A kinetic study and thermometric analysis on waste cooking oil

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Abstract

The objective of this research work is to perform a kinetic study and thermometric analysis on waste cooking oil which has been reported along with the outcomes. Biodiesel was obtained through the process of base-catalyzed transesterification process, and it was carried out at 50 and 60 ℃, methanol/oil molar ratio and catalyst concentration were taken as a 9:1 and 0.45% w/w for potassium hydroxide (KOH), respectively at different stirring speed and reaction time. The experimental data from the transesterification reaction were fitted to first and second-order kinetic models. But the regression coefficient (R^2) showed that the first order was a better fit for the experimental behavior of the kinetic data. The Arrhenius equation was applied to calculate the activation energy and the frequency factor of a given system, resulting in 45.76 kJ/mol and 4.95×10^5 min^{-1}, respectively. At different temperatures, thermodynamic parameters were also taken into consideration in terms of Gibbs free energy, enthalpy, and entropy. Results show that biodiesel obtained from waste cooking oil gave better results and can be used as an excellent substitute of fossil fuels.

Keywords Transesterification · Kinetic · Biodiesel · Waste cooking oil · Regression · Thermodynamic

Abbreviations

R^2	Regression/slope coefficient
CO_2	Carbon dioxide
MT	Metric ton
WCO	Waste cooking oil
FFA	Free fatty acid
MG	Monoglycerides
DG	Diglycerides
E_a	Activation energy (KJmol^{-1})
ΔH	Change in enthalpy (Jmol^{-1})
h	Plank's constant (J)
R	Universal/Molar gas constant (Jmol^{-1} K^{-1})
T	Temperature in kelvin
A	Frequency factor
AFs	Alternative fuels
CI	Compression Ignition
ASTM	American Society for Testing and Materials
KOH	Potassium hydroxide
A.V	Acid value
FAME	Fatty acid methyl ester
ME	Methyl ester
ΔG	Change in Gibbs free energy (Jmol^{-1})
ΔS	Change in entropy
k_b	Boltzmann constant (JK^{-1})
K	Transmission coefficient
k	Reaction rate constant
TG	Triglycerides

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1 Introduction

The constraint of fossil fuel resources and the desire to reduce emissions of greenhouse gases served as a prime motivators for the development of alternative fuels. The growing focus on clean energy has led to the development and implementation of renewable fuels for diesel engines like biodiesel and other biofuels. Its production from renewable resources makes it biodegradable and non-toxic, and its closed carbon cycle reduces CO_2 emissions. These qualities make it ideal for use in products that are environmentally conscious [1–9]. India imports 80% of its annual oil need from other countries, which causes foreign fund outflows and currency fluctuations [10]. In India, about 3 MT of the 23 MT of cooking oil consumed can be utilized to make biofuel, according to

estimates. Waste cooking oil has been recognized as a viable raw material substitute as part of ongoing efforts to reduce raw material costs because WCO is around two to three times less expensive than virgin vegetable oils. Some of the common feedstocks for producing biodiesel are Waste cooking oil (WCO), beef tallow, waste frying oil, and other non-edible [11–13]. Prices for oil-containing products constitute 70 to 80 percent of the cost of biodiesel, therefore making the cost of producing biodiesel 1.5 times that of diesel, but the overall retail price of biodiesel is 10–15% lesser than diesel because of the excess tax imposed on imported crude oil [14, 15]. Homogeneous alkaline catalysts are frequently used to enable rapid response at low temperatures and pressures. Acids, bases, and enzymes are all catalysts that can be used to perform the transesterification of vegetable oils [16]. A study by Coteron et al. found that transesterification with methanol and strong basic homogeneous catalysts proceeded very quickly with oils containing up to 2.8% free fatty acids (FFA) [17]. To complete the transesterification reaction of triglycerides (TG), Diasakou et al. suggested that three steps are required, as depicted below: the first is the reaction with methyl alcohol for the production of diglycerides (DG), in the second stage monoglycerides (MG) is produced by reacting with methanol, and in the final stage, i.e., in the third stage pure methyl ester is produced along with the glycerol as a by-product when it is reacted with methanol, which is shown in the equations below [18]:

$$TG + MeOH \overset{k_1}{\leftrightarrow} DG + ME \tag{1}$$

$$DG + MeOH \overset{k_2}{\leftrightarrow} MG + ME \tag{2}$$

$$MG + MeOH \overset{k_3}{\leftrightarrow} ME + Glycerol \tag{3}$$

From Eq. 1–3, the reaction rate constants for each step are k_1, k_2, and k_3. A mathematical model for the transesterification chemical reaction is simplified by neglecting the in-between reactions of diglycerides and monoglycerides because the final product is methyl ester, and the by-product is glycerol. Because of this, the 3-step reaction can be reduced to a single-step reaction, as shown in Eq. 4.

$$TG + 3MeOH \leftrightarrow G + 3ME \tag{4}$$
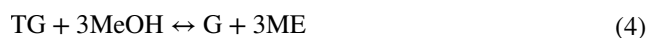
Biodiesel has been gaining more attraction in the research arena as an environment-friendly and sustainable fuel for CI engines, due to its superior properties compared to diesel. The use of alternative fuels (AFs), such as biodiesel and vegetable-based fuels in CI engines results in unsatisfactory performance due to the higher

viscosity and lower energy content of the fuel [19–22]. Kinetic studies and thermometric analyses can be time-consuming and costly to conduct, especially if multiple samples need to be analysed. These types of analyses often require specialized equipment and facilities which may not be readily available to all researchers. Depending on the disposal method of the waste cooking oil, there may be environmental concerns associated with its use in research, particularly if it is not properly disposed of or recycled after the study is completed [20–23]. There were kinetic investigations to characterize transesterification reaction kinetics with KOH acting as a catalyst in this research. At atmospheric pressure, these optimum transesterification values for waste cooking oil transesterification conditions were optimized: Based on the 9:1 methanol/oil molar ratio, the catalyst is employed at 50 to 60 °C with 0.45 wt.% of oil ratio. These findings can be used to predict the reaction's rate constant and activation energy. The main objective of this research work is to perform a kinetic study and thermometric analysis of waste cooking oil. Using the Arrhenius equation, the activation energy and frequency factor were determined accordingly. At different temperatures, thermodynamic parameters were also taken into consideration in terms of Gibbs free energy, enthalpy, and entropy.

2 Material and methodology

2.1 Raw material

In this research, Waste cooking oil is collected from the hostel mess of National Institute of Technology, Hamirpur (Himachal Pradesh), India.

2.2 Methodology

The production/extraction process of pure biodiesel, i.e., B100, is described briefly in the form of the flow chart in Fig. 1 [24]. This flow chart was created by studying the various factors that affect the biodiesel yield on the complete production of pure biodiesel (such as the methanol/oil molar ratio, the amount of catalyst in w/w%, the reaction temperature, and the chemical reaction time) through the transesterification process of waste cooking oil (raw biodiesel in this research work) with biodiesel ASTM standard specifications.

2.3 Determination of acid value

The Isopropyl Alcohol Method is commonly used for analyzing the Free Fatty Acid (FFA) content of oils. This
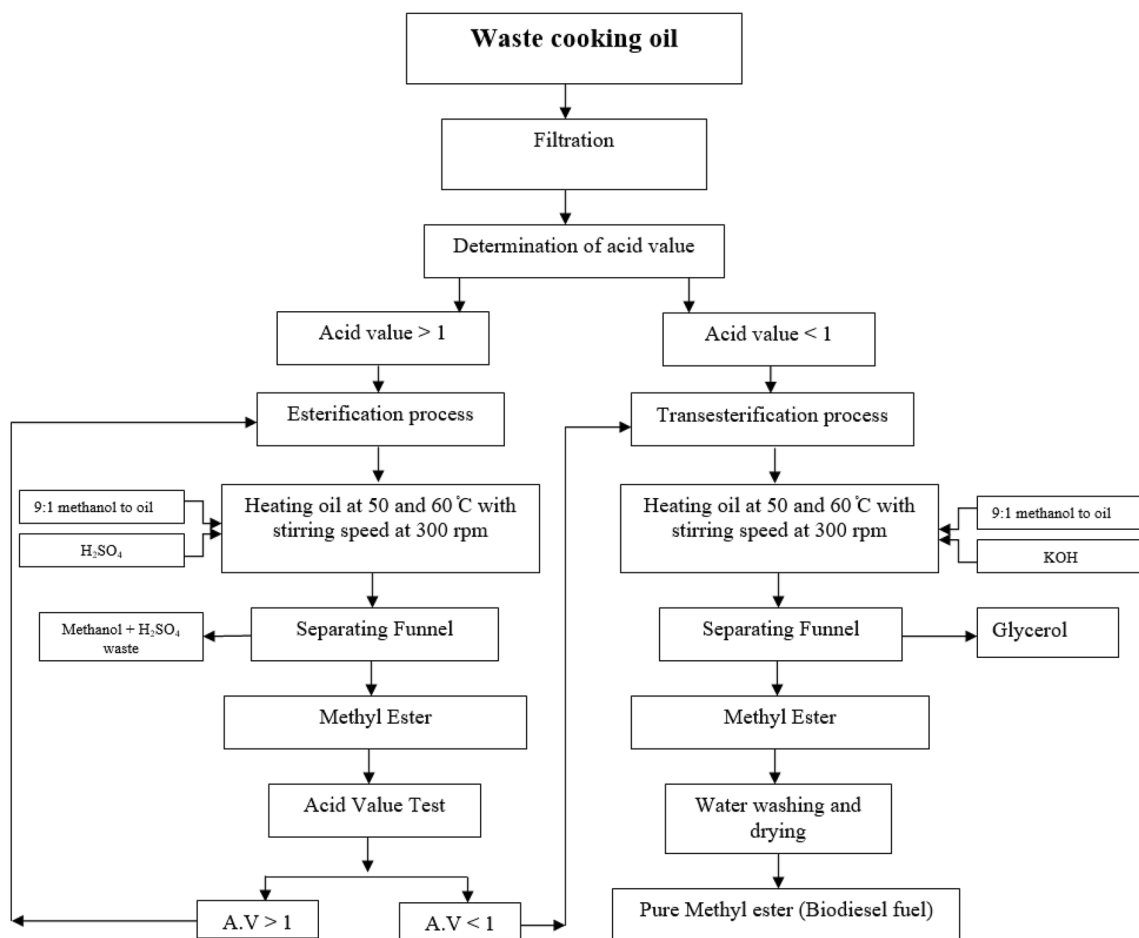
Fig. 1 Proposed biodiesel production flowchart

technique involves mixing 1 g of oil with 10 ml of Isopropyl Alcohol and 2 to 3 drops of Phenolphthalein Indicator. Slowly add 0.1N KOH to the mixture until a pale pink color persists for 10 s. Make a note of the reading to determine the acid value, which marks the end of this process. Make a note of the reading to determine the acid value, which marks the end of this process. This titration method helps in determining our oil sample acid value directly and FFA through a mathematical relation mentioned below in Eq. 5:

$$Acid\ value = \frac{56.1 \times N \times V}{w} \tag{5}$$

where w represents the weight of the oil sample, N represents the normality of the solution, V represents the volume of the solution used in the titration, and 56.1 is the Molecular weight of KOH. Hence the percentage of Free Fatty Acid (FFA) can be calculated as shown in Eq. 6:

$$FFA(\%) = \frac{Acid\ value}{2} \tag{6}$$

3 Result and discussion

Figure 2 shows that 88.81% ME was produced during transesterification at 333 K with 0.45% KOH (w/w) and at an optimum molar/oil ratio of 9:1. As the values of the chemical reaction parameters go above this value, the ME yield goes down because the reaction can go in both directions. In Fig. 4, the value of slope coefficient (R^2) shows that the first order is the best fit. The correlation coefficients for 323 K and 333 K are 0.9277 and 0.9866, respectively. Figure 5 shows that the transesterification reaction did not follow the second-order kinetic model since the correlation coefficient was 0.9661 at 323 K and 0.9732 at 333 K. Figure 5 shows that the transesterification reaction followed the first Order kinetic model because it had a high correlation coefficient. At 323 K and 333 K, the values of the rate constants K were found to be 0.0196 and 0.0327, respectively. The result shows that the rate constant increased as the temperature increased. Therefore, the yield of FAME starts increasing as the temperature increases.
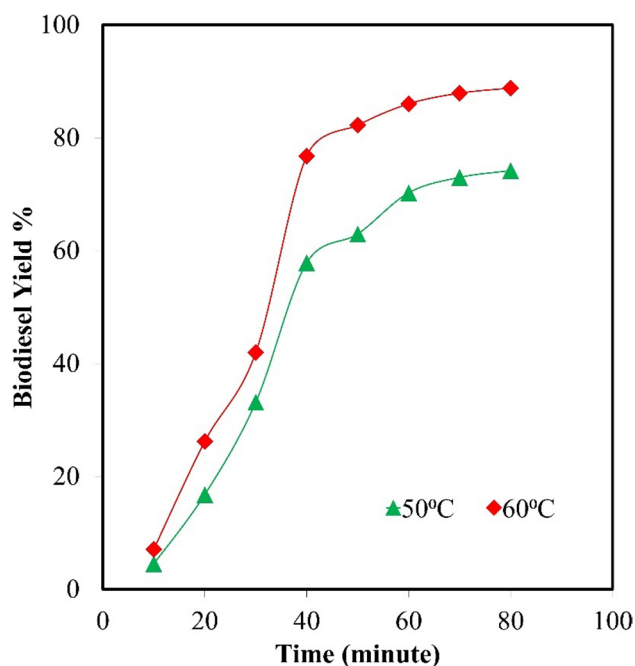
Fig. 2 Effect on biodiesel yield% with a variation of temperature

Fig. 3 Effect on biodiesel yield% with variation of stirring intensity (rpm)

3.1 Biodiesel process optimization

3.1.1 Effect on pure biodiesel yield% due to variation of temperature

Figure 2 shows how the temperature of the process affects the biodiesel yield over time. WCO oil was converted to methyl-ester at a molar ratio of 9:1 (methanol to oil) in the presence of 1.4 g KOH catalyst, and the reaction was carried out at two distinct temperatures of 50 and 60 °C in the presence of 300 rpm of agitation. When studying the transesterification reaction, it is usual practice to carry out the study at temperatures that are somewhat close to the alcohol's boiling point used in the reaction. Pumping and stirring the oil became difficult below 50 °C due to its high viscosity, and it was recommended that oil be stored at a temperature between 50 °C and 55 °C to keep it liquid. Figure 1 shows that the biodiesel yield increases with time, and after 50 min, it is nearly constant. After 60 min, a biodiesel production of 70.24 percent is obtained at 50 °C, compared to an output of 86.01 percent at 60 °C. This is because when the temperature rises, the viscosity of WCO oil lowers, allowing for better interaction with methanol. Glycerol separates and settles out more quickly at higher temperatures, resulting in a faster reaction rate and a higher output of biodiesel.
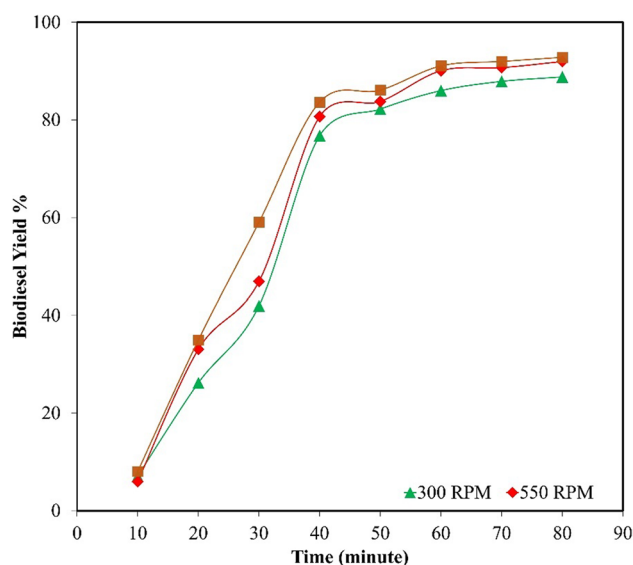
3.1.2 Effect of Stirring speed on Biodiesel yield

While keeping other parameters constant, the influence of stirring intensity on biodiesel yield was tested at 300 rpm, 550 rpm, and 800 rpm. Figure 3 shows how biodiesel yields change with stirring intensity over time. The Figure also shows the stirring intensity impacts on biodiesel yield by escalating the nominal contact area between the two reactants. Increasing stirrer speed shortens reaction time and boosts conversion. Beyond a particular stirrer speed, the yield wouldn't rise. Thus, feedstocks with varying physical qualities require variable stirrer speeds [25]. Figure 3 shows that biodiesel yield increases with agitation speed and does not change much after 50 min. The final biodiesel yield was practically the same when the agitation speed was doubled after 50 min. In this work, 300 rpm is the optimal stirring speed for the reaction.

3.2 Kinetic model

The kinetic analysis that is described here assumes that both pseudo-first and second order irreversible kinetics are at play. The proposed mechanism presupposes that the catalytic process is completely homogenous. In the current investigation, the rate constant of the reaction is determined by taking an appropriate quantity of methyl esters

**Fig. 4** First Order Kinetic reaction at 50 and 60˚c
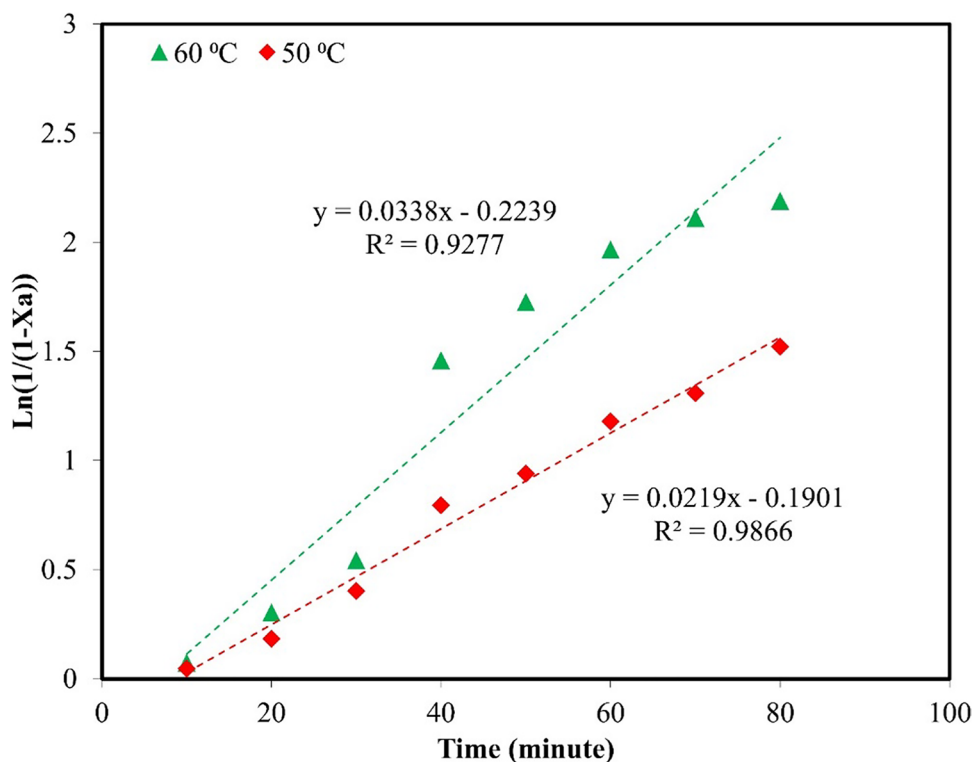
(ME) that were created throughout the reaction; more specifically, the yield of methyl esters as a function of time was employed to investigate the kinetics of the reaction. For the treatment of the kinetic data, the First Order model proposed by Freedman et al. (1986) [5] and the Second Order model proposed by Noureddini and Zhu (1997) [26] were employed, respectively (Figs. 4 and 5).

3.2.1 Determination of reaction order

First order The mathematical relation for the first-order rate constant of the reaction can be written as:

$$Rate = \frac{-dME_A}{dt} = kC^n \tag{7}$$

Fig. 5 Second Order Kinetic reaction at 50 and 60˚c
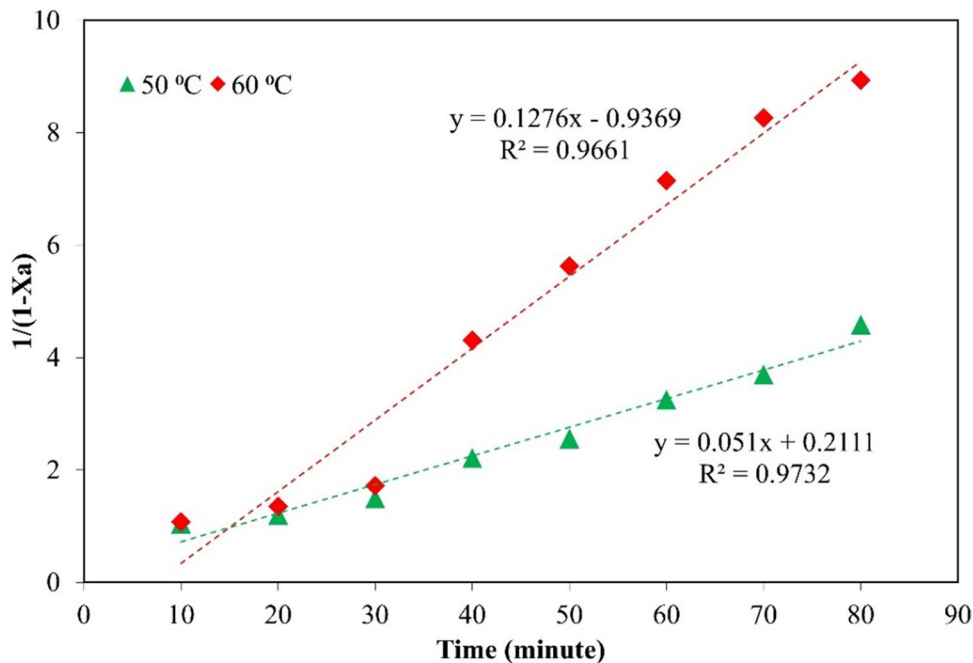
The above mathematical Eq. (7) of first-order may also be written as

$$Rate = \frac{-dME_A}{dt} = kME_A \tag{8}$$

Since Triglyceride (TG) concentration is related to conversion degree.

X_A represents the fractional conversion of the biodiesel (FAME).

$$ME_A = ME_{AO}(1 - X_A)$$

Where ME_A and ME_{AO} are the actual and initial TG concentrations.

$$\frac{-dME_A}{dt} = \frac{dX_A}{dt} = k(1 - X_A) \tag{9}$$

$$\frac{-dX_A}{dt} = k(1 - XA) \tag{10}$$

Integrating the above Eq. 10,

$$\int_0^{X_A} \frac{1}{1 - X_A} = k \int_0^t dt \tag{11}$$

$$-\ln(1 - X_A) = Kt + C \tag{12}$$

where C is the constant of integration and $ME_A = (1 - X_A)$

$$\ln ME_A = -kt \tag{13}$$

The pseudo-first order rate equation can be written as.

$$\ln \frac{1}{(1 - X_A)} = kt \tag{14}$$

where k is denoted as reaction rate constant (min^{-1})

Second order Second order Kinetic model according to Noureddine and Zhu (1997) was used to treat the kinetic data

$$\left(-\frac{r_A}{dt}\right) = \frac{dME_A}{dt} = kME_A{}^2 \tag{15}$$

where $ME.^2 = ME_{AO} \times (1 - X_A)$

$$\frac{dME_A}{dt} = \frac{dX_A}{dt} = kME_{AO}(1 - X_A)^2 \tag{16}$$

where k is denoted as the reaction rate constant (min^{-1})

Separating the variables, we obtain

$$\frac{-dX_A}{(1 - X_A)^2} = kME_{AO}dt \tag{17}$$

The pseudo-second-order equation can be written as.

$$\frac{1}{(1 - X_A)} = kt \tag{18}$$

3.3 Calculation of activation energy

For the determination of the Energy of activation, which is required for the transesterification process of WCO, the Svante Arrhenius theory was applied. The equation which relates activation energy, temperature, and reaction rate constant are expressed mathematically in the Eq. 19:

$$k = Ae^{-Ea/RT} \tag{19}$$

By taking Logarithmic on both sides of the above mathematical Eq. (19) of activation energy,

The final equation obtained is written below:

$$\ln k = \ln A - \frac{Ea}{R} \times \frac{1}{T} \tag{20}$$

where k represents the constant for the reaction rate, A represents the frequency factor of the Arrhenius equation (min^{-1}), R represents the universal/molar gas constant (8.314 Jmol^{-1} K^{-1}), T represents the thermodynamic temperature in kelvin, and Ea represents the energy of activation. The generation of pure biodiesel i.e., from waste cooking oil, was tested at two different temperatures, and KOH was used as the catalyst. The ln k values were determined, and a plot of those values against 1/T was used for the determination of the activation energy (Ea), as shown in Fig. 6. A straight line is obtained when ln k is plotted against 1/T using the Arrhenius method. The activation energy, along with the frequency factor, was determined by taking the slope and the intercept of the linear fit of ln k vs 1/T, respectively. The value that was found for the activation energy of the chemical reaction (Ea) is 45.76 kJmol^{-1}, while the value that was obtained for the frequency factor, A, is 4,95,340.16 min^{-1}. When we talk about a reaction's activation energy, we're talking about the minimal amount of energy that its reacting molecules need to have for the reaction to take place. The lower the activation energy is, the easier it will be for the reaction to take place. Various activation energy values have been recorded in the published research, which pertains to the conversion of WCO to biodiesel, is one of them. However, the activation energy obtained in this work is in the same order as reported in the literature of 56.12 kJmol^{-1} [27], 53.72 kJmol^{-1} [28], 89.7 kJmol^{-1} [29], 113kJmol^{-1}, 148 kJmol^{-1} [30] by making use of a variety of unique approaches, respectively. This calculated activation energy is reasonable; therefore, biodiesel can be made from WCO.

Fig. 6 Arrhenius plot Ln(k) versus 1/T
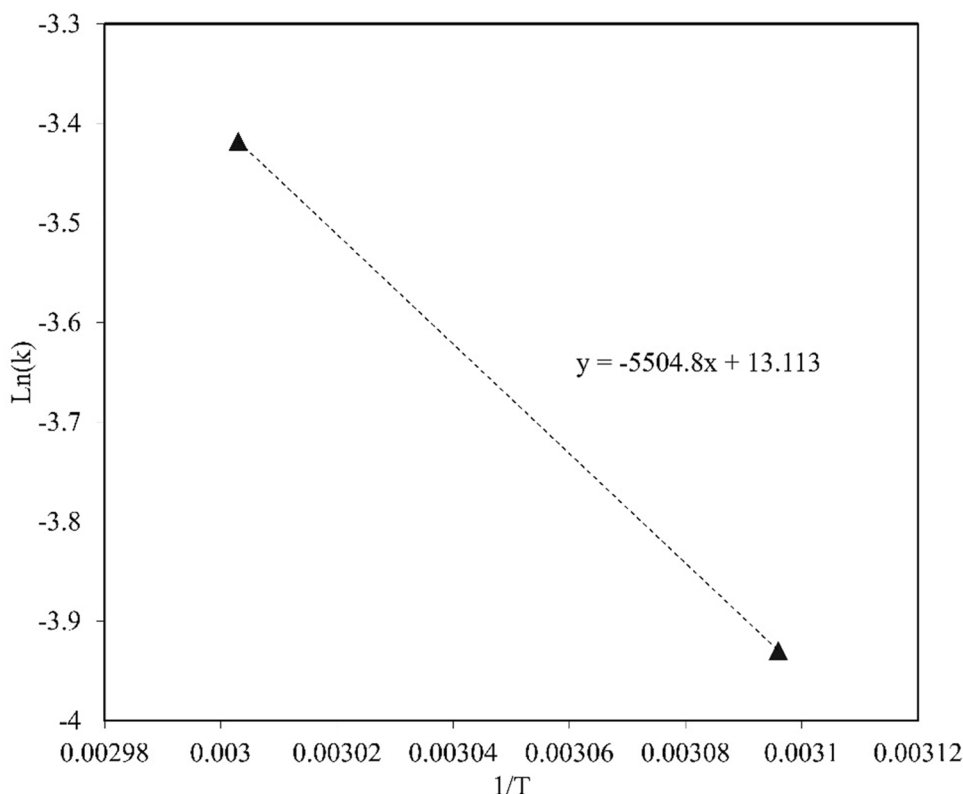
3.4 Thermometric parameters

The equation developed by Eyring-Polanyi to determine the Gibbs free energy (Gibbs) can be expressed as equation.

$$k = \frac{k_b T}{h} \exp\left(-\frac{\Delta G}{RT}\right) \tag{21}$$
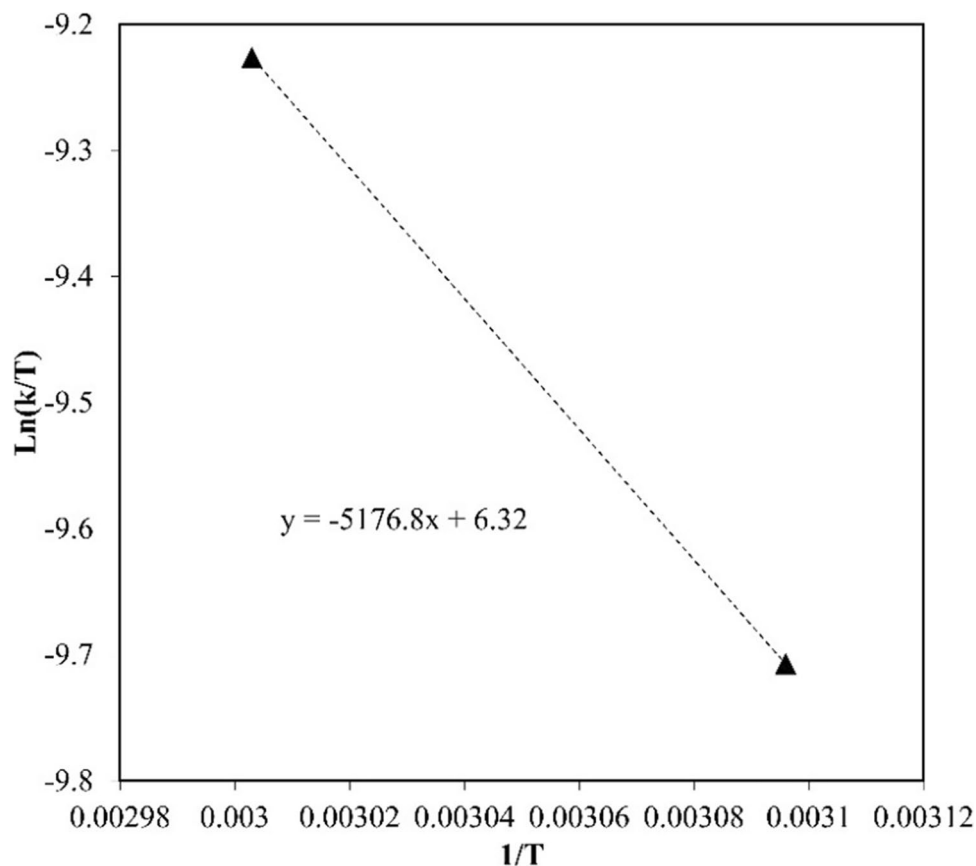
By taking the natural logarithm of above Eq. (19) and substituting $\Delta G = \Delta H - T\Delta S$ into the equation, where ΔH is enthalpy and ΔS is the entropy of activation, respectively, one can write Eq. (21) as follows:

$$ln\left(\frac{k}{T}\right) = -\left(\frac{\Delta H}{RT}\right) + \left[ln K + ln\left(\frac{k_b}{h}\right) + \frac{\Delta S}{R}\right] \tag{22}$$

A universal gas constant of 8.314 J mol⁻¹ K⁻¹ is referred to as R, a Boltzmann constant of 1.38×10^{-23} J/K is referred to as k_b, and a Planck's constant of 6.63×10^{-34} J s is called h. It is common to consider the Transmission Coefficient, K, to be a value of one. The values of change in Gibbs free energy(ΔG), change in Enthalpy (ΔH), and change in Entropy (ΔS) can be computed using Eq. (22), and Eyring plot (Fig. 7) can be used to determine the slope and the intercept of a line between 1/T and ln k/T. Table 1 shows that the values of ΔH and ΔS are 43,039.91 Jmol⁻¹ and -144.99 Jmol⁻¹ K⁻¹,

respectively, based on the data. It was determined that the value of ΔG at 333 K was 91,323.91 J/mol based on the values of ΔH and ΔS. To generate the products, the reactants must be heated to reach the transition state, as shown by a positive value of ΔH, and when ΔS is negative, it demonstrates that the transition state possesses a higher degree of organized geometry and alignment than the reactants do in the ground state. Negative values of ΔG imply an endergonic reaction that was not spontaneous. There are numerous studies in the literature describing the thermodynamic parameters that have been conducted so far, as shown in Table 1, and their S. platensis algae biodiesel has different ΔH and ΔS values than biodiesel made from Chlorella algae [31] along with the leather tanning waste, this is because the manufacture of biodiesel is done in a variety of ways [32]. Chlorella oil was extracted and transesterified in two steps by Ahmad et al. at 60 °C [31]. At 250–325 °C, Ong et al. used supercritical conditions to transesterify the leather tanning waste oil [32]. Furthermore, this research work is considered on the basis of the single-stage biodiesel extraction process and transesterification of algae biomass, i.e., S. platensis, at a specific temperature of 55 °C for the final production of pure biodiesel. A change in the method used to produce biodiesel alters reaction parameters, which in turn alters thermodynamic characteristics.

Fig. 7 Eyring plot

Figure: Eyring plot with y-axis labeled Ln(k/T) ranging from -9.8 to -9.2, and x-axis labeled 1/T ranging from 0.00298 to 0.00312. Linear fit equation shown: y = -5176.8x + 6.32

4 Conclusion

Using the Arrhenius equation, the activation energy and frequency factor were determined, and at different temperatures, thermodynamic parameters were also taken into consideration in terms of Gibbs free energy, enthalpy, and entropy. In this research work, a Single-step transesterification reaction is used to prepare the biodiesel from waste cooking oil with 0.45% w/w KOH as a catalyst and at a 9:1 methanol/oil ratio. The conversion rate of WCO was obtained maximum at 60°C temperature and 300 rpm stirring speed. The kinetic study was carried out with first and second-order kinetic models, amongst which the first-order kinetic described the best model according to R^2. The activation energy of the chemical reaction (Ea) obtained is 45.76 KJmol^{-1} which is comparable to the values procured in other studies. The numerical values of thermodynamic parameters ΔG, ΔH, and ΔS were found to be 91.32 KJmol^{-1}, 43.04 KJmol^{-1}, and -144.99 Jmol^{-1} K^{-1} respectively, which shows that the reaction was not spontaneous, thermodynamically favorable, and endergonic in nature. Studies have revealed that biodiesel made from waste cooking oil is a more suitable and environment-friendly alternative to fossil fuels.

4.1 Future scope

Kinetic study involves the analysis of the rate of reaction and the factors that affect it. This technique can be used to optimize the conversion of waste cooking oil into biofuels

Table 1 The thermodynamic parameters that were considered in the final production of pure biodiesel

Biodiesel (B100)	ΔG (kJ/mol)	ΔH (kJ/mol)	ΔS (J/mol K)	Ref. No
Waste cooking oil	91.32	43.04	-144.99	This research
Spirulina platensis	92.71	16.35	-232.83	[33]
Leather tanning waste	153.64–171.16	31.37	-233.71	[32]
Chlorella	96.12	36.12	-180.83	[31]

and to identify the most efficient catalysts and reaction conditions. Thermometric analysis on the other hand involves the measurement of heat flow during a chemical reaction. This technique can be used to determine the energy content and calorific value of waste cooking oil and its derived biofuels. In addition, the use of waste cooking oil as a biofuel can help to reduce waste and promote a circular economy. Therefore, there is a growing interest in the scientific community and the industry to explore the potential of waste cooking oil as a renewable energy source, and kinetic study and thermometric analysis are likely to play an important role in this area in the future.

Data availability The authors state that the article contains the data that supported the findings.

Declarations

Ethical approval Not applicable.

Conflicts of interest The authors stated that they have no conflicts of interest.

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