated at 120°C for 30 minutes to eliminate any water that may have been present. As the oil dried, a little quantity of methanol and sodium hydroxide were dissolved separately. The methanol-sodium hydroxide combination was then transferred into the reactor while it was being stirred. Temperature range (40–65°C), stirring rate (600 rpm), molar ratio of methanol-to-oil of 6 : 1, and reaction period of 120 minutes were used as the optimization conditions, mentioned by Okullo et al. [20]. The following equation was used to calculate the yield of biodiesel produced:

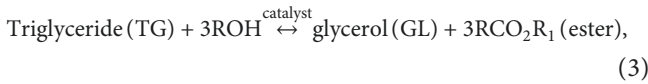
$$\text{Biodiesel yield (\%)} = \frac{\text{Weight of biodiesel}}{\text{Weight of oil}} \times 100. \quad (1)$$

In this work, all results are provided as means (values) of triplicate experimental and GC measurements.

**2.5. Kinetics Model of Transesterification of the Waste Frying Oil.** The transesterification process is a reversible and sequential reaction that is triggered by excess alcohol and a catalyst. The following is a representation of the reaction [13]:



Overall reaction:



where  $k_1$ ,  $k_3$ , and  $k_5$  are the forward reaction rate constants and  $k_2$ ,  $k_4$ , and  $k_6$  are the reverse reaction rate constants. Depending on the working circumstances, the catalyst used, and the molar ratio of alcohol to oil, the three sequential transesterification reactions might be equilibrium, forward, or reverse processes. The following equation was used to represent the kinetic rate constants as a function of reaction time [13]:

$$\begin{aligned} \frac{d[\text{TG}]}{dt} &= k_1 [\text{TGIROH}] + k_2 [\text{DGIME}], \\ \frac{d[\text{DG}]}{dt} &= k_1 [\text{TGIROH}] - k_2 [\text{DGIME}] \\ &\quad - k_3 [\text{DGIROH}] + k_4 [\text{MGIME}], \\ \frac{d[\text{MG}]}{dt} &= k_1 [\text{DGIROH}] - k_4 [\text{MGIME}] \\ &\quad - k_5 [\text{MGIROH}] - k_6 [\text{GLIME}], \\ \frac{d[\text{GL}]}{dt} &= k_5 [\text{MGIROH}] - k_6 [\text{GLIME}], \\ \frac{d[\text{ME}]}{dt} &= k_1 [\text{TRIROH}] - k_2 [\text{DGIME}] \\ &\quad + k_3 [\text{DGIROH}] - k_4 [\text{MGIME}] \\ &\quad + k_5 [\text{MGIROH}] + k_6 [\text{GLIME}], \\ \frac{d[\text{ROH}]}{dt} &= \frac{d[\text{ME}]}{dt}, \end{aligned} \quad (4)$$

where [DG], [TG], [MG], [ROH], [GL], and [ME] represent the mole concentrations (mol/L) of DG, TG, MG, alcohol, GL, and esters, respectively, in the reaction mixture. Using the three-point technique, the differentiation of the mole concentrations with respect to time on the left-hand side of equation (4) was approximated from the experimental mole concentrations at various reaction periods. The least-squares regression approach was used with Origin 8 Pro SRA4 Version 15.8.0.347 and MATLAB to solve the second-order differential system of equations. The equation may be adjusted to provide a system of nonlinear equations with six unknowns:

$$\begin{bmatrix} -a_{11} & -a_{12} & 0 & 0 & 0 & 0 \\ a_{21} & -a_{22} & -a_{23} & a_{24} & 0 & 0 \\ 0 & 0 & a_{33} & a_{34} & a_{35} & -a_{36} \\ 0 & 0 & 0 & 0 & a_{45} & -a_{46} \\ a_{51} & -a_{52} & -a_{53} & -a_{54} & -a_{55} & -a_{56} \\ -a_{61} & a_{62} & a_{63} & a_{64} & -a_{65} & a_{66} \end{bmatrix} \begin{bmatrix} [k_1][b_1] \\ k_2b_2 \\ k_3 = b_3 \\ k_4b_4 \\ k_5b_5 \\ [k_6b_6] \end{bmatrix}, \quad (5)$$

$$\mathbf{E}^2 = \sum_{i=1}^n [b_i - a_{i1}k_1 + a_{i2}k_2 + a_{i3}k_3 + \dots + a_{in}k_n],$$

where  $a_1 - a_6$  are the measured data points (mole concentrations),  $b_1 - b_6$  are the dependent variables (differentiations of mole concentrations on the left-hand side of equation (4)), and  $k_1$ ,  $k_2$ ,  $k_3$ ,  $k_4$ ,  $k_5$ , and  $k_6$  are the rate constants as previously defined. The method used to get the effective rate constants were derived from Figure 1.

### 3. Results and Discussion

**3.1. Characterization of the Oil.** A base catalyst was chosen, given that the waste oil had an acid value of 0.709 (mg of KOH/g of oil) and water content of 0.9 wt%.

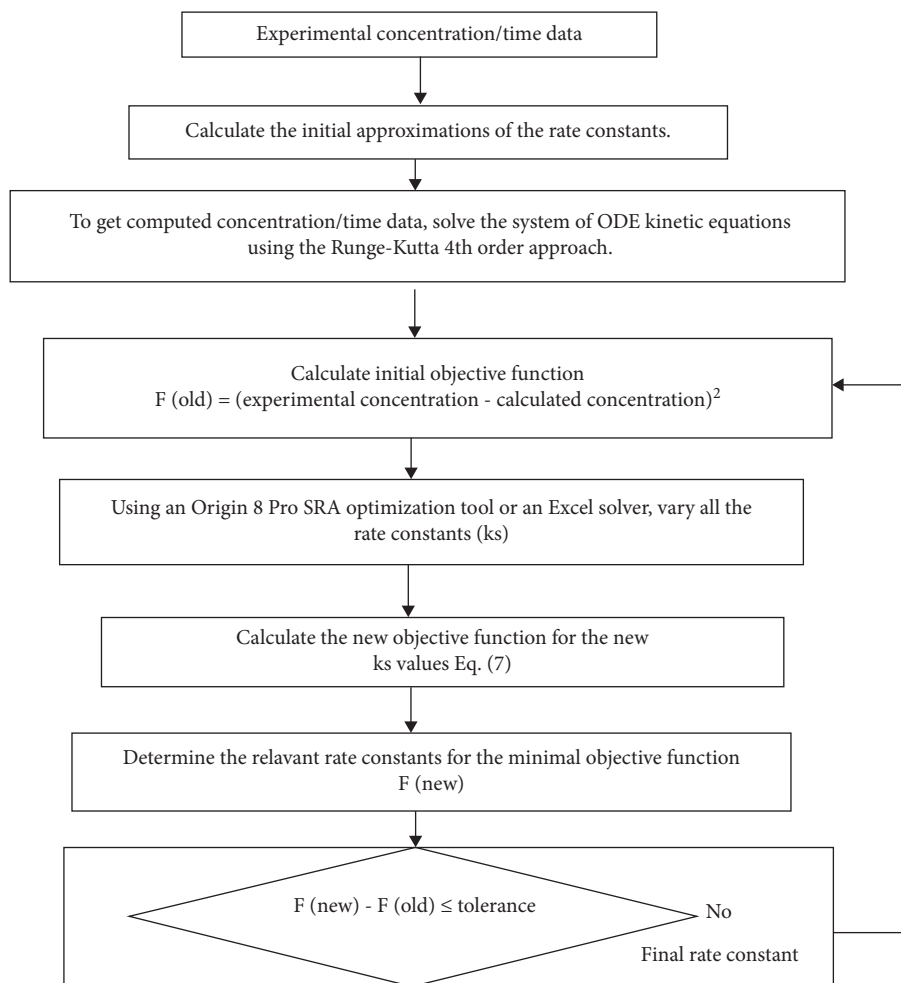


FIGURE 1: Flowchart for the determination of rate constants.

TABLE 1: Calcination condition of fish shell compared with other  $\text{CaCO}_3$ -based natural materials and resulting specific surface areas [17, 20].

Source of catalyst	Calcination temperature ( $^{\circ}\text{C}$ ) and time (h)	Calcination environment	BET surface area ( $\text{m}^2/\text{g}$ )
$\text{CaCO}_3$	900	—*	6
Fish shell	950	Air	21
$\text{CaCO}_3$	900	Air	4.3
The shell of Meretrix Venus			0.5
The shell of a golden apple snail			0.9

**3.2. Characterization of Catalyst.** The catalyst's basic strength was determined to be  $15 < \text{H}^- < 18.4$  and was regarded as a strong basis for the transesterification process. This number is greater than the one published by Liu et al. [21] who found activated  $\text{CaO}$  to have a basic strength of  $10.1 < \text{H}^- < 11.1$ . According to studies, decarbonation of  $\text{CaCO}_3$ -based compounds including limestone often happens at temperatures more than  $800^{\circ}\text{C}$  high atmospheric pressure.  $\text{CaCO}_3$  containing fish shells is transformed into an active catalyst of  $\text{CaO}$ . Table 1 compares the temperature of calcination of the fish shell to that of other  $\text{CaCO}_3$ -based catalysts. Catalyst activity is greatly influenced by the precise surface area and active sites. The specific surface area was

calculated from the table (Table 1) to be  $21 \text{ m}^2/\text{g}$ . This number was comparable to previous research findings [22].

**3.3. X-Ray Diffraction (XRD) Pattern.** The existence of  $\text{CaCO}_3$  in the form of a weak orthorhombic structure was revealed by the presence of wide XRD peaks (Figure 2). Most of these diffraction peaks vanished during calcination, leaving just extremely sharp  $\text{CaO}$  peaks at  $2\theta = 16.2^{\circ}$ ,  $32.3^{\circ}$ , and  $34.8^{\circ}$ . The primary peaks for the unprocessed fish shell were found at  $2\theta = 6.242^{\circ}$ ,  $23.172^{\circ}$ ,  $28.572^{\circ}$ ,  $31.162^{\circ}$ , and  $34.889^{\circ}$ , indicating that calcium carbonate was present. The spikes are comparable to those obtained by Sharma and

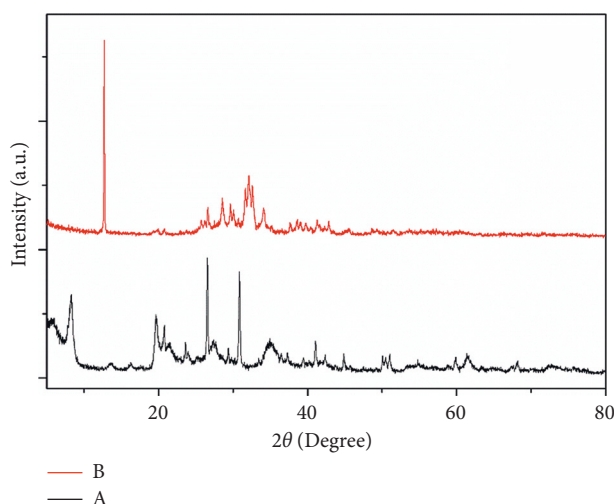


FIGURE 2: X-ray diffraction pattern of (a) uncalcined fish shell and (b) calcined fish shell.

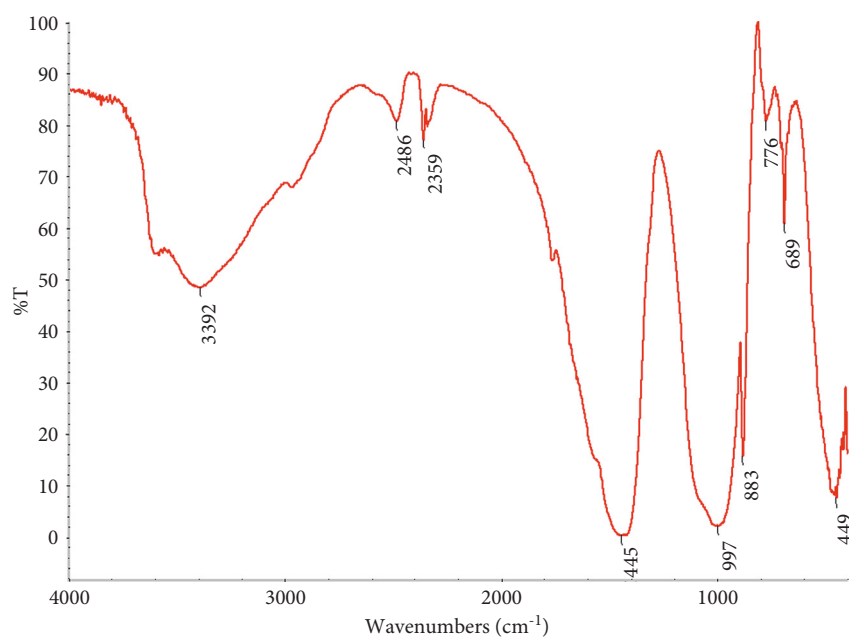


FIGURE 3: Fourier transformed infra-red (FT-IR) of the calcined fish shell.

Korstad [23] who detected CaO peaks at  $112^\circ$ ,  $32.3^\circ$ ,  $37.4^\circ$ , and  $34.9^\circ$  upon calcining discarded eggshell at  $900^\circ\text{C}$ . Wei et al. [24] reported similar peaks when discarded eggshell was calcined at temperatures of  $800^\circ\text{C}$ .

**3.4. Fourier Transformed Infra-Red (FT-IR) of Calcined Fish Shell.** The predominant absorption spectrum in the fish shell was 864, 1479, and  $3448\text{ cm}^{-1}$ , which are linked to carbonate ( $\text{CO}_3^{2-}$ ) species on the surface of the catalyst [23]. The carbonate ion in the fish shell is lost during calcination, and the absorbance spectrum of  $\text{CO}_3^{2-}$  ion is moved to a higher energy state [19]. These peaks reveal the mass loss caused by the loss of the  $\text{CO}_3^{2-}$  ion substituent linked to the surface of the catalyst as presented in Figure 3.

**3.5. Transesterification of Fish Shell Catalyst.** The methanol-to-oil ratio has a significant influence on biodiesel yield [2, 11]. Figure 4 illustrates the molar ratio versus percentage yield and the molar ratio versus % conversion to biodiesel. Biodiesel yield surged as the methanol-to-oil ratio increased, till it attained a constant yield of 87.6% at a molar ratio of 10:1. However, with an increase in the molar ratio past 10:1, the conversion of biodiesel was lowered. The greatest conversion (87.6%) was reached at a molar ratio of 10:1, as shown in Figure 4.

At a catalyst loading of 3.0–3.5%, the yield progressively improved and became steady at 87.6% as shown in Figure 5. With higher catalyst loading, the conversion of triglycerides increased. For 7 hours, the greatest conversion was achieved at 3.0% compared to the oil. The explanation for the lower

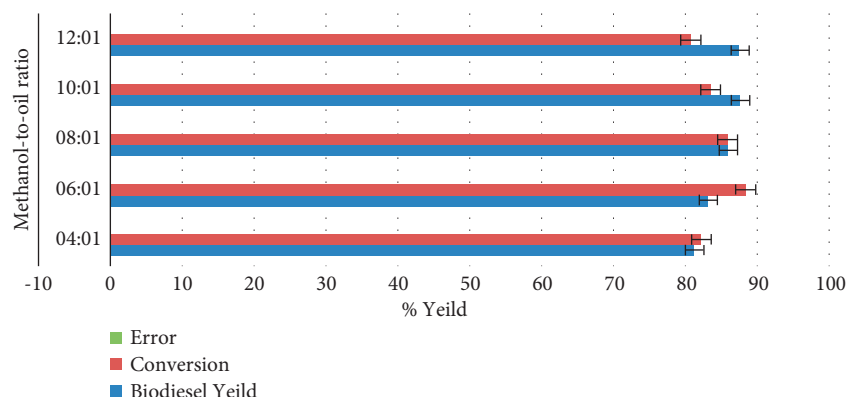


FIGURE 4: Effect of methanol-to-oil ratio on biodiesel yield and conversion (catalyst loading = 2.0 wt.% relative to oil, time = 7 hours, temperature = 60°C).

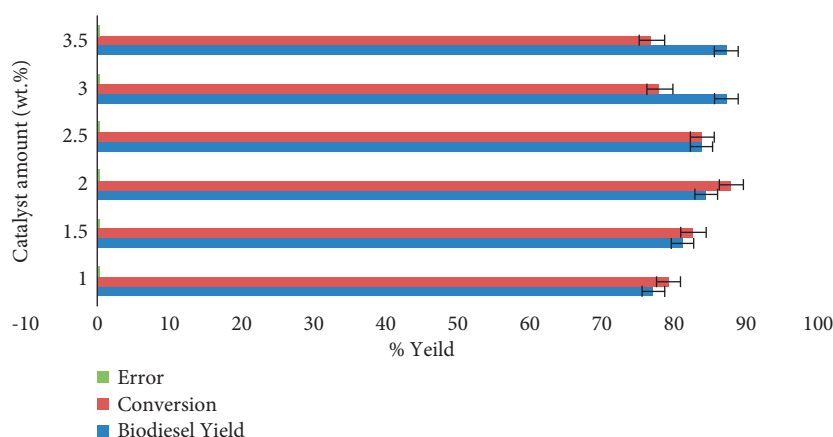


FIGURE 5: Effect of catalyst loading on biodiesel yield and conversion (methanol-to-oil ratio = 10:1, temperature = 60°, time = 7 hours).

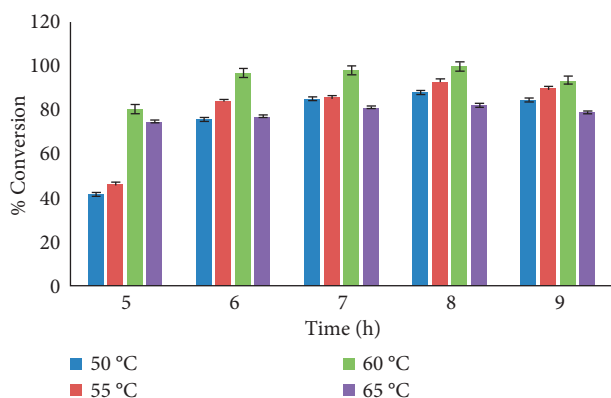


FIGURE 6: Effect of time and temperature on percentage conversion of biodiesel at a catalyst loading 2.0 wt.% relative to oil reaction temperature 60°C for 7 hours.

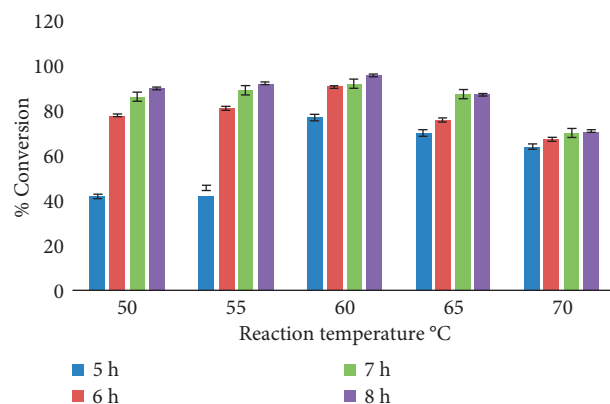


FIGURE 7: Effect of temperature and time on percentage conversion of biodiesel at methanol-to-oil ratio = 10:1 and catalyst loading = 2.0 wt.% relative to oil).

conversion with an additional catalyst might be related to an increase in the viscosity of the reaction mixture [2, 25]. As a result, the optimal quantity of catalyst relative to oil for transesterification of the oil is 3.0 wt.%.

Figures 6 and 7 illustrate a progressive rise in conversion over time from 5 to 8 hours at various temperatures using a 2.0 wt% catalyst in comparison to oil and a 10:1 methanol-

to-oil ratio. In 8 hours at 60°C, the greatest conversion of 99.2% was achieved. This finding is consistent with the findings of Wei et al. [24] who discovered that a higher temperature (65°C) was best for achieving a high yield (>95%). The solvent evaporated and stayed in the vapor phase in the reactor when the reaction was carried out at



TABLE 2: Concentration-time results.

Time (min)	TG (%)	DG (%)	MG (%)	GL (%)	ME (%)	OH (%)
0	1	0	0	0	0	6
5	0.2414	0.1714	0.0516	0.3142	1.5407	4.3481
10	0.1156	0.0215	0.0205	0.531	2.0767	3.8211
15	0.1518	0.0741	0.0174	0.6353	2.2273	3.5625
20	0.1263	0.052	0.0128	0.6956	2.3757	3.4241
40	0.0560	0.0201	0.0044	0.7883	2.6353	3.2525
60	0.0337	0.0202	0.0036	0.8320	2.7238	3.1651
90	0.0224	0.0144	0.0027	0.8480	2.7585	3.1213
120	0.0281	0.0131	0.0016	0.853	2.7814	3.1174

TABLE 3: Rate constant values from the study compared with those of Nouredдини and Zhu [15].

Rate constants (L/mol S <sup>-1</sup> )	Values from the current study	Nouredдини and Zhu [15]
$k_1$	0.102	0.040
$k_2$	0.021	0.110
$k_3$	0.053	0.114
$k_4$	0.010	1.127
$k_5$	0.015	0.231
$k_6$	0.164	0.006

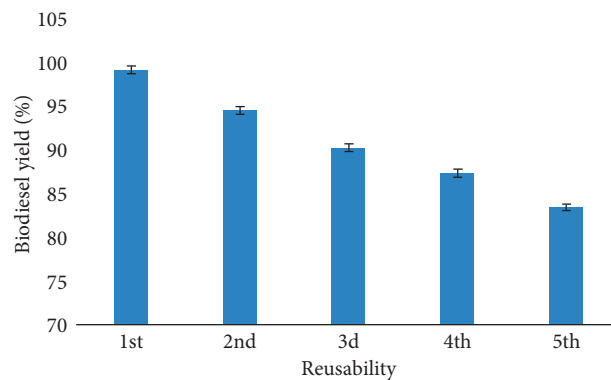


FIGURE 8: Reusability of catalyst.

TABLE 4: Properties of biodiesel compared with international standards.

Fuel property	Regular diesel	Biodiesel range	Biodiesel from the current work
Fuel standard	ASTM D 975	ASTM D 6751	ASTM D 6751
Acid value (mg of KOH/g of oil)	—	0.500	0.480
Density (kg/m <sup>3</sup> )	800–860	860–894	865
Calorific value (kJ/kg)	41,800–44,800	—	35305.87
Kinematic viscosity (40°C) St (m <sup>2</sup> /s) 106	1.3–4.1	1.9–6.0	5.4
Cloud point (°C)	–35–5	–3–15	1
Pour point (°C)	–35–15	–5–10	–4.00
Flash point (°C)	60–80	100–170	169

65°C, which is above the boiling point of methanol, resulting in a decrease in methanol in the reaction medium.

**3.6. Kinetics of Waste Frying Oil to Biodiesel.** The following concentration-time data in Table 2 were obtained from a simulation using the developed Origin 8 Pro SR4 and MATLAB .mfile and the rate constants reported by

Nouredдини and Zhu [15]. Using equation (4) and the concentrations at 10 minutes in Table 2, the approximate rate of constants was obtained as given in Table 3.

**3.7. Catalyst Reusability.** Each cycle of the stability testing employed a new reaction combination of methanol and waste frying oil. Figure 8 depicts the average findings of

biodiesel yield with reaction time as a function of the number of recycled cycles. The rate of reaction was quite high at the end of the 7 hours reaction time for all five runs (with biofuel production surpassing 83%). The decrease in methyl ester production ranged from 99.2% for fresh usage to 83.5% for the fifth reuse as indicated in Figure 8.

**3.8. Determination of Fuel Properties of Biodiesel Obtained from Waste Oil.** Vegetable oil undergoes a variety of chemical and physical changes while being used, resulting in the formation of certain unwanted and unknown substances. Some of these products are polymers that may be disrupted during the frying and transesterification processes [3]. The oligomeric substances generated during frying enhance the molecular mass of the oil and diminish its volatility. As a result, biodiesel derived from waste oils influences fuel properties such as decreasing burning characteristics and increasing viscosity, resulting in a higher quantity of carbon residue [3, 26]. Table 4 shows that the fuel characteristics of biodiesel were equivalent to ASTM standards and regular diesel.

## 4. Conclusions

Calcined fish shell catalyst showed significant activity for the transesterification of waste frying oil with methanol to biodiesel, with a yield of 99.2% attained under suitable conditions. By employing waste material as a catalyst and feedstock, this study tackles both environmental and economic concerns of biodiesel synthesis. As a result, it can be argued that this approach is adequate for predicting biodiesel and determining rate constants. The qualities of the fuels were within international guidelines.

## Data Availability

The data used to support this study are included within the article.

## Conflicts of Interest

The author declares that there are no conflicts of interest.

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