Utilization of Bifunctional Catalyst for Biodiesel Production from Palm Kernel Oil

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Authors' contributions

This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.

Original Research Article

ABSTRACT

Palm empty fruit bunch (PEFB) and Titanium oxide (TiO_2) formulation was considered as a heterogeneous bifunctional catalyst for simultaneous esterification-transeterification of palm kernel oil (PKO) for biodiesel production. The bifunctional catalyst, Titanium oxide-Palm empty fruit bunch catalyst (TPEFBC), was synthesized from acid impregnated PEFB carbon and TiO_2, calcined at 600°C for 3 hours. Characterization result of PKO revealed high FFA (4.50%). Interactive effect analyses depict that the investigated process variables: reaction time, temperature, catalyst loading and methanol-oil-ratio (MOR) had significant effect on biodiesel yield. Reaction kinetics study was carried out at temperaures 45,60 and 75°C. The kinetic data at 60°C recorded coefficient of determination value (0.975) closest to unity thus best fitted the kinetic model with reaction order, 1.074. Gas chromatography result show that a biodiesel yield of 78.51% was achieved at 12:1 methanol to oil ratio, 1% catalyst loading, 333K reaction temperature and 180 minutes contact time. Results obtained suggest the capability of TPEFBC as a bifunctional catalyst for biodiesel production.

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1. INTRODUCTION

There is no gain saying the fact that energy plays a very important role in the society. Currently, the energy sector depends primarily on fossil fuel and this has increased greenhouse gas emissions into the ecosystem. This challenge has increased research interest in the quest for alternative supplies of energy that are of regenerative resource and environmentally friendly such as solar, wind and biofuel. Biodiesel is a prominent example of biofuel. According to Ramli et al., [1], "among explored biofuels, biodiesel has received a great deal of attention as compared to other biofuels due to its domestic and renewable origin, nontoxic nature, biodegradability, environmental benefits, and excellent lubricity".

"Biodiesel is the name for a variety of ester based oxygenated fuel from renewable biological sources. It can be used in compression ignition engines with little or no modifications" [2]. "Biodiesel is referred to as the monoalkyl esters of long-chain-fatty acids produced from a range of renewable resources such as vegetable oils (edible and non-edible), waste cooking oils (WCOs), animal fats, and oleaginous microorganisms" [2,3]. "Generally, biodiesel is manufactured by four methods: direct use (blending vegetable oil with diesel fuel), pyrolysis, micro-emulsion and trans/esterification reactions" [4]. Conventionally, commercial biodiesel is produced via liquid base-catalyzed transesterification of triglycerides components of oil/fat with short-chain alcohols [5] and has strict restrictions on the free fatty acid (FFA) and water content (<2%) to avoid saponification [6]. In scenarios of this restriction, reduction of FFA by esterification is usually applied to solve the problem of saponification.

It is established that esterification process has assisted with the problem of FFA reduction, however, the two different steps (esterification and transesterification) demand different catalysts and would unavoidably raise the production cost [7-9]. "In response to this problem, there are recent advances in the synthesis of bifunctional solid acid/base catalysts for sustainable biodiesel production with a focus on solid catalysts bearing acid-base bifunctionality or Brønsted-Lewis dual acidity and the synergistic effect of the active sites for simultaneous esterification of free fatty acids and transesterification of triglycerides" [5].

Previous researches have reported various levels of success on bifunctional catalysts synthesis for simultaneous esterification-transesterification reaction. Elias et al., [10], investigated "the simultaneous esterification-transesterification of waste palm oil and waste sunflower oil using CaO/Al_2O_3 catalyst in a conventional batch process". Prameswari et al., [7] studied "novel iron sand‑derived $\alpha\text{-}Fe_2O_3/CaO_2$ bifunctional catalyst for waste cooking oil‑based biodiesel production". Bharti et al. [11] found "their synthesized Sn-CaO bifunctional catalyst suitable for the conversion of waste cooking oil to biodiesel in a single-step reaction procedure". Pulungan et al., [12] demonstrated "the possibility of biodiesel production from rubber seed oil using natural zeolite supported metal oxide bifunctional catalyst".

In the current research, palm empty fruit bunch, an agrowaste of regenerative resource and Titanium oxide were used in synthesizing bifunctional heterogeneous catalyst to enhance the simultaneous esterification and transesterification of palm kernel oil for the production of biodiesel. To the best of the researchers' knowledge, the catalyst precursor combination for biodiesel production from palm kernel oil has not been reported. The interactive effect of reaction temperature, methanol-to-oil ratio, contact time and catalyst loading on the yield of biodiesel was explored.

2. MATERIALS AND METHODS

2.1 Materials

The biodiesel feed stock, Palm kernel oil, was purchased from a local vendor at Eke-Awka Market. Palm empty fruit bunch (PEFB) selected as the carbon support for the catalyst preparation was collected from a farm in Nise, Awka South Local Government Area, Anambra State. Sulfuric acid, methanol and titanium oxide were sourced from a chemical vendor in Awka, Anambra State. All chemicals used in this research were of analytical grade and used without further purification.

2.2 Methods

2.2.1 Physicochemical analysis of the Palm Kernel Oil (PKO)

The physicochemical properties of the PKO were determined according to methods earlier reported in Okoye et al. [13].

2.2.1.1 Density

A known volume of the sample was weighed in a digital mass balance, the mass obtained was tabulated and divided using the same measured volume of the sample. The measurements were made in triplicate and then averaged.

2.2.1.2 Determination of Free Fatty Acid (FFA) content

25ml of isopropyl alcohol was added to 25ml of PKO in a beaker. 2-3 drops of phenolphthalein was introduced in the mixture as indicator and agitated afterwards. The content of the beaker was agitated. The content of the beaker was then titrated against 0.1M of KOH solution until a faint pink color which lasted for about 15secs was obtained. The process was repeated 3 times in order to determine the average titer value.

The acid value was calculated using equation 1 [14]:

$$Acid\ value\ \left(\frac{mgKOH}{g}\right) = \frac{56.1 \times V \times N}{W} \qquad (1)$$

Where: V (cm^3) is the volume in of potassium hydroxide used in the titration, N (M) is the concentration of Potassium hydroxide, W (g) is the weight of the oil sample.

Free fatty acid (FFA) content is approximately half of the Acid value.

$$FFA = \frac{Acid\ value}{2}\ (\%) \qquad (2)$$

2.2.2 Determination of FAME content in biodiesel

2 g of the oil was first washed using 50ml of n-hexane, 0.5g sodium silicate and 1g of magnesium trisilicate powder. The mixture was allowed to stand for 7 minutes in a separating funnel. The filtrate from the mixture was then collected and analyzed using gas chromatography.

2.2.3 Pretreatment of Palm Kernel Oil (PKO)

The palm kernel oil obtained was filtered with the aid of a filter paper in order to remove particles that might be present in the oil. Thereafter, the filtered oil was heated to about 100ºC to remove any water content that must have been present on collection and probably due to exposure to the atmosphere. The water free oil is then stored in

a gallon to prevent further reactions and oxidation.

2.2.4 Synthesis of PEFB-based bifunctional catalyst

Palm empty fruit bunch-based carbon acid catalyst was prepared by direct impregnation method using concentrated H_2SO_4 acid according to the method reported by Thushari and Babel, [15]. Oven dried PEFB powder (108g) and concentrated H_2SO_4 were mixed (1:5wt/wt) at 100˚C in a glass beaker for 1hour using magnetic stirrer coupled with hot plate to facilitate in situ incomplete sulfuric carbonization. The mixture was kept for 24hrs, then diluted, filtered and repeatedly washed with hot distilled water (>80˚C) until the excess H_2SO_4 acid was removed. The catalyst was kept in an oven at 120˚C for 2 hours to eliminate all moisture. The sample was denoted as PEFB-DS-SO$_3$H [15]. Titanium oxide (20g) was partially mixed with 5ml of concentrated H_2SO_4 to make a thick slurry then oven dried at 100˚C for 1 hour. The oven dried acidified titanium oxide was size reduced in a rice cup crucible and mixed with the PEFB-DS-SO$_3$H. The mixture, Titanium oxide-Palm empty fruit bunch based bifunctional catalyst (TPEFBC) was calcined in a muffle furnace at a steady temperature (600˚C) for 3 hours. The resultant sample was stored in a dessicator to avoid contact with air or moisture.

2.2.5 Biodiesel production

The simultaneous esterification-transesterification process to produce biodiesel was carried out in a 500ml two-neck glass reactor equipped with a reflux condenser to avoid alcohol evaporation, magnetic stirrer and a thermocouple. A known amount of TPEFBC (catalyst) was dissolved in a calculated amount of alcohol (methanol) to form an alkoxide solution. A predetermined amount of the pretreated oil was gradually added to the alkoxide solution in the reactor. The content of the reactor was heated with constant agitation (300rpm) at a set time and temperature. At the elapse of the reaction time, the product of the reaction was transferred to a separating funnel for about 4 hours. The product was then separated from glycerol. Washing is carried out by spraying water over the biodiesel in a separating funnel with mild stirring to avoid foam formation. The water (containing the impurities) phase was drained off from the funnel and properly disposed while the biodiesel layer was heated to about 110˚C to evaporate traces of

remnant water molecules. The percentage of the biodiesel yield was determined by comparing the weight of biodiesel produced with the weight of PKO used [13].

$$\%\text{Yield} = \frac{\text{weight of biodiesel}}{\text{weight of PKO used}} \times 100 \qquad (3)$$

2.2.6 Reaction kinetics

The step-wise transesterification reaction is presented in equations 4 to 6 [13]:

$$TG + MOH \leftrightarrow DG + ME \qquad (4)$$

$$DG + MOH \leftrightarrow MG + ME \qquad (5)$$

$$MG + MOH \leftrightarrow G + ME \qquad (6)$$

TG, DG, G, MOH and ME represent triglyceride, diglyceride and glycerine, methanol and methyl ester respectively.

The overall transesterification reaction is written in equation 8:

$$TG + 3MOH \leftrightarrow 3ME + G \qquad (7)$$
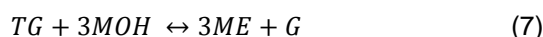
The relative rates of reaction of the various species in equation 8 are given by:

$$\frac{-r_{TG}}{1} = \frac{-r_{MOH}}{3} = \frac{r_{ME}}{3} = \frac{r_G}{1} \qquad (8)$$

The rate of disappearance of triglyceride can be expressed by the rate law:

$$-r_{TG} = \frac{-dC_{TG}}{dt} = KC_{TG}^a C_{MOH}^b \qquad (9)$$

Where a, b and C_{TG}, C_{MOH} are reaction orders and concentration at time t with respect to triglyceride and methanol respectively.

To ensure forward reaction, the reaction was run in excess of methanol therefore the reaction (equation 9) was considered irreversible as shown in equation 10. Since methanol is in excess, the concentration of methanol at any time, t, is nearly the same as its initial concentration and the rate law is independent of the concentration of methanol ($C_{MOH} \cong C_{MOHO}$) [13,16].

Therefore, $-r_{TG} = \frac{-dC_{TG}}{dt} = kC_{TG}^a \qquad (10)$

Where

$$k = KC_{MOH}^b \qquad (11)$$

$$C_{TG} = C_{TGO}(1 - X_{TG}) \qquad (12)$$

Substituting equation12 in 10:

$$C_{TGO}\frac{dX}{dt} = kC_{TGO}^a(1 - X_{TG})^a \qquad (13)$$

$$\frac{dX}{dt} = \frac{kC_{TGO}^a}{C_{TGO}}(1 - X_{TG})^a \qquad (14)$$

$$\frac{dX}{dt} = k'(1 - X_{TG})^a \qquad (15)$$

Taking natural logarithm

$$ln\left(\frac{dX}{dt}\right) = ln(k') + aln(1 - X_{TG}) \qquad (16)$$

The reaction order (a) is obtained from the slope of the plot of $ln\left(\frac{dX}{dt}\right)$ against $ln(1 - X_{TG})$

3. RESULTS AND DISCUSSION

3.1 Characterization

The palm kernel oil was characterized to determine the properties of the oil. The properties of oil such as acid value, FFA, etc, play significant role in the yield and quality of biodiesel. The FFA value recorded in table 1 is 4.50%. The high concentration of FFA value that characterize the biodiesel feedstock can negate fatty acid methyl ester (FAME) production in transesterification process given that FFAs saponify on reacting with base homogeneous catalyst. Bi-functional catalysts such as TPEFBC emerge as hopeful alternative since they provide considerable increase in the yield of biodiesel; allow the use of oils with high FFA in a single stage esterification-transesterification process thereby maneuvering the conventional two-stage process.

Table 1. PKO characterization result

Composition	Values
Acid Value	8.967mgKOH/g
Density	0.9128g/ml
pH	5.9
FFA	4.50%

The results from Table 2, suggests that the renewable feedstock, palm kernel oil, is a potential source of biodiesel hence will contribute in meeting the demands of the shortfall in supply of environmentally friendly energy. The %Yield of fatty acid methyl ester (FAME) from palm kernel oil was recorded to be 78.51 %.

3.2 Response Surface Plots for PKO Biodiesel Production

The interactive effect of factors that affect the transesterification process for biodiesel production is important in explaining the interdependence of variables on biodiesel yield. Figs. 1 to 5 depict 3D surface plots of %Yield of biodiesel against any two independent variables; and also models describing the trends of the plots. The plots and models describing the behaviour of the process were obtained using Statistica software.

3.2.1 Interaction effect of reaction time and temperature

The interaction effect of reaction time and temperature on %Yield of biodiesel in 3D Surface plot is displayed in Fig. 1. There was a corresponding increase in %Yield of biodiesel as time and temperature increased. However, at time greater than 140 minutes, a decrease in %Yield of biodiesel was observed. The increase in %Yield of biodiesel as temperature was increased over time possibly could be as a result of enhanced solubility of methanol in oil phase due to temperature increase [17].

3.2.2 Interaction effect of % catalyst loading and reaction time

Fig. 2 show the interactive effect of %catalyst loading and reaction time on %Yield of biodiesel. An initial increase in %Yield of biodiesel is observed at simultaneous increase in %catalyst loading and reaction time. A quasi-plateau was observed around 1.4% catalyst loading and 140 minutes reaction time. Any further increase in catalyst loading did not appreciably change the %Yield of biodiesel. Catalysts generally enhance the rate of a reaction process by lowering the activation energy. Thushari and Babel [15] reported that esterification of FFAs produces water as a byproduct. Excess water accumulation in the reaction medium can deactivate the catalyst thus resulting to a decrease of FAME production. Furthermore, encumbrance of the catalyst active sites by polar molecules in the reaction mixture is capable of lowering the ester yield with increasing time. This possibly explains the declining trend observed beyond 160 minutes reaction time.

Table 2. Gas chromatography–mass spectroscopy result for biodiesel derived from PKO

Component	Retention	Area	Height	External	Units
Monoglyceride	10.05	11304.8	784.333	2.6096	Ppm
Alcohol	24.24	37281.2	1607.25	3.9022	Ppm
Glycerol	25.35	7329.21	574.671	2.2294	Ppm
Triglyceride	26.67	13129.1	788.1	2.883	Ppm
FAME	30.866	52569.6	3155.62	45.8057	Ppm
Diglyceride	34.903	2580.56	203.001	0.9111	Ppm

$$\%\text{Yield} = -14.5522 + 0.8288*x + 0.9973*y - 0.0027*x*x - 0.0022*x*y - 0.0056*y*y$$
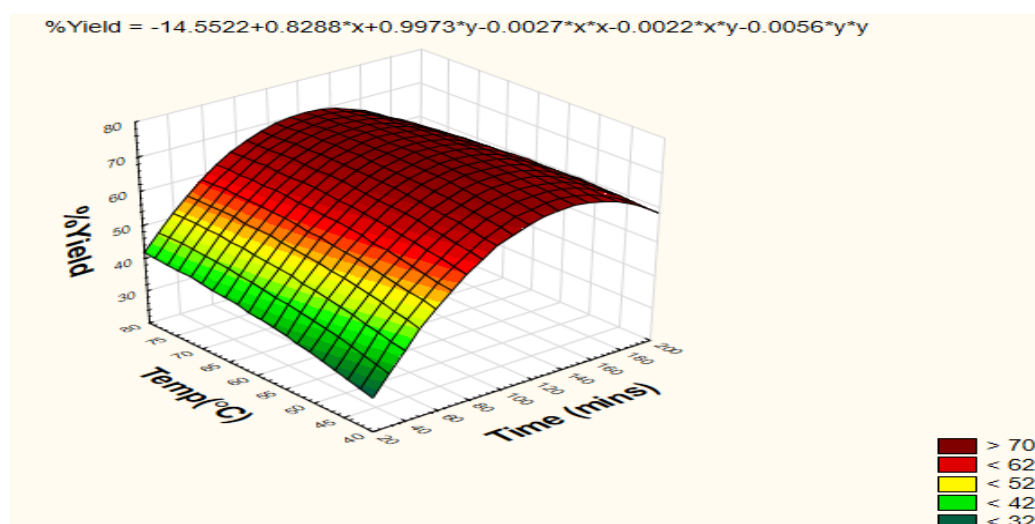
Fig. 1. Effect of reaction time and temperature on biodiesel yield

%Yield = -15.2221+64.8508*x+0.6021*y-20.9284*x*x-0.0169*x*y-0.002*y*y
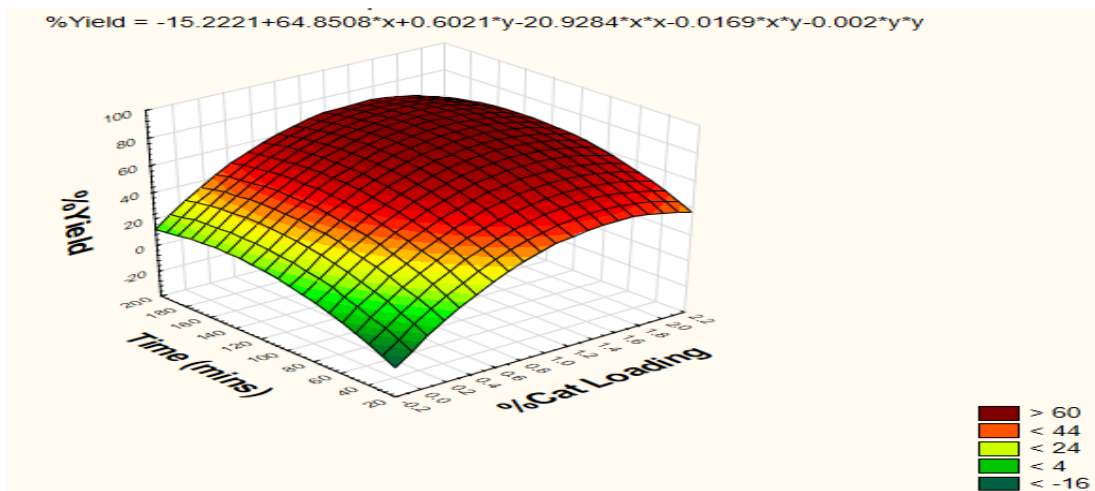
Fig. 2. Effect of % cat. loading and time on biodiesel yield

%Yield = 52.3588-0.2135*x+0.1064*y
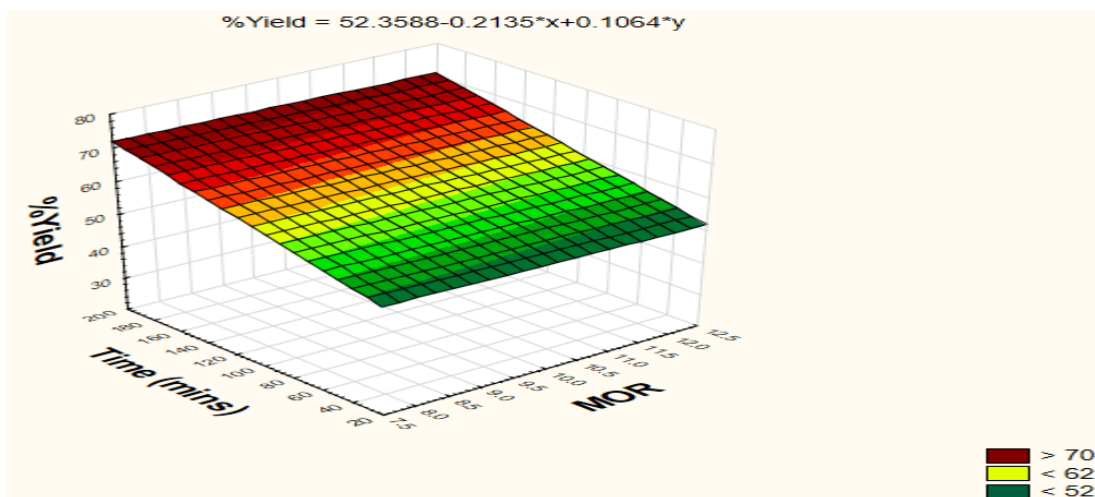
Fig. 3. Effect of MOR and reaction time on biodiesel yield

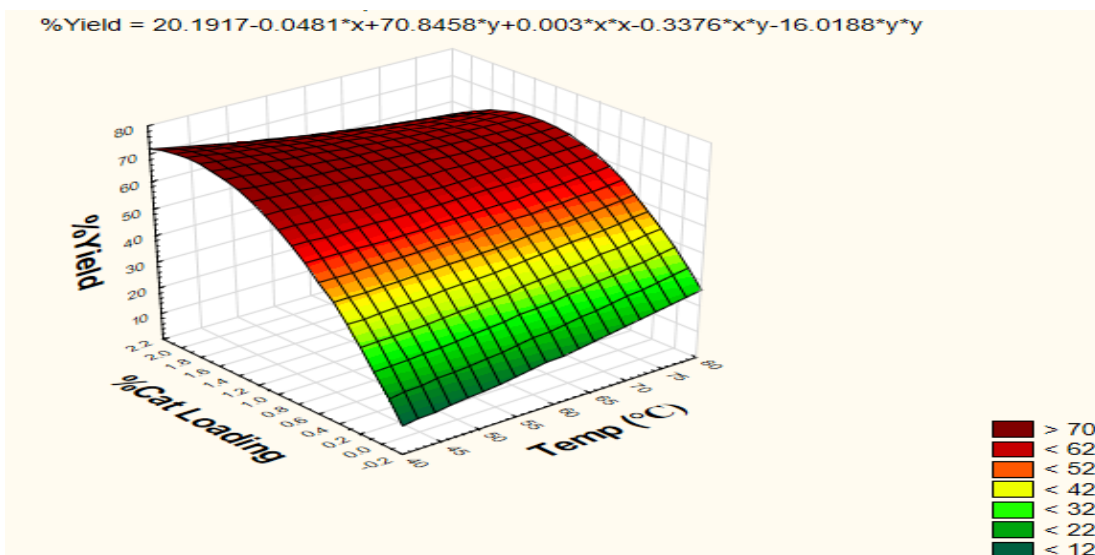%Yield = 20.1917-0.0481*x+70.8458*y+0.003*x*x-0.3376*x*y-16.0188*y*y

Fig. 4. Effect of temperature and %Cat. loading

3.2.3 Interaction effect of methanol-to-oil ratio and reaction time

The surface plot (Fig. 3) suggests a linear relationship between the independent variables and the dependent variable. It is noticed that simultaneous increase in time and MOR within the limits of the experimental values investigated was synergetic on the response variable (% Yield of biodiesel). Theoretically, transesterification reaction requires a 3:1 methanol-to-triglyceride molar ratio to achieve a forward reaction. Methanol, however, is required in excess for esterification reaction to neutralize excess free fatty acid in the medium. According to Le Chatelier's principle, it is essential to have excess methanol in the reaction medium to compel the reaction toward formation of FAME, owing to the fact that both esterification and transesterification reactions are reversible [15].

3.2.4 Interaction effect of reaction temperature and % catalyst loading

Fig. 4 displays a 3D surface plot of %Yield of biodiesel against temperature and %Catalyst loading. The %Yield of biodiesel increases with increasing temperature and % Catalyst loading. Beyond 2% Catalyst loading and 60°C, an insignificant effect was observed on the %Yield of biodiesel. This observed trend may be as a result that increase in temperature and catalyst loading above certain experimental conditions would lead to increased emulsion formation. Also, increase in the yield of biodiesel would occur as temperature increases although with low or intermediate amount of catalyst [18,19].

3.2.5 Interaction effect of reaction methanol-to-oil ratio and %catalyst loading

Fig. 5 shows the combined interactive effect of MOR and %Catalyst loading on %Yield of biodiesel. As MOR and %Catalyst loading values were reviewed upwards, a consistent positive effect on the response variable (%Yield of biodiesel) was clearly observed within the range of the investigated experimental values.

3.3 Reaction Kinetics

The heterogeneous-catalyzed transesterification reaction kinetics of palm kernel oil was studied at 45, 60, and 75°C reaction temperatures. Figs. 6 - 8 present the plots of $\frac{dX}{dt}$ against $\ln(1-X)$, at various temperatures of 45,60, and 75°C. From Table 3, the highest rate constant was recorded at 60°C. This may suggest that appreciable increase in conversion may not be recorded beyond 60°C within the investigated experimental conditions. The coefficient of determination value, 0.975 obtained at 60°C is closest to unity when compared with 0.963 and 0.964 recorded at 45°C and 70°C respectively. This is an indication that experimental data obtained at 60°C best fitted the kinetic model. In terms of equation 10, the rate equations for the reactions at temperatures 45°C, 60°C and 75°C can be expressed as: $-r_{TG} = 0.8878C_{TG}^{1.067}, -r_{TG} = 0.9364C_{TG}^{1.074}, -r_{TG} = 0.8813C_{TG}^{1.082}$ respectively [20-22].
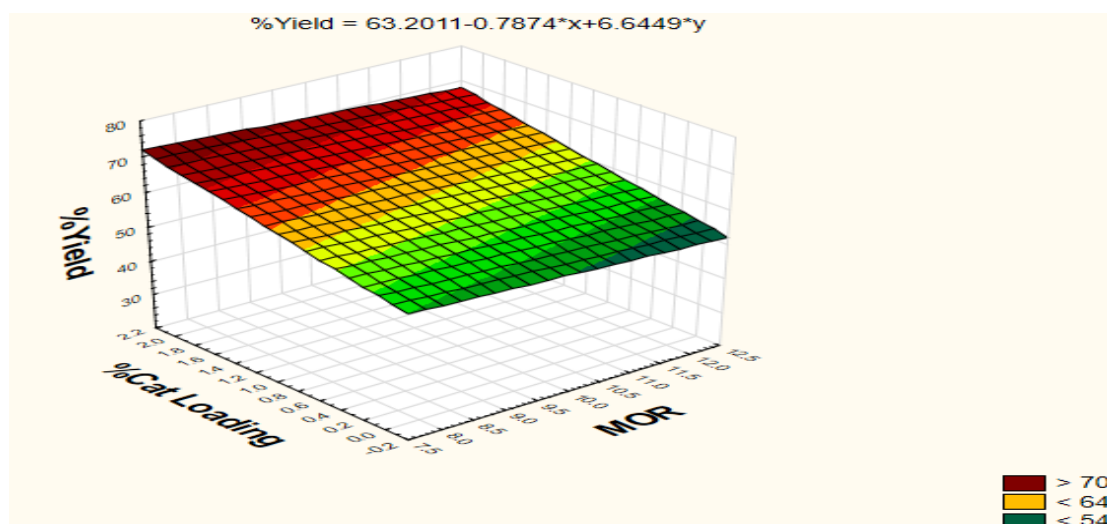

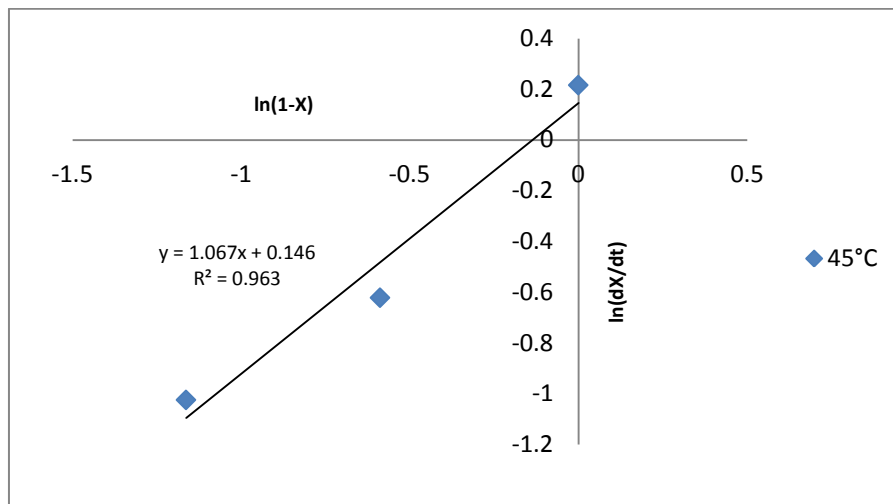**Fig. 5. Effect of MOR and %Cat. loading on biodiesel yield**

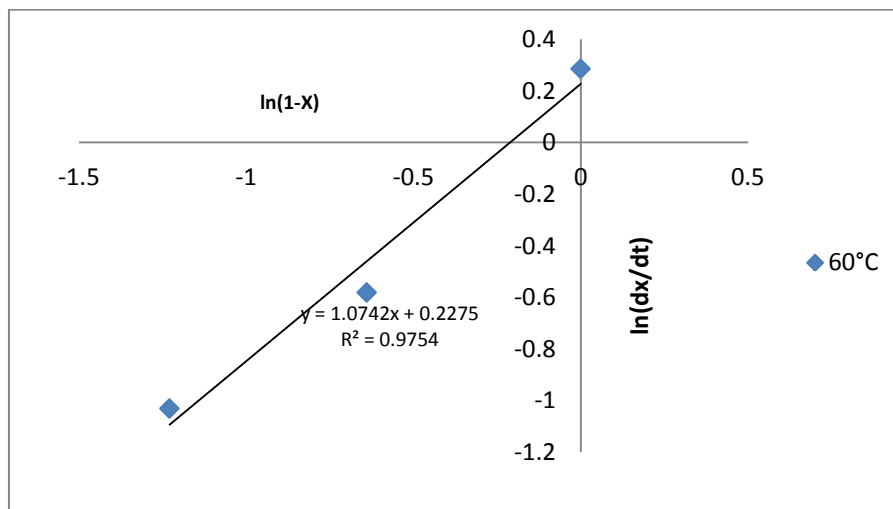**Fig. 6. Transesterification kinetics plot at 45˚C**

$y = 1.067x + 0.146$
$R^2 = 0.963$

Fig. 7. Transesterification kinetics plot at 60˚C

$y = 1.0742x + 0.2275$
$R^2 = 0.9754$
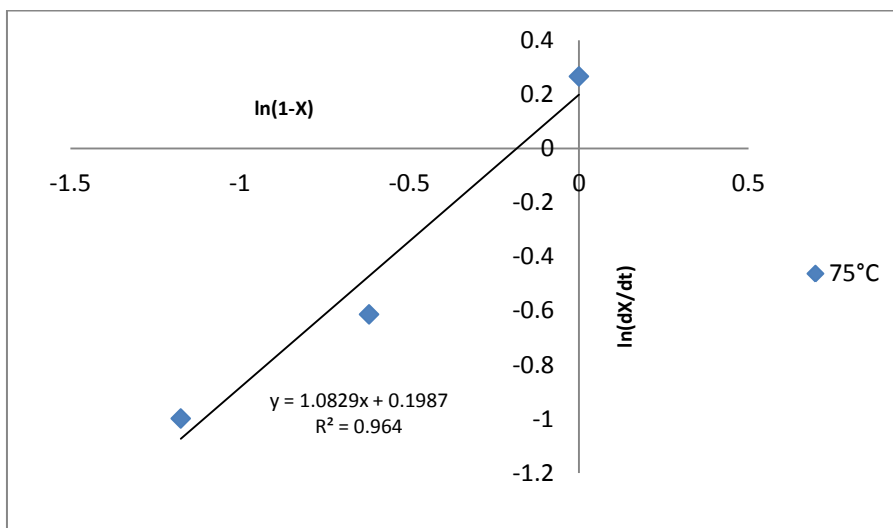
Fig. 8. Transesterification kinetics plot at 75˚C

$y = 1.0829x + 0.1987$
$R^2 = 0.964$

Table 3. Rate constants and reaction order at different temperatures

Temperature	$k(hr^{-1})$	α (reaction order)	R^2
45 ℃	0.8878	1.067	0.963
60 ℃	0.9364	1.074	0.975
75 ℃	0.8813	1.082	0.964

4. CONCLUSION

This study has achieved the production of biodiesel from palm kernel oil in a single stage esterification-transesterification reaction using a newly formulated bifunctional catalyst. Interaction of process variables: reaction time, temperature, catalyst loading and methanol-oil-ratio (MOR) revealed significant effect on the dependent variable (biodiesel yield). Gas chromatography result presented biodiesel yield of 78.51% achieved at 12:1 methanol to oil ratio, 1% catalyst loading, 333K reaction temperature and 180 minutes contact time. The high coefficient of determination value recorded suggests that kinetic data obtained at 60°C reaction temperature best fitted the kinetic model. The rate equation derived at 60°C is expressed as $-r_{TG} = 0.9364 C_{TG}^{1.074}$.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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