Preparation of biodiesel from soybean oil by using heterogeneous catalyst

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Abstract
The predicted shortage of fossil fuels and related environmental concerns has recently attracted significant attention to search alternative fuel. Biodiesel is one of the alternatives to fossil fuel. Now-a-days, most biodiesel is produced by the transesterification of oils using methanol and a homogeneous base catalyst. The use of homogeneous catalysts is normally limited to batch mode processing followed by a catalyst separation step. The immiscible glycerol phase, which accumulates during the course of the reaction, solubilizes the homogeneous base catalyst and therefore, withdraws from the reaction medium. Moreover, other difficulties of using homogeneous base catalysts relate to their sensitivity to free fatty acid (FFA) and water and resulting saponification phenomenon. High energy consumption and costly separation of the catalyst from the reaction mixture have inspired the use of heterogeneous catalyst. The use of heterogeneous catalysts does not lead to the formation of soaps through neutralization of FFA and saponification of oil. In the present paper, biodiesel was prepared from crude (soybean) oil by transesterification reaction using heterogeneous base catalyst name calcium oxide (CaO). Various reaction parameters were optimized and the biodiesel properties were evaluated.
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1. Introduction
Majority of the worlds energy needs are supplied through petrochemical sources, coal and natural gases, with the exception of hydroelectricity and nuclear energy, of all, these sources are finite and at current usage rates will be consumed shortly [1].
Diesel fuels have an essential function in the industrial economy of a developing country and used for transport of industrial and agricultural goods and operation of diesel tractor and pump sets in agricultural sector. Economic growth is always accompanied by commensurate increase in the transport. The high energy demand in the industrialized world as well as in the domestic sector and pollution problems caused due to the widespread use of fossil fuels make it increasingly necessary to develop the renewable energy sources of limitless duration and smaller environmental impact than the traditional one. This has stimulated recent interest in alternative sources for petroleum-based fuels. An alternative fuel must be technically feasible, economically competitive, environmentally acceptable, and readily available. One possible alternative to fossil fuel is the use of oils of plant origin like vegetable oils and tree borne oil

seeds. Vegetable oil is one of the biomass resources and is used as a feedstock of an alternative to fossil diesel fuel. The alternative fuel, which is termed as ''Biodiesel'', consists of fatty acid methyl esters produced by transesterifying vegetable oil with methanol [2]. In addition to the renewable nature peculiar to the biomass resources, biodiesel has another advantage of the good fuel properties: high flash point, good lubricity and so on [3, 4]. In 2003, European Community has decided to replace at least 5.75% of the yearly consumed fossil fuels with biofuels, by the year 2010. This decision accelerated the use of biodiesel and its production has been constantly growing. The total of biodiesel yearly produced in the world was 7.75 million metric tons in 2008 [5].

For the existent biodiesel production process, vegetable oil is transesterified with the help of homogeneous base catalysis of alkali hydroxide dissolved in methanol. The base-catalyzed transesterification is faster than the acid-catalyzed one for which sulfonic acid or p-toluenesulfonic acid is employed [6].

However, for the existent process, these are some technological problems resulting in costly production of biodiesel. The typical problem is a massive amount of the waste water, which is due to the purification to wash the homogeneous catalyst off the crude biodiesel with water. And besides, emulsification of biodiesel occurs during the purifying operation, which causes not only obstruction of the process operation but also loss of biodiesel.

With a view of resolving the technological problems mentioned above, Kusudiana and Saka [7, 8] studied a catalyst-free process in which vegetable oil was transesterified with super-critical methanol. It took only 4 min. to convert rapeseed oil into biodiesel, even though the high temperature (523–673 K) and high pressure (35–60 MPa) were required for making methanol reach the supercritical state. The very fast transesterification was due to the large solubility of vegetable oil in super-critical methanol and intensified nucleophilic nature of super-critical methanol. Apart from the catalyst-free process, the enzymatic process can be applied to biodiesel production [9-11]. Although biocatalyst consisting of lipase allows vegetable oil to be transesterified into its methyl esters at room temperature, it takes more than 24 h to achieve the perfect conversion [12].

Solid base can leads to the heterogeneous catalytic process, which promises the cost reasonable biodiesel production. Since the solid base catalyst is easy to separate from the transesterified product. The solid base catalyst is active in the transesterification at the temperature around boiling point of methanol. For the purpose of studying the heterogeneous catalytic process, many researchers tested a variety of solid base for the catalytic activity. Xie et al. [13] prepared potassium loaded on alumina as the solid base catalyst and the prepared catalyst was employed for transesterification of soybean oil at reflux of methanol. Under the same transesterifying condition, they examined the solid base catalysis of the magnesium–aluminum (Mg–Al) mixed oxide that was prepared by calcining the corresponding hydrotalcite [14]. Shibasaki-Kitagawa et al. [15] elucidated that anion-exchange resin catalyzed a transesterification of triolein which is a model of vegetable oil.

From the economical point of view, we have focused our attentions on calcium oxide (CaO) as a candidate for the solid base catalyst. A major source of calcium oxide is limestone having the advantages of good availability and cheap cost. Moreover, calcium oxide can be prepared from the waste matters consisting of calcium carbonate, such as mollusk shells. The use of the waste matters is not only effective in enhancing the cost advantage of CaO catalyst but also related to recycle of the naturally mineral resources.

In this paper, biodiesel was prepared from soybean oil by heterogeneous solid catalyst e.g. CaO. Biodiesel conversion was measured by the measurement of viscosity. The reaction parameters were optimized. The kinetics of CaO-catalyzed transesterification of soybean oil and methanol was studied. The reaction rate constant (k) at optimal reaction condition was determined. A pseudo-first order kinetic model was proposed for the system and the experimental data are fitted in this model.

2. Materials and methods
2.1 Materials
Soybean oil was obtained from City Oil Mill (City group Bangladesh Ltd.). The soybean oil was of the 875.3 g mol^{-1} average molar mass having the following fatty acid distribution: alpha-Linolenic acid (C-18:3) 7–10%; linoleic acid (C-18:2) 51%; and oleic acid (C-18:1) 23%. It also contains the saturated fatty acids stearic acid 4% and palmitic acid 10% [16]. CaO was obtained from Merk (Germany) Ltd. Methanol (technical pure) was supplied by Merk (Germany) Ltd. Sodium hydroxide, hydrochloric acid,

iso-propanol, potassium hydroxide were purchased from Merk (India) Ltd. All the reagents were analytical grade.

2.2 Purification of raw oil
Raw crude soybean oil contains gum, coloring materials and other impurities. To remove these impurities from the crude oil adsorption technique was applied. For this purpose, a column adsorption technique was applied. In a column active charcoal was placed and oil was passed through this column. Typically 20 gm active charcoal was placed in a column of 1 inch diameter and oil was passed under vacuum.

2.3 Catalyst preparation
Catalyst was prepared from reagent grade CaO. For this purpose 50 gm of CaO was taken in a crucible and calcined in a muffle furnace at 450 0C temperatures for 3h before use for the reaction. At this temperature all the impurities present in CaO was converted to the corresponding metal oxide.

2.4 Calibration curve for determining conversion by viscosity method
Biodiesel conversion can be measured by different methods. Zhang et. al. [17] measured the biodiesel conversion by GC analysis and Kusdiana and Saka [7] measured the biodiesel conversion by HPLC method. Here a new technique was proposed for biodiesel yield measurement e.g. viscosity measurement method. At first certain amount of soybean oil undergoes base catalyzed transesterification reaction, described by Saydut et. al. [18] and biodiesel was produced. This produced biodiesel were separates and again undergoes base catalyzed transesterification and the produced biodiesel was assumed 100% biodiesel and viscosity was measured. Then different proportion of raw soybean oil was blended with produced biodiesel and the viscosity of each blend was measured. And the percentage conversion was calculated from the viscosity change. Viscosity versus percentage conversion plot was presented in Figure 1 which used for the determination of conversion giving transesterification reaction.
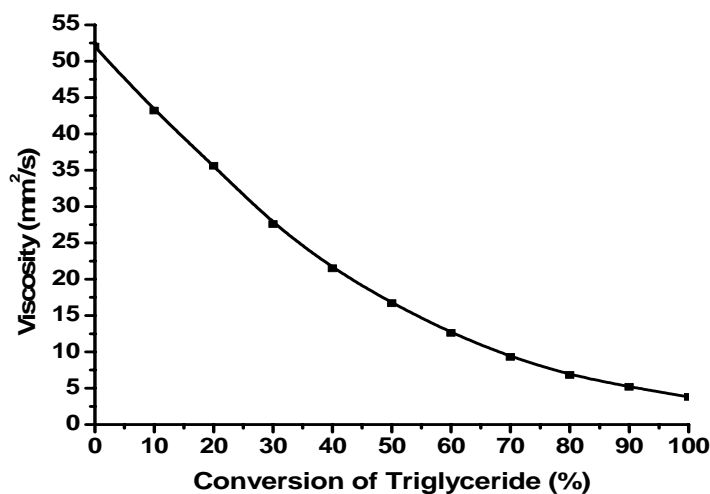

Figure 1. Viscosity Vs Conversion of Biodiesel. [Viscosity measured at 40°C, Reaction temp. 60°C, oil / methanol ratio 1:6, Catalyst (NaOH) concentration 1% wt of oil, under reflux with vigorous stirring, Reaction time 90 min]

2.5 Transesterification of soybean oil with methanol
A 500 ml three-neck glass flask with a magnetic stirrer, a watercooled condenser, and a thermometer was used for the transesterification reaction. Typically, soybean oil (43.8 g, 0.05 mol) and methanol (19.3 g, 0.60 mol) were placed displaced into the flask at a predetermined temperature. To the stirred solution, CaO (3.51 g, 0.063 mol) was added to initiate the reaction. At the end of reaction, the catalyst was removed by filtration and the residual methanol was separated from the biodiesel by vacuum distillation.

2.6 Kinetics experiment
Kinetics experiments were performed in a 1000 ml four-necked flask equipped with a magnetic stirrer, a water-cooled condenser, a thermometer and a sampling port. The molar ratio of methanol to soybean oil

and the catalyst amount were fixed under the optimal condition (molar ratio of methanol to soybean oil: 12:1; catalyst amount: 8.0%). The total reaction volume was 380 ml and was regarded as constant during transesterification process considering the volume of samples withdrawn was no more than 1% of the total reaction volume. Sample was filtered through filter paper to separate catalyst. Viscosity of the sample was measured and hence the reaction conversion was measured.

2.7 Analytical methods for oil and biodiesel
FFA in the oil and biodiesel samples was analyzed by the method described in AOCS Aa 6-38. To determine FFA of sample, 4-5 g of samples were dispersed in isopropanol (75 ml) and hexane (15 ml) followed by titration against 0.25 N NaOH. Saponification value (SV) was determined by the method described by Jeffery *et al.*, [19] 1gm sample was taken with 25 ml alcoholic KOH, heated for one hour in a steam bath with occasional shaking and titrated the excess KOH with the 0.5 M hydrochloric acid. The iodine value (IV) were determined by titrating 0.01 N sodium thiosulfate to the mixture of tested fuel and chemical reagents until the disappearance of the blue color based on the analysis methods of American Oil Chemist's Society [20]. IV was calculated by the following Eq. 1:

$$\text{Iodine value} = (B - S) \times N \times 0.001269 / W \tag{1}$$

where, *S* and *B* are the amounts (in unit of ml) of sodium thiosulfate titrated for the tested sample and blank sample, respectively; *N* is the molar concentration (in unit of mol/L) of sodium thiosulfate; and *W* is the weight (in unit of g) of the tested sample.
Physical properties such as color, moisture content and density of the oil were determined by following ASTM D 1500, ASTM D 1744 (Karl fisher method), ASTM D 1480/81 and ASTM D 240 respectively. Viscosity, flash point, pour point and cloud point were determined by standards ASTM D445, ASTM D 93 (Pensky- Martens Flashpoint Apparatus, Lazer Scientific Inc, Germany), ASTM D 2500 and ASTM D 97 respectively.

3. Result and discussion
3.1 Characterization of soybean oil
The properties, such as the specific gravity, kinematic viscosity, free fatty acid content, moisture content, saponification value, ester value, iodine value, odor, color was measured by the methods was mentioned above and presented in Table 1.

Table 1. Raw material (**Soybean oil**) specification

Property name	Value
Specific gravity, at 25°C	0.92
Kinematic viscosity (mm^2/s), at 40°C	52.0
Kinematic viscosity (mm^2/s) at 40°C after adsorption	50.0
Free fatty acid content (%)	4.5
Moisture content (%)	0.08
Saponification value (mg KOH/gm oil)	194
Iodine value	120
Odor	Mild flavor

3.2 Study of reaction system
It is well known that methanol is polar solvent. In our study, CaO was used as a catalyst. . Because the presence of catalyst in solution implies a contribution of the homogeneous reaction, it is necessary to evaluate the catalyst CaO against dissolution in this reaction medium. In order to evaluate the contribution of soluble species to the overall catalytic activity, the conversion of oil to biodiesel after removing of catalyst was also studied as shown in Figure 2. It was obvious that the conversion of oil to biodiesel increased with increasing of reaction time. Once we removed the catalyst from the reaction medium after 2.0 h of reaction, the conversion of oil to biodiesel was not enhanced significantly, from which we could conclude that the soluble species was negligible and had no effect on the overall catalytic activity. The base catalyst CaO could be used as heterogeneous catalyst in the reaction system generally.
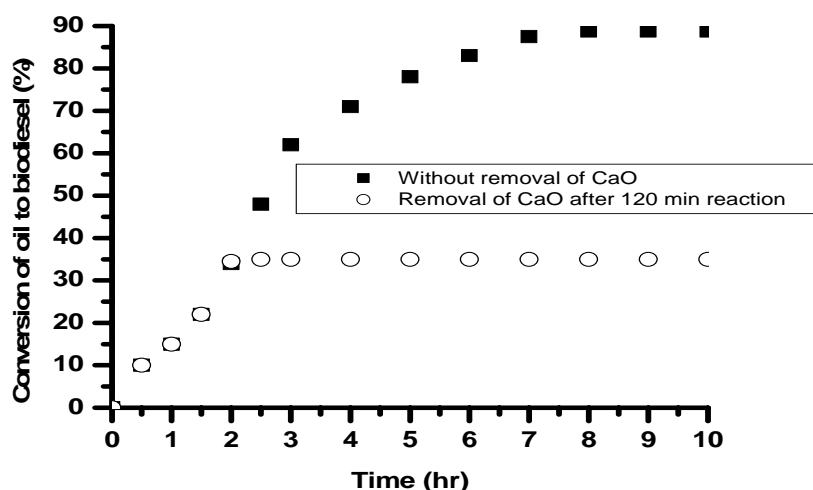
Figure 2. Study of solubility of CaO in methanol by measuring biodiesel conversion after removal of catalyst [Reaction temperature 65^0 C, molar ratio of oil to methanol = 1:12, catalyst concentration 8 wt% of oil, under reflux with vigorous stirring]

3.3 Transesterification reaction

Transesterification of soybean oil with methanol by heterogeneous catalyst is relatively slow and needs activation either by high temperature to achieve a higher conversion. All the reactions were carried out at refluxing temperature. Effects of reaction parameters on the conversion of biodiesel were explored as follows.

3.3.1 Effect of oil to methanol molar ratio

The oil to methanol molar ratio is one of the most important variables affecting the conversion of transesterification reaction of soybean oil to biodiesel. It was evident that the transesterification of soybean oil required three molars of methanol for each mole of oil. Since the reaction was a reversible reaction, methanol was used in huge excess with respect to soybean oil in order to drive the reaction towards the formation of biodiesel. As can be seen from Figure 3, the biodiesel conversion increased with increasing of the molar ratio of methanol to oil over the range from 3:1 to 12:1. The highest conversion (88.8%) was obtained at the molar ratio of 12:1 for 8 h. With further increasing of the molar ratio, a little decrease in the biodiesel conversion was observed. This effect could be attributed to that excessive methanol dilute the concentration of oil in the reaction system, thus reducing the frequency of collision of soybean oil and the catalyst CaO. Therefore, the optimal molar ratio of methanol and soybean oil was to be 12:1.
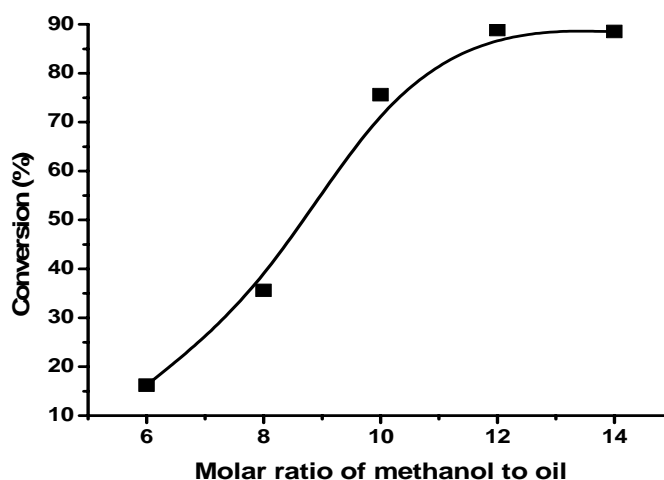

Figure 3. conversion of soybean oil to biodiesel at different methanol to oil molar ratio [Reaction temp. 65°C, Catalyst concentration 8% wt of oil, vigorous stirring, Reaction time 8.0 hr]

3.3.2 Effect of catalyst concentration on transesterification

The conversion of soybean oil to biodiesel is greatly affected by catalyst concentration. The effect of the amount of catalyst on the conversion was investigated within the range of 3.0-12.0% CaO (based on the oil weight) and the results are illustrated in Figure 4. It was found that, the biodiesel conversion was not more than 10% when the catalyst amount was less than 5.0%. As we all know, the catalytic activity was extremely influenced by alkalinity and the amount of basic sites. When CaO was used as the catalyst, the more of the catalyst amount, the more of the basic sites, which is favorable for biodiesel conversion. With increasing of the amount of catalyst from 5.0% to 8.0%, the conversion of biodiesel increased rapidly, which could be attributed to the availability of more basic sites.

When the catalyst amount was above 8.0%, the conversion of biodiesel was almost not enhanced anymore, indicating that the catalyst amount was enough for the transesterification of soybean oil. Too much catalyst could only make the mixture of reactants too viscous, leading to the problem of mixing and separation. In order to avoid this problem and reduce the costs, the optimum amount of the catalyst was to be 8.0% CaO (on the basis of oil weight).
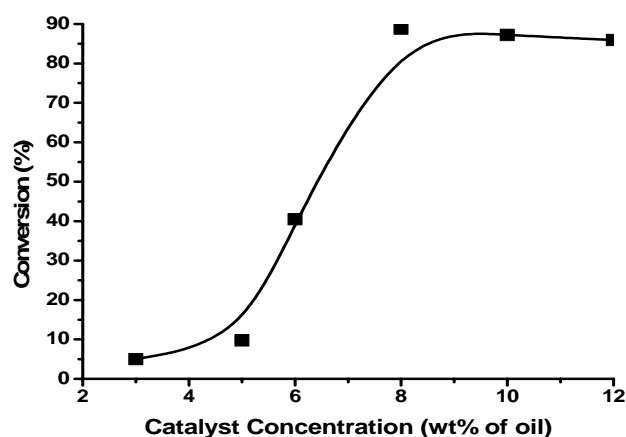

Figure 4. conversion of soybean oil to biodiesel at different catalyst concentration [Reaction temp. 65°C, oil to methanol ratio 1:12, vigorous stirring, Reaction time 8 h]

3.3.3 Effect of temperature on transesterification

Temperature is another important variable that effecting the conversion of oil to biodiesel and the conversion increases with increasing of temperature. Usually higher temperature is required for hetero generous catalysed transesterification system. The reaction was conducted from 40-70^{0}C temperature and the results are represented in Figure 5. When the reaction temperature was less than 40^0 C the conversion was less than 30%. With increasing the temperature from 40^0 C to 65^0 C the conversion of biodiesel increased rapidly. When the temperature was above 65^0 C the conversion of biodiesel was almost not enhanced anymore, indicating that the temperature was enough for the transesterification of soybean oil. The optimum temperature was to be 65^0 C.
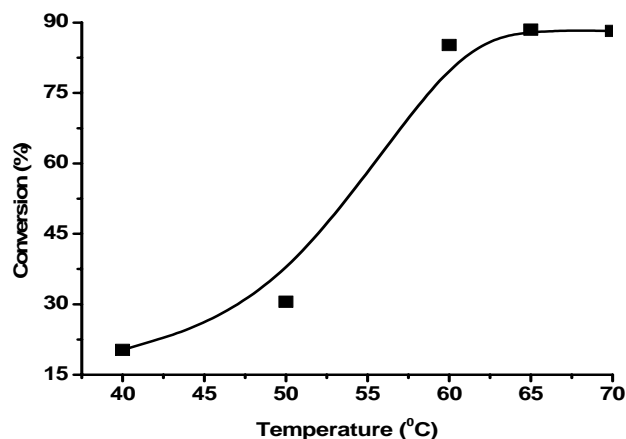

Figure 5. conversion of soybean oil to biodiesel at different temperature [oil to methanol ratio 1:12, catalyst concentration 8.0% CaO, under reflux with vigorous stirring, Reaction time 8 h]

3.3.4 Effect of reaction time on the conversion of oil to biodiesel
The effect of reaction time on the conversion of biodiesel at the catalysis of CaO was studied and the results are shown in Figure 6. The reaction time was varied within the range from 0 h to 10 h. A gradual increase in the biodiesel conversion was observed with the increasing of time up to 8 h , and then remained nearly constant. The optimum reaction time was 8 h, at which the highest biodiesel conversion could reach 88.9%.
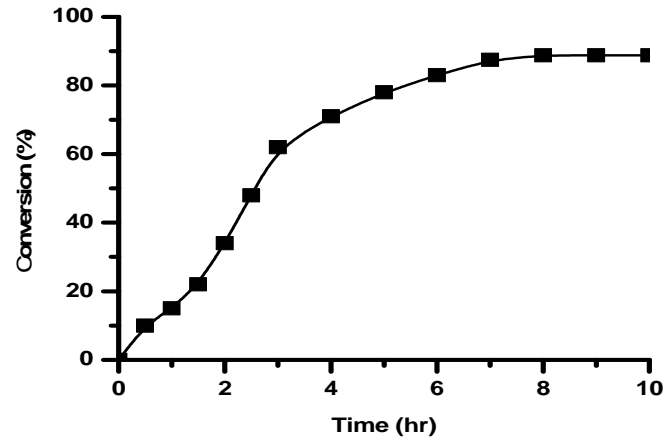

Figure 6. conversion of soybean oil to biodiesel at different reaction time [oil to methanol ratio 1:12, reaction temperature 65^0 C, catalyst concentration 8.0% CaO, under reflux with vigorous stirring]

3.4 Kinetic study
The overall transesterification reaction can be written in following form

TG (oil) + 3* Methanol $\leftrightarrow$ 3* ME (biodiesel) + G (Glycerin)

This reaction is assumed to proceed in the pseudo first-order reaction as a function of the conversion of oil (TG). The equilibrium constant can be determined by the following relationship

$$K = \frac{C_{Ge}}{C_{TGe}} = \frac{X_{TGe}}{1 - X_{TGe}} = \frac{k_1}{k_2}$$

(2)

where, X_{TGe} is the equilibrium conversion of TG
The rate expression can be expressed as below,

$$\frac{dC_G}{dt} = -\frac{dC_{TG}}{dt} = C_{TG0} \times \frac{dX_{TG}}{dt} = k_1 C_{TG0}(1 - X_{TG}) - k_2 C_{TG0} X_{TG}$$

(3)

Substituting equation (2) in to (3) we get,

$$\frac{dX_{TG}}{dt} = k_1 [1 - aX_{TG})$$

(4)

where, $a = 1 + \frac{1}{K}$

By integrating Equation (4) we get,

$$-\frac{1}{a} Ln(1 - aX_{TG}) = k_1 t$$

(5)

Or,

$$X_{TG} = \frac{1}{a}(1 - Exp(-k_1 at))$$

(6)

A plot $-\frac{1}{a}Ln(1-aX_{TG})$ vs. t gives a straight line, as shown in Figure 7 and k_1 is determined from the slop of the line and the value was 0.470 hr^{-1}. The value of equilibrium constant was determined from the experiment and the value was 21.22. The value of $k2$ is determined from Equation (2) and the value was 0.022 hr^{-1}.

The theoretical conversion vs time is determined by putting the values of the rate constants and concentrations at different time in Equation (3). The theoretical and experimental kinetic plot is presented in Figure 8. It can be seen that the overall reaction follows the pseudo first-order reaction kinetics.
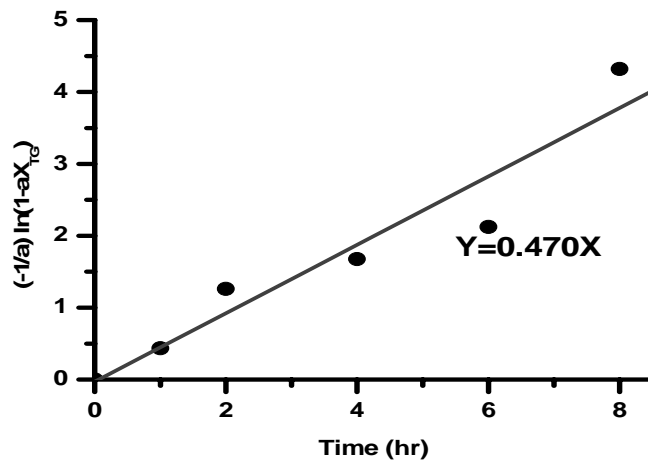

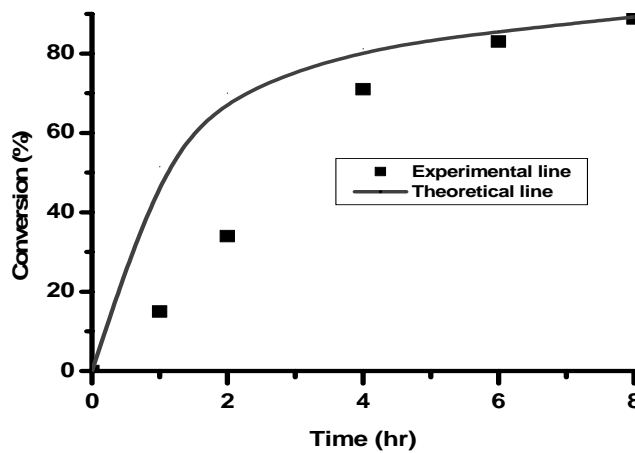Figure 7. Determination of k$_1$ at optimal reaction condition

Figure 8. Conversion of biodiesel-Time curve for transesterification reaction [Reaction temp. 65°C, oil to methanol ratio 1:12, Catalyst concentration 8.0% wt. of oil, under reflux with vigorous stirring with vigorous stirring]

3.5 Properties of biodiesel
Properties of the produced biodiesel and comparison with biodiesel standard are given in Table 2.

Table 2. Properties of biodiesel produced from soybean oil and comparison with standard value

Properties	Produced biodiesel value	Biodiesel Standard [21-22]	Diesel standard [22]
Specific gravity, at 25 ^{o}C	0.85	0.88 (at 15.5^0 C)	0.85(at 15.5^0 C)
Kinematic viscosity (mm^2/s), at 40°C	5.4	1.9–6.0	1.3 – 4.1
Free fatty acid content (%FFA)	0.5	-	-
Calorific value (Mj/Kg)	38.2	37.5	42
Moisture content (%)	0.12	0.05% max.	0.161
Flash point (^{0}C)	135	100 to 170	60 to 80
Cloud point (^{o}C)	-3	-3 to 12	-15 to 5
Pour point (^{o}C)	-6	-15 to 10	-35 to -15
Iodine value	88	-	-
Saponification value	194	-	-
Cetane no	48	-	-

4. Conclusion

In the present study, CaO was identified as an effective heterogeneous catalyst for the transesterification of soybean oil and methanol. The effects of reaction conditions (molar ratio of methanol to soybean oil, catalyst concentration, reaction temperature and reaction time) on biodiesel conversion were studied and the optimum condition was identified, which could provide valuable experimental data for future industrial application. Kinetics of this CaO-catalyzed transesterification reaction was investigated at optimal reaction condition. Finally biodiesel was produced at the optimum condition and the physical properties of the produced biodiesel were evaluated and compared with biodiesel and petro-diesel standard.

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