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Kinetic Study of the Transesterification of Waste Cooking Oil

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Abstract: *The rate and cost of biodiesel production has been hampered by the nature of the catalysts and feedstocks used. The bifunctional catalyst prepared from the mixture of 40 wt% sulphonated corncob (acid source) and 60 wt. % calcined cowbone (base source) was employed in the kinetic studies of the waste cooking oil transesterification for biodiesel production. The kinetic reaction tends towards fitting into a pseudo-first order reaction with activation energy (Ea) and collision factor of 21.4833 kJ/mol and* $0.4909s^{-1}$ *, respectively. The effects of each parameter on the production of biodiesel were highlighted. The physicochemical properties such as the flashpoint (403 K), pour point (281.2 K) and cloud point (283.11 K) of the synthesized biodiesel were also obtained. Furthermore, a test on reusability was performed for the bifunctional catalyst and it was found to retain its catalytic potential until the fifth cycle with a percentage catalyst loss of* $7\ \%$*. This result indicates that the bifunctional catalyst was effective in the kinetic study of the transesterification of waste cooking oil.*

Keywords: *Bifunctional Catalyst; Biodiesel; Heterogeneous Catalyst; Kinetic Study; Physicochemical Properties; Reusability Study.*

INTRODUCTION

The exploitation of energy resources such as natural gas, fossil – fuel and coal has always been known to fuel the growth of the global economy. However, as the human population increase steadily and urbanization as well as industrialization takes their respective shapes, the depletion of the world energy reserve and the emission of greenhouse gases (such as carbon dioxide and methane gas) has become a concern (Loy *et al.,* 2019). For instance, Kontorovich *et al.* (2013) reported that global coal production is expected to attain its peak between 2030 and 2040 and then gradually turn down at the climax of the 20th century after a brisk production. It was also reported that due to the burning of petroleum fuel, 22 x 10^9 tons of carbon dioxide per year is usually released into the atmosphere. These challenges have prompted researchers to seek an alternative source of energy (Babatunde *et al.,* 2019). Other sources of energy, like biodiesel, have been prospective. It is known to be the side product of the reaction between oils or fats and low chain alkanols like methanol or ethanol. The production of biodiesel from the blend of waste cooking oil and castor oil by transesterification method was reported by Fadhil *et al.,* (2017) where an optimum yield of 95.20 ± 2.5 % was obtained. Several researchers such as Obadiah et al (2012) and Maneerung *et al.,* (2016), amongst others, have also obtained interesting results on biodiesel productions. It has also become attractive due to its low sulphur content and biodegradability (Mardhiah *et al.,* 2017). Although biodiesel remains attractive, its cost of production has hindered its marketability. The use of low-cost feedstocks had in time played imperative roles, but these materials tend to possess high free fatty acid which consequently limits the rates of reaction and reduces the effectiveness of the catalyst.

Therefore, a heterogeneous bifunctional catalyst derived majorly from biomass sources has been designed by physical mixing methods (Aderibigbe *et al.,* 2021). The optimization studies performed showed that the acid-base bifunctional heterogeneous catalyst formulated as 40 wt.% sulfonated corncobs (acid source) and 60 wt% calcined cow-bone (base source) is most efficient for the conversion of waste cooking oil to biodiesel (Aderibigbe *et al.,* 2021). Therefore, to have sufficient information about the rate of reaction, activation energy, configuration of the transesterification reactors, process temperature, variable optimization and scale-up processes, it was justifiable to investigate the reaction kinetics (Birla *et al.,* 2012; Moradi *et al.,* 2015). In a kinetic model developed from the transesterification of waste cooking oil by Farag et al (2013), it was observed that the model fitted a first-order kinetic law. In another study, the kinetic study of two basic heterogeneous catalysts sourced from waste mussel shells and demineralized water treatment was utilized in the conversion of waste cooking oil (Moradi *et al.,* 2015). The activation energies (E_a) of the catalysts were found to be 77.09 and 79.83 kJ/mol respectively. It was established that the catalyst from the waste mussel shell was faster and may be suitable for biodiesel production on an industrial scale.

The lower activation energy (11.64 kJ/mol) has been documented by (Maneechakr *et al.,* 2015) when a solid acid catalyst was used in the kinetic study of waste cooking oil. The reaction also followed a first-order kinetics model. Furthermore, activation energy ($\mathrm{E_a} = 23.99\ \mathrm{kJ/mol}$) and frequency factor ($A = 1.62\ x\ 10^6\ min^{-1}$) were reported in a kinetic study for the transesterification of waste cooking oil using anthill-eggshell-Ni-co mixed oxide composite as catalyst (Yusuff *et al.,* 2018). Hence, the novelty of this research is to investigate the kinetic behaviour of acid-base heterogeneous bi-functional catalyst derived from cow bone and sulfonated corncob in the transesterification of waste cooking oil. The research was performed using an optimal operating condition of 6 h time, 11.75:1 methanol to oil ratio, reaction temperature of 338 K and 6 wt. % catalysts loading as reported in our previous research (Aderibigbe *et al.,* 2021). Based on the background highlighted above, this study aims to perform the kinetics of waste cooking oil to biodiesel viz-a-viz the bi-functional catalyst. The effect of reaction temperatures 323 K, 328 K, 333 K, and 338 K on biodiesel yield was investigated. The activation energy and frequency factor were calculated. The reusability test was also conducted to investigate how cost-effective the catalyst would be. Furthermore, the properties of the biodiesel produced were examined and were found to be within the American standard for testing materials (ASTM D6751)/European Standards.

MATERIALS AND METHODS

2.1 Materials

Analytically graded materials: methanol (98.9 %) and Tetraoxosulphate (iv) acid (98.9 %) were purchased from Sigma-Aldrich distributors (Zayo-Sigma) in Lagos, Nigeria. The distilled water was purchased from the Chemical Engineering laboratory, University of Ilorin, Ilorin, Kwara State, Nigeria. Also, junk food sellers at the University of Ilorin, Ilorin, Kwara State, Nigeria were freely provided with the waste cooking oil. In order to eliminate the moisture and impurities from the waste cooking oil, it was preheated at a temperature greater than 100 ℃ and then filtered. The corncobs were freely given by the roadside sellers along university road, Tanke, Ilorin, Kwara state. The corncobs were washed with distilled water, crushed and sieved. Lastly, the cowbones were acquired from the abattoir in Akerebiata, Kwara state, Nigeria. Catalyst preparation and characterization have been discussed extensively in our previous paper (Aderibigbe *et al.,* 2021).

2.2 Methods

2.2.1 *Kinetic Studies*

The transesterification reaction between the waste cooking oil and methanol was performed in a 250 mL flask placed on a magnetic stirrer. The flask was fitted with a thermometer and a condenser. The optimum conditions: temperature, methanol to waste cooking oil ratio, time, and catalyst uptake were used as obtained by Aderibigbe *et al.* (2021). However, in this kinetic study, the catalyst uptake and methanol: oil ratio were kept constant. The study was performed with temperatures 323K, 328K, 333K, and 328K, through different times of 0.5 h, 2 h, 4 h, and 6 h. After each operation, the product was discharged into the separating funnel through a filter paper. It was then allowed to separate overnight. The separated biodiesel was collected, washed with warm distilled water (343K) and dried for 2 h at 373K. Biodiesel yield was calculated using Equation (1) (Aderibigbe *et al.,* 2021).

$$\%\text{ oil Yield} = \frac{\text{weight of the biodiesel produced}}{\text{weight of the waste cooking oil used}} \text{ x } 100\% \qquad (1)$$

The kinetics of the reaction was studied by measuring the effect of the time and temperature on the operating condition while putting into consideration the following assumptions: the reversible reaction could be ignored since methanol and catalyst were in excess; and the occurrence of a single step transesterification reaction (Konwar *et al.,* 2014). Hence, the rate of reaction was calculated from Equation (2)

$$-r_a = -\frac{d[TG]}{dt} = \frac{[TG - TG_o]}{dt} = K'.[TG].[MeOH]^3 \qquad (2)$$

Where TG is the final concentration of triglycerides, TG_0 is the initial concentration of triglycerides. Since methanol was in surplus, thus change in its concentration was neglected. Hence, the reaction could be assumed as a pseudo-first order reaction (Birla *et al.,* 2012; Freedman *et al.,* 1984).

Where $k = K'.[MeOH]^3$

k = Overall reaction constant, $K' = Equilibrium\ constant$, $[MeOH]$ represents the concentration of Methanol. By substituting k in Equation (2), we have Equation (3)

$$-r_a = -\frac{d[TG]}{dt} = k.[TG] \qquad (3)$$

Given that,

$$X_{ME} = 1 - \frac{[TG]}{[TG_0]}, \qquad (4)$$

where X_{ME} = conversion of waste cooking oil

Hence, by integrating Equation (3) and substituting Equation (4) into it, we have Equation (5)

$$-\ln(1 - X_{ME}) = k.t \qquad (5)$$

The values for "k" at four different temperatures of 323 K, 328 K, 333 K, and 338 K were determined by plotting $-\ln(1 - X_{ME})$ against t.

2.2.2. Determination of Activation Energy

The Equation (6) that relates to activation energy (E_a), pre-exponential factor (K_O) and temperature (T) has been established by Svante August Arrhenius in 1908. It has then been used to determine the activation energy and pre-exponential factor of the reaction by plotting. $\ln k\ against\ \frac{1}{T}$ Hence, the slope and the intercept equal $\frac{-E_a}{R}$ and $ln\ K_O$ respectively. $where\ R$, represents the universal gas constant (8.314 $J/mol/K$).

$$\ln \mathrm{k} = -\frac{E_a}{R} \mathrm{x} \frac{1}{T} + \ln k_o \qquad (6)$$

2.2.3 *Reusability Study at Optimal Condition*

The used catalyst was improved by washing with n-hexane to get rid of deposited material on active sites and then dried at 333 K for 20 mins before reuse. This process was repeated after each cycle without the addition of a small amount of catalyst as an enhancement. The percentage loss of the catalyst was also calculated using Equation (7). Moreover, the effect of each factor on biodiesel yield and the physicochemical properties of the biodiesel produced was also investigated.

$$\%\ loss\ of\ catalyst = \frac{m_1 - m_2}{m_1} \mathrm{x} \frac{100}{1}. \qquad (7)$$

where,

$m_1 = initial\ mass\ of\ the\ catalyst,\ m_2 = final\ mass\ of\ catalyst(\ at\ the\ last\ cycle)$

The effect of each factor on biodiesel yield Investigation of the physicochemical properties

RESULTS AND DISCUSSION

3.1 Determination of the Reaction Kinetics

The kinetic study of this work was carried out at the optimum condition required for the transesterification process. As shown in Fig. 1, the conversion of waste cooking oil at different temperatures of 323 K, 328 K, 333 K, and 338 K for the time range of 0.5 h to 6 h had been examined. The values of the biodiesel yield were used to evaluate the rate constants at the earlier mentioned temperatures and the gradients 0.01356, 0.01277, 0.01236, and 0.00926 each having R-squares of 0.9869, 0.948, 0.895 and 0.901 obtained from Fig. 2.

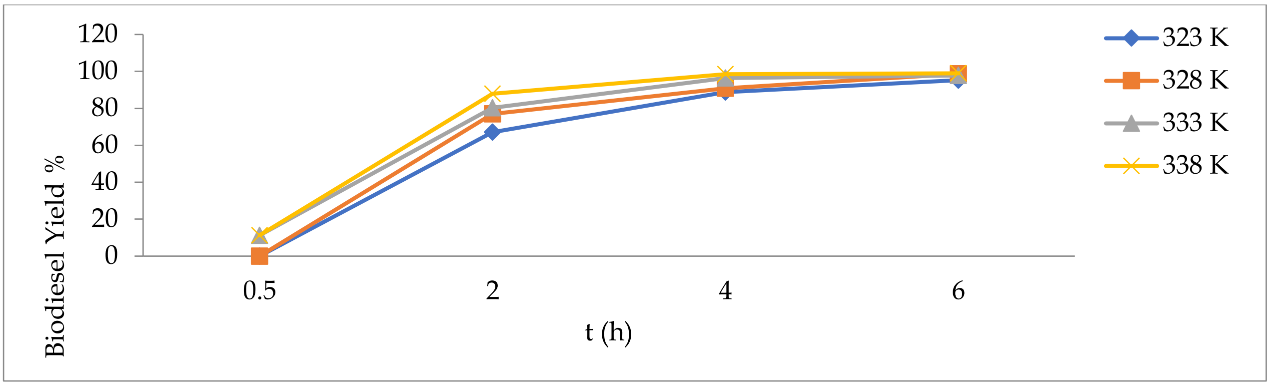

Fig. 1 The Plot of biodiesel yield against time

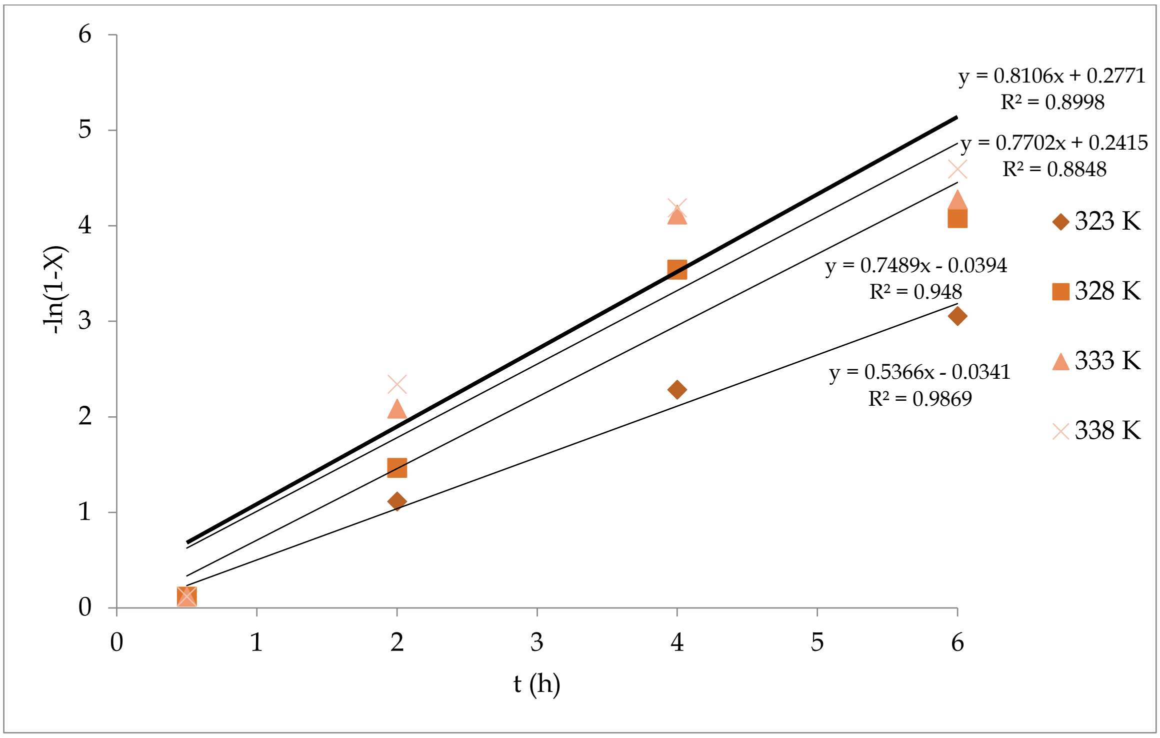

Fig. 2 The plot of $-ln(1-x)\ \ against\ t$

3.2 Determination Activation Energy

The plots (Fig. 1 and 2) revealed that the transesterification process follows a pseudo-first order reaction. Furthermore, activation energy (E_a) and pre-exponential factor (K_O) from Fig. 3 were obtained as 21.4833 kJ/mol and 0.4909 s^{-1} respectively with an R-square value of 0.926. A similar result for activation energy (21.65 kJ/mol) was also reported by (Naeem *et al.*, 2021) in the kinetic and optimization study of sustainable biodiesel production from waste cooking oil using a novel heterogeneous catalyst. The pre-exponential factor or collision factor indicates the number of times the molecules of the catalyst and reactants collide to gain energy to overcome the energy barrier which is the activation energy. A similar result was also reported to be 23.99 kJ/mol using an anthill-eggshell-Ni-Co composite catalyst (Yusuff *et al.*, 2018). The value of the activation energy (E_a) and pre-exponential factor (K_O) depends on the types of catalysts, feedstocks, and the oil used.

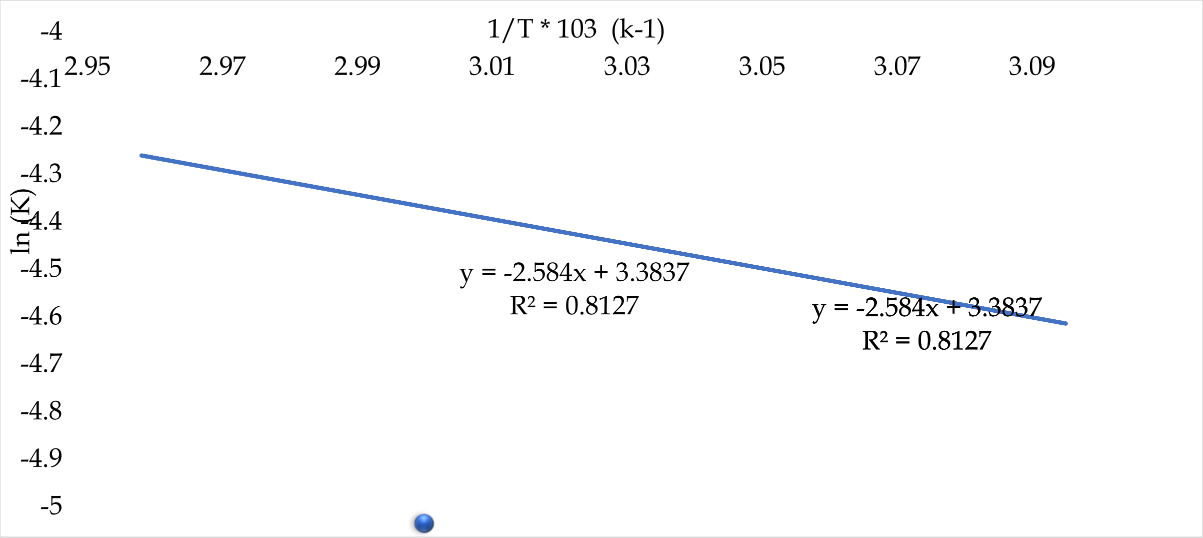

Fig. 3 Arrhenius plot of ln (K) against $1/T \ast 10^3$

3.3 Reusability Studies

To establish the reusability cycle of the bifunctional catalyst, it was tested under the optimum condition of the transesterification process with a reaction time of 6 h, methanol to oil ratio of 11.75:1, temperature 338 K and catalyst loading of 6 wt.%. The result in Fig. 4 revealed that the catalyst slightly maintained its stability for five cycles but at the fifth time the yield tends to decrease significantly. This may be a result of the deposition of glycerol, loss of catalyst (mass), unreacted oil or the leaching away of the catalyst site. The percentage loss of catalyst was obtained as 7 % in the fifth cycle. This may be due to the several washing of the bifunctional catalyst. In subsequence work, more research could be done to increase the stability of the bifunctional catalyst. This should consequently reduce the percentage loss of the bifunctional catalyst far below < 7% with an increase in the number of cycles without having to augment the amount of catalyst after each cycle. Furthermore, after the fifth use, the FTIR spectrum in Fig. 5 shows the disappearance of some of the wavenumbers such as 1033.88 cm^{-1}, 1165.04 cm^{-1}, 1249.91 cm^{-1} at the fingerprint region and 2314.66 cm^{-1}, at the diagnostic region. This indicates that there are some deactivations in functional groups on the active site of the catalyst.

Fig. 4 The reusability test of the bi-functional catalyst

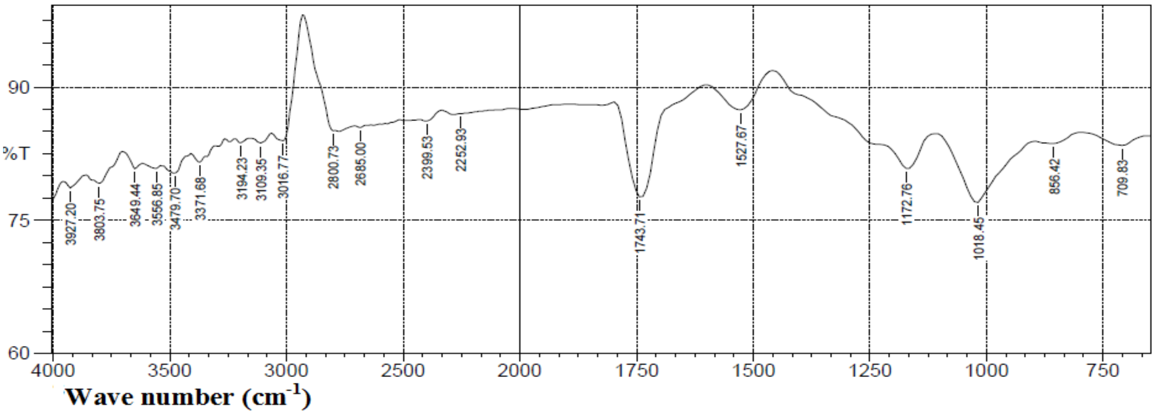

Fig. 5 FTIR spectrum of the bi-functional catalyst after five cycles

3.3.1 *The Effect of each Factor on Biodiesel Yield*

Shown in Fig. 6(a) is the relationship between temperature and biodiesel yield while maintaining other factors constant at the optimum condition. The graph revealed that when the temperature rises, the biodiesel yield rises. However, between 328 K and 333 K, the biodiesel yield (fatty acid methyl esters) began to attain a steady-state value of about 98.10 %. Fig. 6(b) shows the interaction between catalyst loading and biodiesel yield. It can be noted that at little catalyst loading the yield remains below 45 %, this may be caused by the low catalyst site available for reaction, but as the catalyst loading increases the biodiesel yield also rises until it gets to the utmost point at 6 wt.%, beyond which it begins to reduce. Also, it could have been a result of an increase in the viscosity of the reaction mixture, which then causes a resistance between the mass transfers of the methanol-oil-catalyst system. A similar situation was also reported, where a further increase in catalyst loading above 1.5 wt% resulted in a decrease in biodiesel yield (Essamlali *et al.,* 2018). Fig. 6(c) illustrates the relationship between time and biodiesel yield. It was observed that as time increases the biodiesel yield also increases and that there was no point within the time range of studies that the biodiesel yield reduces with respect to time.

This result is in agreement with the study of Leilei *et al.* (2015). Lastly, Fig. 6(d) illustrates the relationship between biodiesel yield and methanol to oil ratio. It was noticed that at lower methanol to oil ratio the biodiesel yield remains below 89 %. This may be due to the inability of the methanol to shift the reaction to the right to favour the forward reaction. However, as the reactant concentration increases i.e., methanol, more of the product (biodiesel) was produced but at 15: 1, there was a decrease in biodiesel yield. The result was in agreement with Maneerung *et al.* (2016).

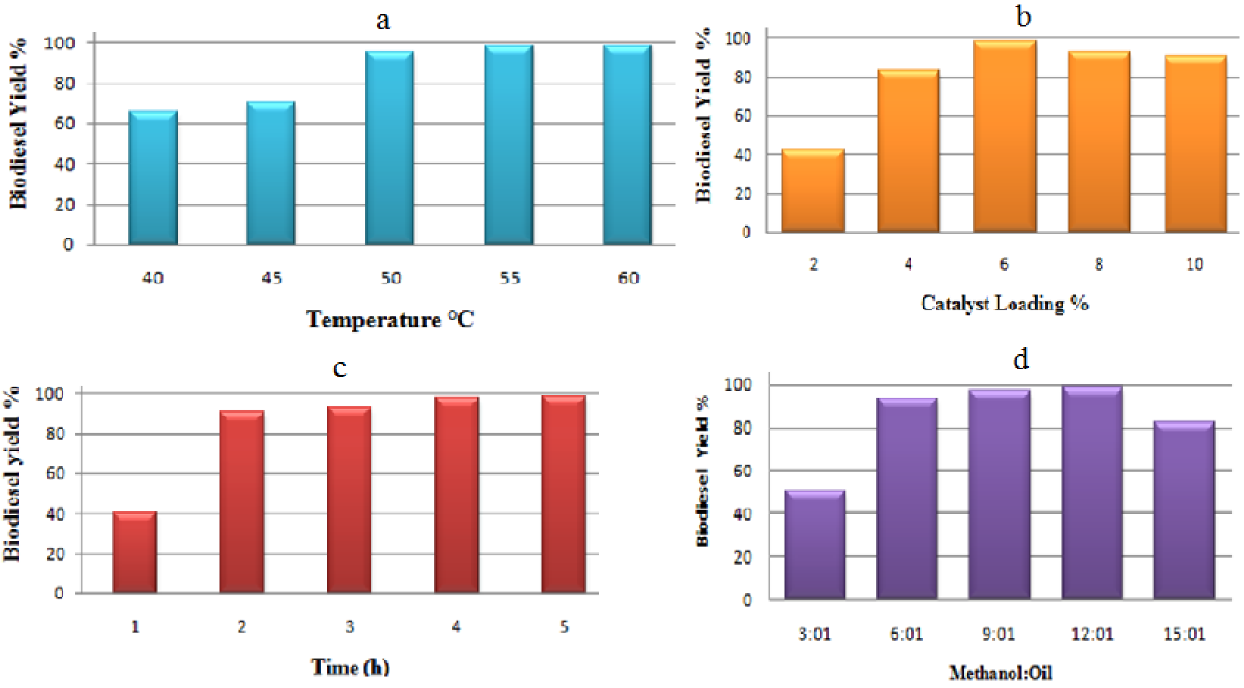

Fig. 6 Effect of (a) temperature, (b) catalyst loading, (c) time, (d) methanol: oil ratio on biodiesel yield

3.3.2. *Investigation of the Physicochemical Properties*

The chemical compositions and retention times of the synthesized biodiesel were obtained using the Gas chromatography Mass Spectrometer (GCMS) as shown in Table 1. The highest percentage composition of methyl ester was Octadecanoic acid methyl ester (26.89 %) followed by 9, 12-Octadecadienoic acid methyl ester (17.90 %), Pentadecanoic acid methyl ester (15.75 %) and so on. It also shows their retention on the major chromatographs at different retention times of 20.531, 21.731, 23.653, 25.517, 26.680 and 29.195 which correspond to the types of FAMEs present in the synthesized biodiesel. Similar results were also obtained by Djayasinga *et al.* (2022) when biodiesel produced from waste cooking oil was characterized using GCMS. Furthermore, physicochemical properties of the waste cooking oil and waste cooking oil-biodiesel were also examined. The values of these properties indicate the energy content, storage possibilities and clean-burning features of biodiesel. For instance, the higher the values of iodine and cetane number, the higher the unsaturated fatty acid and the combustion efficiency of the oil respectively. The results obtained were then compared with ASTM and EN standards as shown in Table 2. These properties were found to be comparable and in good agreement with ASTM/EN standard. Thus, these results revealed that the acid-base bifunctional catalyst showed good potential for the production of quality biodiesel.

Table -1 The fatty acid methyl esters in the biodiesel produced

Peak Numbers (Retention time)	FAMEs	FAMEs Compositions (%)
20.531	Eicosanoic acid methyl ester	12.89
21.731	11-Eicosanoic acid methyl ester	9.34
23.653	Octadecanoic acid methyl ester	26.89
25.517	9,12-Octadecadienoic acid methyl ester	17.90
26.680	Pentadecanoic acid methyl ester	15.75
29.195	Hexadecanoic acid methyl ester	11.97

Table-2 Physicochemical Properties of Biodiesel Produced

Properties	Units	ASTM D-6751	EN 14214	WCO (Current work)	WCO-Biodiesel (Current work)
Iodine value	g Iodine/100g	-	<120.0	54	80.45
Saponification value	mg KOH/g	-	-	182.66	172.98
Acid value	mg KOH/g	≤0.5	≤0.8	4.9360	0.673
Cetane number	-	≥ 47.0	-	67.90	73.25
Flashpoint	K	>393	-	414.00	403.00
Smoke point	K	-	-	358.00	357.00
Fire point	K	-	-	423.00	418.00
Viscosity(cp) (313 K)	Centipoises	1.9-6.0	3.50-5.00	8.97	5.08
Pour point	K	258.0 to 283	258 to 278	285.00	281.20
Cloud point	K	270 to 285	-	289.00	283.11
Specific gravity	g/ml	0.86-0.84	0.86-0.90	0.91	0.89
Higher Heating Value	kJ/mol	-	-	41.39	42.03

CONTRIBUTION TO KNOWLEDGE

The kinetics of bifunctional acid-base catalyst derived from cow bone and sulfonated corncob in the transesterification of waste cooking oil was studied successfully. Also, the research has shown that the catalyst is highly stable and could be reused up to the fifth cycle before losing its potency.

CONCLUSION

This work has studied the kinetic of waste cooking oil in a transesterification reaction using an acid-base heterogeneous bifunctional catalyst. By investigating the effect of different temperatures over a time range, reaction activation energy was found to be 21.4833 kJ/mol. The bifunctional catalyst was reused five times with a percentage catalyst loss of 7 %. The current study would aid to give the design information for a transesterification reactor and also help to mitigate the overall cost of biodiesel production. More research could be done to enhance the stability and catalytic activities of the bifunctional catalyst by the modification of the active sites.

DECLARATION OF CONFLIT OF INTERST

There is no conflict of interest for this research work.

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