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Optimization and kinetics of biodiesel from jojoba (*Simmondsia chinensis* L. Schneider) oil

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ABSTRACT

Kinetic studies for the production of biodiesel from jojoba oil and methanol in the presence of biocatalyst Novozyme 40013 (lipase from *Candida antarctica*) has been studied in the present research investigation along with optimization of reaction parameters. Optimum conditions identified for the present transesterification reaction are 5:1 molar ratios of alcohol to jojoba oil, 65 °C temperature with 5% concentration of catalyst for 7 hrs of reaction duration and 600 rpm of mixing intensity which contributed 95.12% conversion. Pseudo homogeneous first order kinetic model has been applied to fit the experimental data and results have been analysed on the basis of model data. Equilibrium constants (K) have been evaluated through this kinetic model using optimized reaction parameters. By analyzing the kinetic model, the frequency factor and the activation energy of the transesterification reaction have been determined. The properties of jojoba biodiesel and diesel fuel are in good agreement which encourages us to use it as an alternative fuel in future.

***Keywords*:** *Candida antarctica*, Kinetic model, Jojoba oil, Equilibrium constant, *Simmondsia chinensis*, Biodiesel

1. INTRODUCTION

Production and utilisation of renewable energy sources throughout the world are supposed to be the most challenging job at present. Scarcity of non-renewable energy sources

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and present environmental degradation have compelled to think us more rigorously for the sources of alternative energy [1-5]. It has been observed that world energy consumption doubled between 1971 and 2001 and also been estimated that the energy demand will increase 53% by the year 2030 [6]. The main reason is the consumption of fossil-fuel resources within 40–60 years if the present consumption rate remains constant [7]. Moreover, the price hiking of fossil fuels has been considered as a serious threat to countries with limited financial and economic resources. Several alternatives of energy sources [8-10] are used and elevating progress is still in concern. A statistical review shows that renewable energy (including biofuels) posted a record increase in consumption in energy terms which was also the largest increment for any source of energy in 2019. Wind energy provided the largest contribution to renewables growth followed closely by solar energy. By country, China was the largest contributor to renewables growth followed by the US and Japan [11].

India is a promising country regarding the production of biofuels from vegetable oils. Biofuel like biodiesel has several advantages over diesel fuel. It is environmet friendly, biodegradable and renewable fuel. Other advantages of biodiesel are transportability, low sulphur and aromatic content, high combustion efficiency and high cetane number. So as a fuel, it creates considerable attention throighout the world for the last few decades. Different edible and non-edible vegetable oils and fats are used for this purpose [12-16]. Non edible oils like jatropha curcas oil [17-20], karanja oil [21, 22], waste cooking oils [23-25] etc are mainly used for biodiesel production [39-41]. Production of biodiesel can be done using chemical or biocatalyst. Biocatalyst confers several advantages over chemical catalyst. Use of biocatalyt requires low temperature which saves lot of energy as the recation proceeds for 7-8 hrs. Chemical catalyst requires higher temperature for biodiesel conversion. Apart from saving enery, biocatalyst produce no or minimum by product which is a big advantage because in that case, seperation of ultimate product would be easier. Biocatalyt can be recycled many times which minimizes the cost of production. So utilisation of biocatalyst for the production of biodiesel not only saves energy and cost but also the process is clean and recyclable. Among the non edible vegetable oil sources, jojoba (Simmondsia chinensis L. Schneider) oil has been recognised as an important sources for biodiesel production and creates a lot of attention for this purpose. Jojoba is a shrub which grows in many parts of the world especially in the semi-arid region of the Sonora desert in northern Mexico and the south-western USA [26]. It is now cultivated in many countries like United States, Israel, Mexico, Peru, Australia, India, Egypt, Thailand, South West Africa, Costa Rica, Argentina, Chile and other countries [27].

Jojoba seeds (**Figure 1**) contain 40 to 60% of oil which is known as liquid wax (esters of long chain alcohol and long chain acid) unlike most vegetable seed oils that are composed of triglycerides [28]. "Jojoba oil" and "jojoba wax" are generally used interchangeably due to the visual appearance of wax in the oil but as a wax it is composed almost entirely with mono-esters of long-chain fatty acids and alcohols along with a little fraction of triglycerides.

The chemical composition of jojoba oil accounts for its extreme shelf-life stability and extraordinary resistance to high temperatures compared with true vegetable oils. Most important use of jojoba oil-wax are in the cosmetics and pharmaceutical industry, but it can be identified as new energy crops for the last few decades [29]. Abdelmoez *et al*. [30] used jojoba oil for biodiesel production through process modeling and simulation. Hamamre and Salaymeh [31] studied the physical properties of different bends of jojoba oil with fuels like biodiesel and diesel. Taiseer *et al*. [32] optimized biodiesel production from jojoba oil using red sea coralline limestone as a heterogeneous Catalyst.

Jojoba oil as biofuel was also studied by other researchers [33-35]. Some researchers studied the kinetics for the transesterification reaction of jojoba oil for the production of biodiesel. Sánchez *et al*. [36] studied the kinetics of jojoba oil methanolysis using a waste from fish industry as catalyst.

Kinetics of biodiesel production from other non edible vegetable oils are studied by many researchers. Present authors also studied the kinetics of biodiesel from soybean oil deodorizer distillate and identified the activation energy and frequency factor of the transesterification reaction [37]. Roy *et al*. [38] studied the effect of mass tranfer kinetics of biodiesel from jatropha curcas oil through mathematical approach. Very little studies have been found for the kinetics of biodiesel production from jojoba oil using enzyme catalyst. Present research has been done on optimization of biodiesel from jojoba oil along with kinetics analysis of reaction parameters in the presence of enzyme catalyst Novozyme 40013. Very good conversion has been achieved in the present research investigation which encourages future researchers to start thinking about this line of approach.

Figure 1. Jojoba plant and seed

2. EXPERIMENTAL

2. 1. Materials

Jojoba oil was collected from Aromatic Herbals Private Limited, Kolkata, West Bengal. Novozyme 40013, an immobilized nonspecific lipase from *Candida antarctica* was used as catalyst in the reaction with ester synthesis activity of 10000 propyl laurate unit/g. Methanol was purchased from Scientific and Laboratory Instrument Co., Kolkata. Except otherwise specified all other chemicals were A.R. Grade.

2. 2. Methods

2. 2. 1. Transesterification of jojoba oil

Initially 500 mL of jojoba oil was filtered and taken in an Erlenmeyer flask and heated up to 80 °C to drive off moisture by continuous stirring for about 1 h. After that, methanol was

added to it for transesterification reaction through stepwise manner in an appropriate proportion using solvent hexane at a specified temperature for 7 hours. Enzyme Novozyme 40013 was added in definite proportion (w/w) to the reaction mixture. The progress of reaction or production of biodiesel was monitored by thin layer chromatographic (TLC) method and the typical yield of each reaction product was determined separately by column chromatography using silicic acid as an adsorbent and 160 mL of hexane diethyl ether (99:1) as eluting solvent.

At the end of the reaction, the product was filtrated through separating funnel to remove the enzyme and allowed to separate. The lower layer was then evaporated under vacuum in order to remove excess methanol and the final product was collected. The enzyme was washed with hexane, dried and reused for the next experiment. Biodiesel characterization was done according to the American Standard Testing Method (ASTM). Values are reported as mean ± s.d., where n = 3 (n = no of observations).

2. 2. 2. Gas chromatographic analysis

Fatty acid composition of jojoba oil was determined by a gas liquid chromatographic (GLC) method after converting into methyl ester. The HP 5890A GLC was connected with a HP 3390A data integrator. The GLC was fitted with a glass column (1.83 m X 3.175 mm id) packed with 10% DEGS supported on Chromosorb – WHP (100/200 mesh) of HP make. The oven temperature was programmed from 100 to 190 °C at 5° per min. The injector and detector block temperatures were maintained at 230 and 240 °C, respectively. IOLAR-2 nitrogen was used as the carrier gas (flow rate 30 mL/min). The fatty acid esters peak was identified and calibrated with standard methyl esters. Data were represented an averages of three determinations.

3. RESULTS AND DISCUSSIONS

3. 1. Analysis of jojoba oil

The physicochemical properties and fatty acid profiles of jojoba oil are shown in **Table 1** and **Table 2** respectively. It has been observed from **Table 1** that jojoba oil has higher flash point at nearly 280 °C with a kinematic viscosity of nearly 24 mm/s^2. Higher iodine value (81.29) agrees with higher percentage of unsaturated part of this oil. It is shown from **Table 2** that the eicosenoic acid is the major FFA present in jojobal oil. It represents approximately 67.32% of the on weight basis. Apart from that, erucic acid content is much lower than that but it is the second highest which contributes 12.41 %. Oleic acid and behenic acid are also present in almost same amount accounting to approximately 7%. Lauric acid and palmitic acid are also present in small amounts.

Table 1. Physicochemical properties of jojoba oil

Properties	Value	Test method
Specific gravity	0.863±0.003	ASTMD 1298
Flash point (°C)	279±0.772	ASTMD-93

Kinematic viscosity (mm/s^2)	23.89±0.101	ASTM-D445
Acid value (mg KOH/g)	0.67±0.004	ISO 660-2009
Ash content (% w/w)	0.057±0.002	ASTMD 2584
Heating value (MJ/Kg)	49.11±0.372	ASTMD 4868
Iodine value (g/100g)	81.29±0.361	ISO 3961- 2009
Saponification value	93.12±0.405	ASTMD 1962

Table 2. Fatty acid composition of jojoba oil

Fatty acid	Amount (%)
Lauric acid (C12:0)	2.05±0.008
Palmitic acid (C16:0)	0.44±0.002
Oleic acid (C18:1)	7.35±0.009
Eicosenoic acid (C20:1)	67.32±0.402
Behenic acid (C22:0)	7.02±0.005
Erucic acid (C22:1)	12.41±0.101

3. 2. Analysis of parameters for biodiesel production and kinetic study

3. 2. 1. Analysis of molar ratio of alcohol to jojoba oil

Different molar ratios of alcohol to jojoba oil have been studied for the conversion of jojoba oil to biodiesel. Among the molar ratios from 1:1 to 6:1 (MeOH: Jojoba oil), it has been observed that 5:1 molar ratio is the optimum ratio for the conversion process as shown in **Figure 2**. Enhancing the ratio of alcohol to jojoba oil does not increase the conversion as evidenced from the **Figure 2.** It may be due to the fact that increasing the amount of alcohol does not enhance the possible collision among reactants with the catalyst which is essential for higher conversion.

Kinetic studies of the conversion of biodiesel for molar ratio 5:1 (MeOH : Jojoba oil) based on pseudo-homogeneous first order model by varying time of reaction shows that a plot of log (a-x) vs time (as shown in **Figure 3**) gives slope of -0.1700 which identifies the value of rate constant (K) of 0.3915 hr^{-1} at temperature 65 °C in the presence of 5% biocatalyst. Duration of reaction time has been optimized for 7 hrs of continuous stirring with 600 rpm.

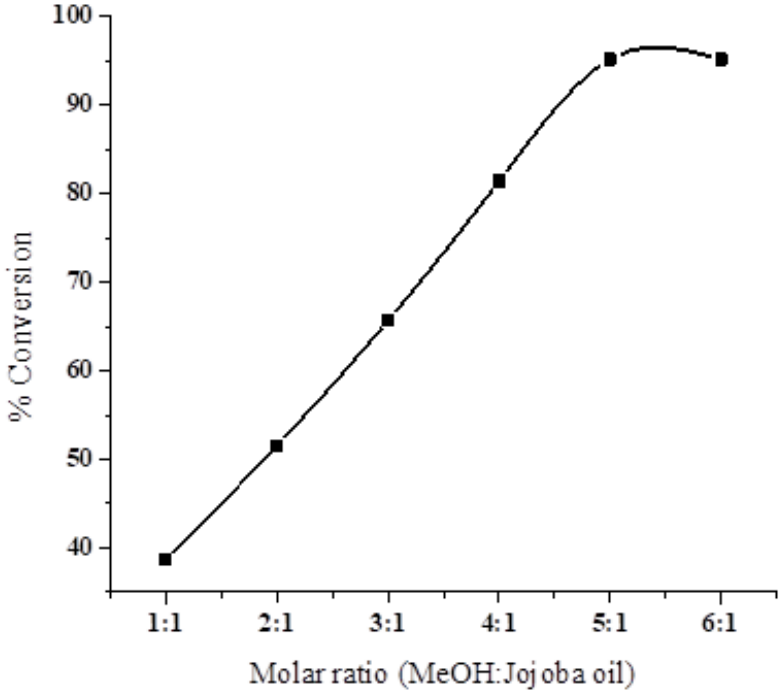

Figure 2. Analysis of molar ratio of alcohol to jojoba oil vs % conversion of biodiesel

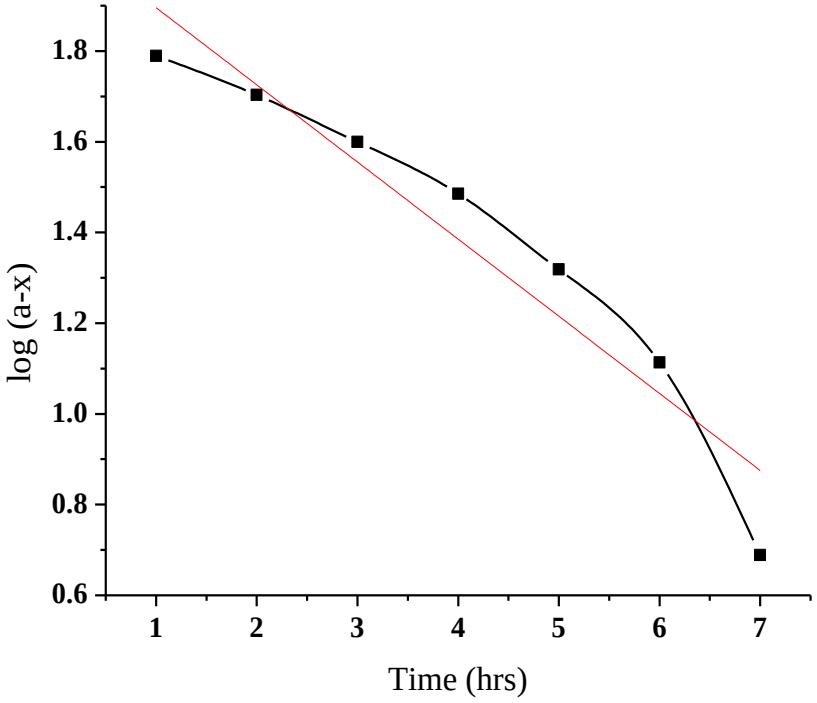

Figure 3. Kinetic analysis of plot of log (a-x) vs time (hrs) with molar ratio 5:1 (MeOH : Jojoba oil)

3. 2. 2. Analysis of temperature of reaction

Dynamics of transesterification reaction depends on activation energy of the reaction and the activation energy is direclty proportional to applied temperature of transesterification reaction.

So temperature study is important in this respect. For the conversion of jojoba oil to biodiesel with the reaction with methanol, a range of temperature from 35 °C to 75 °C have been studied using 5% biocatalyst for 7 hrs of continuous stirring with 600 rpm. Molar ratio of 5:1 (MeOH : Jojoba oil) has been maintained all through the reaction. The study has been envisaged through **Figure 4** and it has been observed that 65 °C is the optimum temperature for biodiesel conversion from jojoba oil.

Enhancing tempertaure does not increase the conversion rate as the enzyme shows its maximum efficiency at a certain temperature. Beyond that particular temperature, probably the deactivation of enzyme starts which has an effect on the rate of conversion of biodiesel.

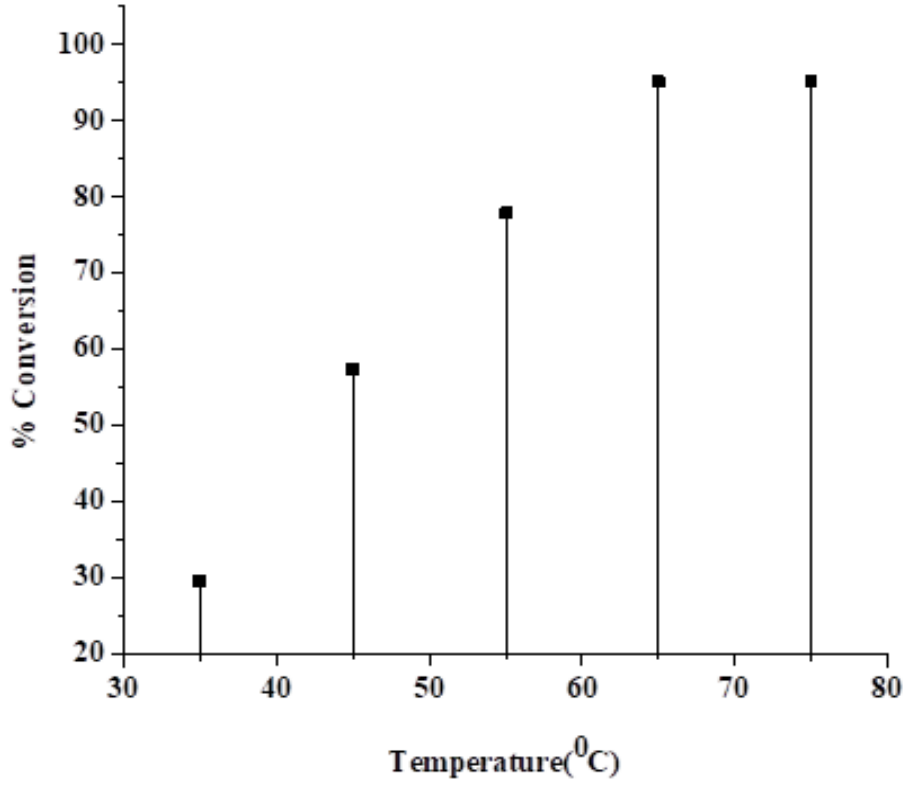

Figure 4. Analysis of temperature for % conversion of biodiesel

Kinetic studies of the conversion of biodiesel for temperature 65^0C based on pseudo-homogeneous first order model by varying time of reaction shows that a plot of log (a-x) vs time (as shown in **Figure 5**) gives slope of -0.1847 which identifies the value of rate constant (K) of 0.4254 hr^{-1} at the molar ratio 5:1 (MeOH : Jojoba oil) in the presence of 5% biocatalyst. Duration of reaction time has been optimized for 7 hrs of continuous stirring with 600 rpm.

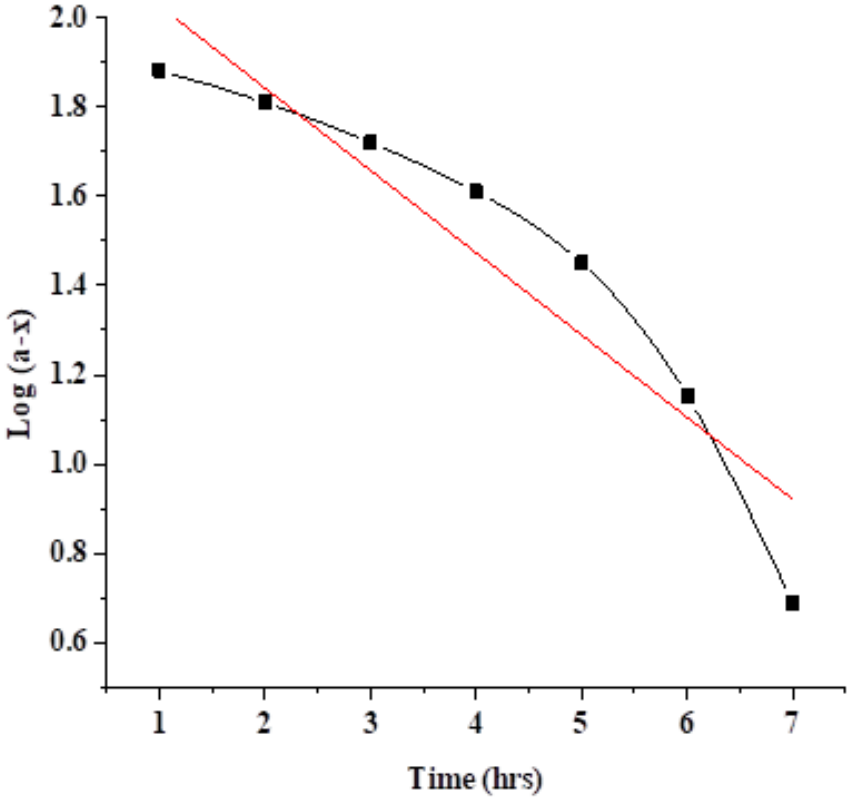

Figure 5. Kinetic analysis of plot of log (a-x) vs time (hrs) at temperature 65 °C

3. 2. 3. Enzyme concentration

Catalyst concentration, here, enzyme Novozyme 40013 plays a big role for the conversion of jojoba biodiesel. In our present study, by changing the concentration of enzyme from 2-6% at the temperature of 65 °C for 7 hrs of continuous stirring with 600 rpm maintaining a molar ratio of 5:1 (MeOH : Jojoba oil), optimum concentration has been identified which is shown in **Figure 6**.

It has been observed from the figure that 5% concentration of enzyme Novozyme 40013 contributed optimum conversion for biodiesel production from jojoba oil through transesterification reaction with methanol. Increasing the concentation of enzyme beyond 5% does not affect the conversion rate. This may be due to the agglomeration of enzyme which inhibits the interaction between reactants and active sites of enzyme.

Kinetic studies for the conversion of biodiesel in the presence of 5% enzyme concentration based on pseudo-homogeneous first order model by varying time of reaction shows that a plot of log (a-x) vs time (as shown in **Figure 7**) gives slope of -0.1848 which identifies the value of rate constant (K) of 0.4255 hr^{-1} at the molar ratio 5:1 (MeOH : Jojoba oil) at 65 °C temperature. Duration of reaction time has been optimized for 7 hrs of continuous stirring with 600 rpm.

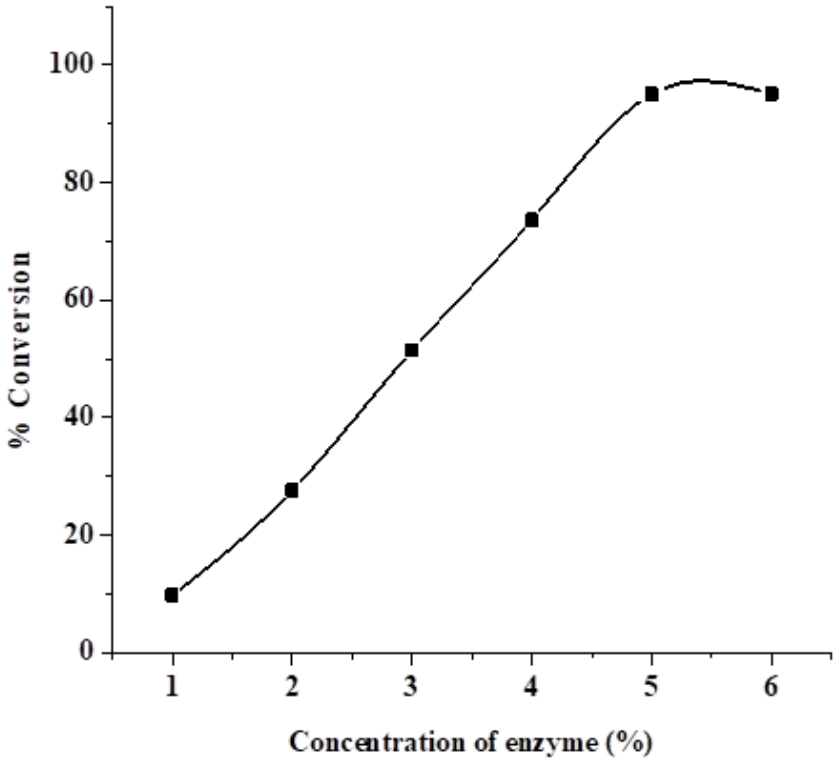

Figure 6. Analysis of enzyme concentration for % conversion of biodiesel

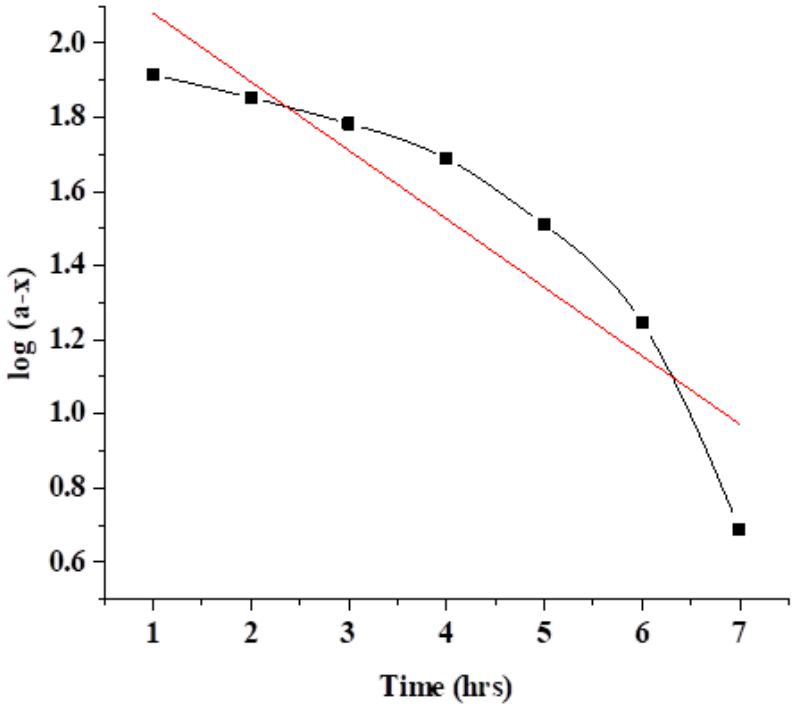

Figure 7. Kinetic analysis of plot of log (a-x) vs time (hrs) at 5% concentration of enzyme

3. 3. Activation energy and frequency factor

The identification of activation energy of any reaction is much important because it shows the minimum energy which is required to start a reaction. In the present study, energy of activation for the reaction was evaluated from the slope of Arrhenius plot (**Figure 8**) of log K vs 1/T and was found to be 23.445 kJ mol^{-1}. The frequency factor or pre-exponential factor is another important parameter which represents the frequency of collision between reactant molecules. Here the frequency factor obtained from the kinetic results was 1.053×10^4 collisions s^{-1}

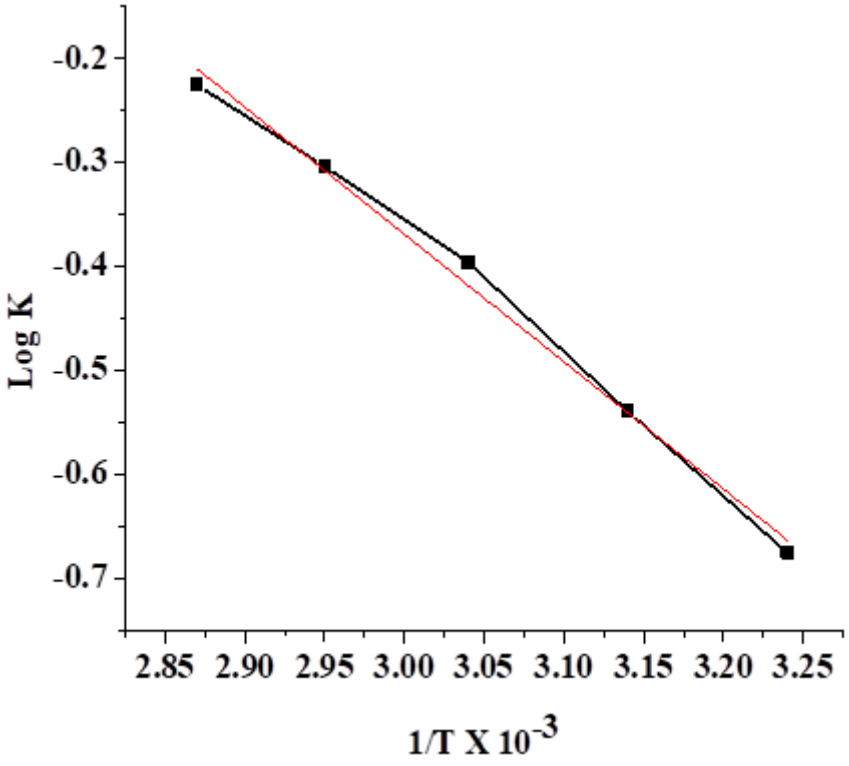

Figure 8. Plot of log K vs 1/T

3. 4. Physicochemical properties of jojoba biodiesel

Table 3. Physicochemical properties of jojoba oil

Property	Jojoba biodiesel	Diesel fuel
Specific gravity	0.872	0.74
Flash point (°C)	157	52-96
Kinematic viscosity (mm/s^2)	5.61	1.3

Acid value (mg KOH/g)	0.15	0.1
Ash content (%w/w)	0.09	0.01-0.02
Heating value (MJ/Kg)	36.81	45

Physicochemical properties like specific gravity, flash point, kinematic viscosity, acid value, ash content and heating value are compared for jojoba biodiesel and diesel fuel as shown in **Table 3**. It has been observed from **Table 3** that the characteristics of biodiesel are comparable with biodiesel standards in most of the properties. Higher flash point of biodiesel is significant due to safe handling than diesel fuel. The calorific value of diesel fuel is higher than biodiesel but with regard to other characteristics, jojoba biodiesel can be used safely without modification of engines.

4. CONCLUSION

Jojoba oil has been utilized for the production of biodiesel through transesterification reaction with methanol in the presence of biocatalyst. Initially the physicochemical characteristics and fatty acid composition of jojoba oil were analysed. Reaction parameters like molar ratio of methanol to jojoba oil, temperature and concentration of enzyme have been optimized along with the kinetic analysis. For fitting the experimental results, a pseudo-homogeneous kinetic model was employed and it is in agreement with the observed optimum conditions. The activation energy and frequency factor for the experimental condition of conversion of biodiesel from jojoba oil and methanol for the forward reaction were found to be 23.445 kJ mol^{-1} and 1.053×10^4 collisions s^{-1} respectively.

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