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Effect of Reaction Time and Temperature on Biodiesel Production from Vegetable Oil

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Abstract

An alternative fuel made from renewable resources such as leftover cooking oil, vegetable oils, and animal fats is called biodiesel. With the world's population growing and the need for diesel fuel rising, the search for alternative fuels is becoming increasingly important. The process used in this study's experiment is turning corn oil into biodiesel. Sodium hydroxide (NaOH) was chosen as the catalyst, and methanol was utilised as the alcohol. This project aims to analyze the properties blended in biodiesel, such as yield percentages, density, kinetic viscosity, and F-TIR spectrum, according to different conditions of reaction time and reaction temperature. Reaction time took 1 and 2 hours through different temperatures, 50 °C and 60 °C to produce 4 samples each. Ingredients to make analyzed biodiesel in this experiment were 400 ml of corn oil, 101.97 ml of methanol, and 2.79 g of sodium hydroxide (NaOH). 4 samples were produced in analysis determination. Sample 1 was done under 1 h and 50 °C, sample 2 was done under 1 h and 60 °C, while sample 4 was done under 2 h and 50 °C. Lastly, sample 4 was done under 2 h and 60 °C. The best result sample biodiesel yield was sample 3 out of all samples produced using a 2-hour reaction time at 50 °C. In the meantime, the density of each sample produced is about the same, and the kinematic viscosity increases with temperature over the course of two hours of reaction time. Consequently, it is discovered that as the temperature rises, the yield decreases with each reaction time. The best sample in terms of kinetic viscosity is sample 1. Nonetheless, the goal of producing a percentage of biodiesel production between 72% and 75% was accomplished. Hence, this study can improve renewable energy with less hydrocarbon emissions for a healthy future.

1. Introduction

One renewable energy source is biodiesel, which is made from non-edible oil, vegetable oil, and animal fats. The creation of liquid fuels from renewable sources has received increased attention lately, and biodiesel has been proposed as one of those fuels because of its potential to be produced in a carbon-neutral manner. The manufacture of liquid fuels from renewable sources has received increased attention lately, and biodiesel has been proposed as one of those fuels because of its potential for carbon-neutral production [1]. Since biodiesel is oxygenated, the gases it releases during burning are virtually the same gases the plant absorbed from the atmosphere during its growth it is recommended that diesel derived from petroleum be replaced with it. By

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trans esterifying vegetable or animal fat with alcohol such as methanol or ethanol in a batch process, biodiesel is created. It is renewable, biodegradable, and favorable to the environment [2].

In this study, biodiesel was created by the transesterification method utilizing corn oil combined with alcohol and NaOH as a catalyst through 4 different conditions, which are 1 and 2 hours for each 50 and 60 °C. A researcher completed a transesterification procedure in six hours, aside from that. On the other hand, biodiesel dissolves slowly and its yield percentage decreases over time [3]. If transesterification is done under high-temperature values, saponification is enhanced, lowering the yield of biodiesel [9]. Consequently, the characteristics of the biodiesel were investigated after the reaction time was reduced from six hours to one or two hours. The purpose of this research is to investigate the properties of density, kinetic viscosity, yield percentage and biodiesel F-TIR spectrum.

The reaction time studied for alkali transesterification at different time intervals ranging from 0.5-6 hours revealed that about 2 hours of reaction was sufficient for the completion of the alkali transesterification process and optimum conversion of palm olein vegetable oil to biodiesel (about 80%) [3]. Numerous studies have shown that reaction times are invariably connected to the synthesis of biodiesel. Put another way, the amount of biodiesel produced increases with reaction time. However, a two-hour response period yields the highest amount of biodiesel. This tendency is linked to the production of more soap during longer reaction times during the production of biodiesel. The reversibility of the transesterification reaction, which facilitates the reaction between methanol and glycerol to produce soap, is the source of soap creation [11].

2. Materials

Daisy corn oil is purchased from a nearby supermarket that works as a raw material. Next, the chosen methanol as the solvent in the oil assists in the breakdown of the base catalyst. 0.75wt.% of NaOH and drops of acetic acids were used as a catalyst and neutralizer. Alcohol and catalyst were supplied by the UTHM Faculty Fuel Laboratory. Biodiesel that was produced had been calculated through the 6:1 alcohol-to-oil conversion ratio. Table 2 presents the material description relative molecular mass and density of raw oil and alcohol and Figure 1 displays the Daisy Corn Oil as the raw material.

Table 1 Material description

Process condition	Values
Relative Molecular Mass Corn Oil	885.48 g/mol
Relative Molecular Mass Methanol	32.04 g/mol
Density of Corn Oil	0.93 g/mol
Density of Methanol	0.79 g/mol

Fig. 1 Daisy Corn Oil

2.1 Experimental Design

This project describes in detail the process settings that include the dilution of the alcohol-catalyst combination, the transesterification process, the separation process, the filtering and purification steps, and the analysis test for the qualities of biodiesel. There were 4 samples produced at the end. Sample 1 was done under 1 h and 50 °C, sample 2 was done under 1 h and 60 °C, while sample 4 was done under 2 h and 50 °C. Lastly, sample 4 was done under 2 h and 60 °C. Two distinct temperatures were employed in the transesterification process: 50 °C and 60 °C. One- and two-hour increments of reaction times were recorded for each temperature, and the mixing rate remained constant at 600 rpm during the whole procedure. By the project's conclusion, four viable samples had been produced. The yield %, F-TIR (Perkin Elmer Spectrum 100, UK), density at 15 °C, and kinematic viscosity at 40 °C were all measured and examined for this biodiesel. Every detail about the chemical substance and parameter that was used was written, as seen in Table 2.

Table 2 Transesterification process condition

Process condition	Values
Corn oil, (ml)	400 ml
Methanol (ml)	101.97 ml
Sodium Hydroxide (NaOH), (g)	2.792 g
Reaction time, (h)	1h, 2h
Reaction temperature, (°C)	50 °C, 60 °C
Rate of mixing	600 rpm

2.2 Preparation of Alcohol-Catalyst Mixture

Alcohol, catalyst, and unrefined vegetable oil were combined to create biodiesel at a consistent temperature, mixing rate, and reaction time. Methanol was the alcohol of choice for this experiment. NaOH was the catalyst utilized in this experimental transesterification to create biodiesel. First, 101.97 ml of methanol is put into a beaker. Next, using an electronic weighing scale, weigh a tiny beaker of sodium hydroxide (NaOH) at approximately 2.792 g. Stir the methanol at 600 rpm while diluting the NaOH once heated to between 40 and 45 °C.

2.3 Transesterification Process of Corn Oil and Alcohol-Catalyst Mixture

Firstly, fill a pot with pipe water and heat on a hot magnetic plate. The three-neck flask is placed inside the filled water pot such that the flask's base touches the pot's base. So that the magnetic stirrer can easily spin smoothly inside. Subsequently, the condenser is affixed to the center of the flask with three necks, and the rubber pipe is linked to the water flow both in and out. A condenser was used to maintain the pressure inside the three-neck flask and avoid water vapor and explosion. Three-neck flask and condenser must be clamped perfectly to the retort stand to avoid fall issues. Next, add 400ml of corn oil inside the three-neck flask and started to heat the oil until it gets 50 °C at first sample. Other than that, the second hole of the three-neck flask must be shut with a cork and thermometer to control the temperature. The corn oil must be heated slowly by regulating the hot plate knobs. The corn oil is stirred under 600 rpm conditions. As the corn oil was heated to 50 °C, pour the alcohol-catalyst mixture into the hot corn oil and start the 1-hour timer as it is the first sample. Figure 2 indicates the transesterification process with the complete apparatus.

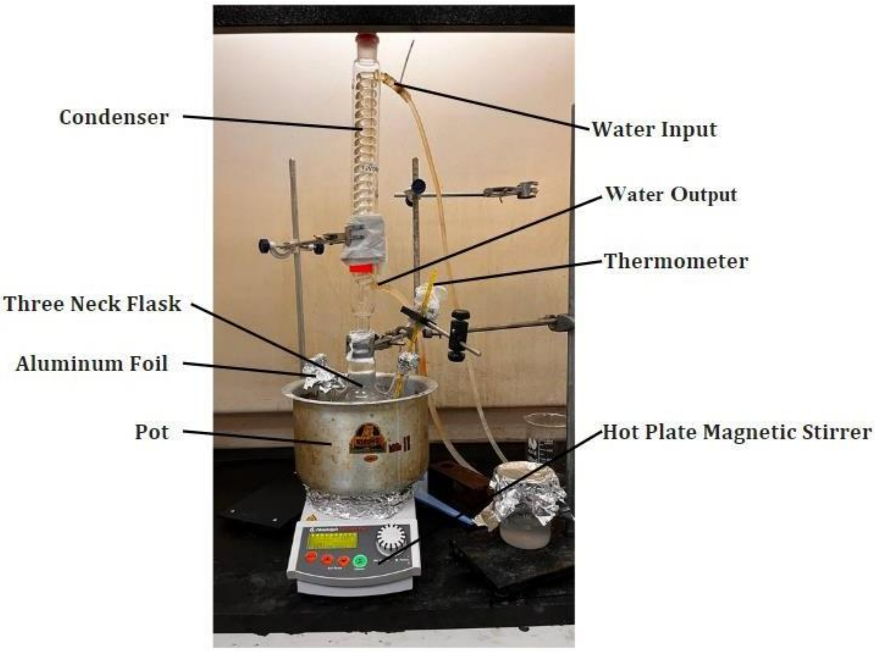

Fig. 2 Complete apparatus transesterification process

2.4 Phase Separation of Biodiesel

Following the transesterification procedure, the ester needs to be let to cool in a beaker for approximately five minutes after pouring the three-neck flask through the third hole. The observation's result is that two layers are visible in the beaker when the ester cools down after five minutes. After that, transfer the ester mixture into a separation funnel and leave it there for a night to separate. The separation is complete after a night. It is possible to draw conclusions from the observation that two layers were formed inside the separating funnel. Glycerol is the bottom product while biodiesel is the top product. The primary goal is understanding how biodiesel is made and glycerol is discarded.

2.5 Filter Biodiesel Product

In addition to methyl ester, biodiesel also contains a lot of other products such excess methanol, water, hydrocarbons, excess NaOH, and glycerin. Biodiesel with high levels of excess content needs to be filtered using standard grade quantitative filter paper with a radius circle of 15 mm. The purpose of a biodiesel filter is to remove superfluous material from the fuel.

2.6 Purification

After testing all four samples, the pH values ranged from 8 to 9. It indicates that the biodiesel was alkaline before neutralization. The biodiesel needs to be neutralized in order to get its pH down to 7, which can be achieved by mixing it with distilled water and one to three drops of acetic acid. Measure 400 ml of distilled water into a beaker, then add one to three drops of acetic acid.

The neutralized step involved adding acidic distilled water to the biodiesel after it had been poured into a one-liter beaker and gently stirred. After that, pour once more into the separation funnel to let the water and biodiesel separate for about a minute. After that, discard the acidic distilled water that has formed at the bottom of the product. Reintroduce the biodiesel into the beaker lastly. If the pH of the biodiesel was still alkaline, the neutralization procedure was repeated a second time.

When the neutralization procedure is complete, a pH test must be performed to ensure the pH is 7. Warm distilled water must be used twice to wash neutral biodiesel. The distilled water should first be heated to 50 °C before being poured into a one-liter beaker. To remove extra methanol and excess acid, softly swirl and pour. Biodiesel has to be cleaned till the distilled water becomes transparent.

Biodiesel still has surplus content that needs to be removed after washing. The final stage in biodiesel processing is drying. By applying a lot of heat and keeping the temperature at 101 °C for an hour, excess content in biodiesel can be eliminated.

2.7 Determination of Biodiesel Properties

Testing the qualities of the biodiesel was done to ascertain whether the production of biodiesel was accomplished and whether it complies with international standards. Fourier Transform Infrared Spectroscopy (F-TIR), flash point, kinematic viscosity, and density at 15°C are used to analyze the biodiesel. The international biodiesel standard was compared to determine the density and kinetic viscosity of the sample. In order to obtain averages for more accurate values, each property was performed twice.

3. Result and Discussion

3.1 Determination of Density at 15 °C

The difference between the pycnometer's initial and final weights yields the density value. The weight in (g/cm^3) is the total difference between the pycnometer's starting and final weights. In general, one can alter a condition's temperature or pressure to alter the density value. Density values will rise in response to pressure increases. In order to prevent losses in this experiment, it is then recommended to adjust the temperature to 15 °C. This serves as a reference point for density testing. The outcome is acquired by weighing the biodiesel using a pycnometer to determine how much biodiesel was created. Table 3 displays the outcome. The density of the product falls between 0.8878 g/cm^3 and 0.8799 g/cm^3. Nonetheless, in accordance with the international EN ISO 12185 and EN ISO 3675 standards. The value's density should be between 860 and 900 g/cm^3. The density data for each production biodiesel sample is presented in Table 3 and is compared to the international standards EN ISO 3675 and EN ISO 12185. The density bared graph in Figure 3 initially increases when reaction time and temperature increase.

Table 3 Data collected for density for each sample

Sample	Density (kg/cm^3)	Density, EN ISO 3675 EN ISO 12185 (kg/cm^3)
1	878	
2	889	860 to 900
3	878	
4	879	

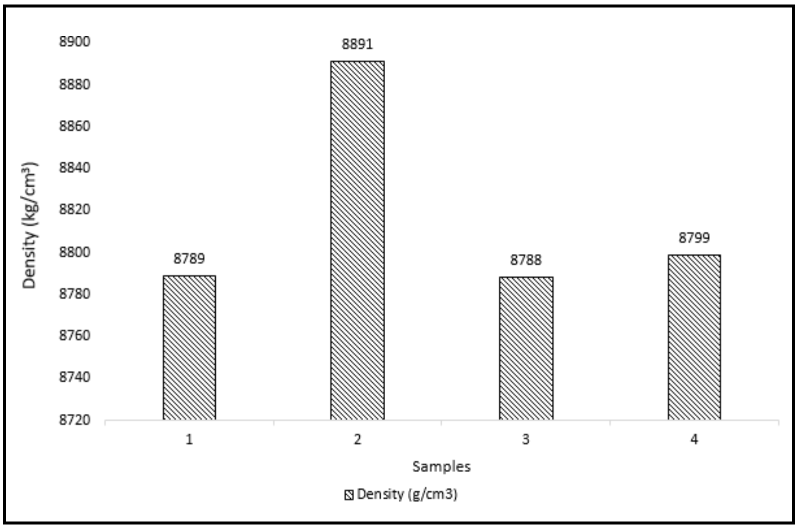

Fig. 3 Density against sample graph

First, the precise characteristics needed to produce biodiesel are displayed in Table 1. Every sample was assembled using the worldwide EN ISO 12185 and EN ISO 3675 standards. It can be said that every sample met the density standard. Every sample displays a density that is nearly identical to 0.88 g/cm^3. With reference to the graph shown in Figure 1, Sample 3 had the lowest density of the four samples. Previous study indicates that a decreased density can lead to an increase in fuel consumption. This indicates that biodiesel is more efficient if the density value is near 860 kg/cm^3 [4]. Upon closer inspection, the graphic illustrates that density increases with temperature and reaction time. The biodiesel analysis done by the previous researcher was a little bit different. According to Rusdianasari, who produced biodiesel from leftover cooking oil, the density decreases with increasing reaction temperature [5]. Apart from that, the graph shows that a greater density value corresponds with a longer reaction time during transesterification. When compared to the other reaction times used here, the results obtained with Lakshmana, a previous researcher, are remarkably different; a satisfactory outcome was obtained with the 120-minute reaction time. Stated differently, the yields of biodiesel rise and the amount generated decreases as the reaction time grows for higher and lower alcohol concentrations [6].

3.2 Result of Kinetic Viscosity at 40 °C

The EN ISO 3104 standard is an international standard used to analyze kinetic viscosity. The kinematic viscosity values of all the samples taken throughout the experiment are displayed in Table 4. The collected statistics were compared to the EN ISO 3104 international standard. This comparison demonstrates that the kinematic viscosity of every sample was found to be successful. The worldwide EN ISO 3104 standards for kinematic viscosity specified a standard range of 3.5 mm2/s to 5 mm2/s. The data gathered for kinetic viscosity is displayed in Table 4 and is contrasted with the worldwide EN ISO 3104 standards. The kinetic viscosity bared graph against samples is shown in Figure 4. The kinetic viscosity bared graph versus samples is displayed in Figure 3.

Table 4 The data collected for kinetic viscosity

Sample	Kinetic Viscosity at °C (mm^3/s)	Kinetic Viscosity EN ISO 3104 (mm^3/s)
1	4.0	
2	3.6	3.50 to 5.00
3	3.5	
4	3.6	

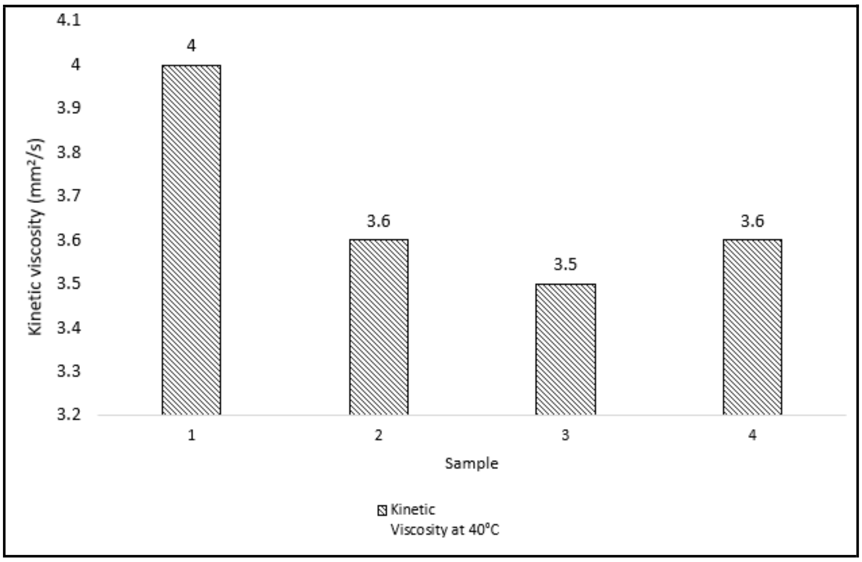

Fig. 4 Kinetic viscosity against sample graph

Kinetic viscosity was measured using thermal analysis at 40 °C. The sample with the highest kinetic viscosity value, measured at 50 °C for one hour, was the first to be recorded. According to an earlier study, the biodiesel product produced will be less viscous with the higher reaction temperature [5].

Therefore, given that biodiesel yields the same statistics, it can be concluded that cooking oil waste is a typical phase. Sample 3 exhibits the lowest value of kinetic viscosity, with a reaction time of 1 hour at 50°C. Overall, the kinetic viscosity of the sample with a 1-hour reaction time is lower than that of the sample with a 2-hour reaction time, which is operating at the same level. The graph can also show that sample 1 had the best kinetic viscosity value for biodiesel. According to the findings of other researchers, high-viscosity biodiesel fuels may cause larger droplets to form during injection, which may reduce fuel atomization and increase spray angle and tip penetration, ultimately leading to poor combustion [7]. One clear factor influencing the kinetic viscosity values was the temperature at 40 °C. In general, viscosity drops as temperature rises. While temperature tends to decrease the viscosity of biodiesel, different feedstocks may have varying relationships with temperature and viscosity [8].

3.3 Result of Yield Percentage

Each percentage of yield that varies with reaction time and temperature will be assessed once the biodiesel yield has been determined. The percentage of each sample with a different condition is displayed in Table 5. The graph of the biodiesel yield percentage for each sample is displayed in Figure 5.

Table 5 The percentage of each sample

Sample	Yield Percentage (%)
1	74.34
2	69.25
3	74.54
4	72.20

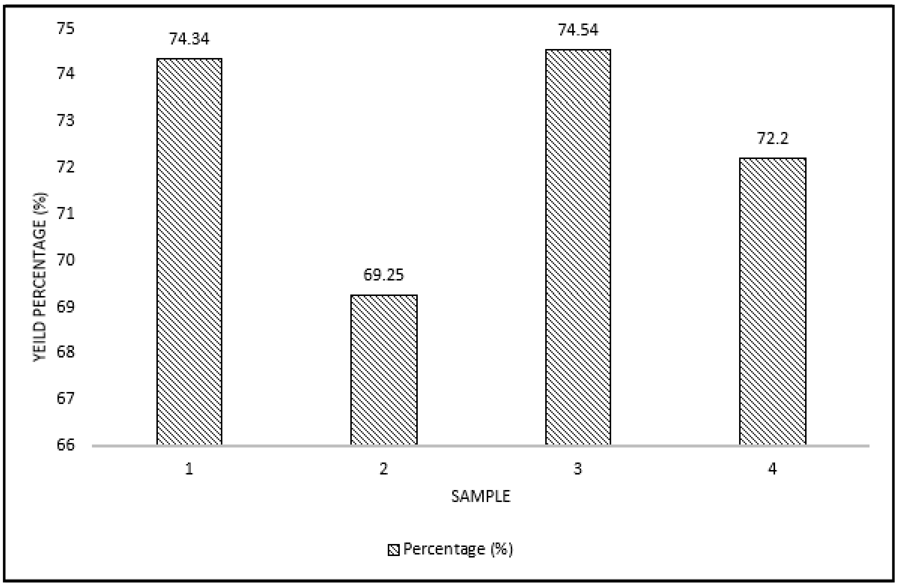

Fig. 5 Yield percentage against graph

All things considered, the observation on the bared graph displays four samples under four distinct conditions. Samples 1 through 4 produced 74.34%, 69.25%, and 72.20% of biodiesel respectively. Samples 3 and 4 produced nearly identical amounts of biodiesel, 74.54% and 72.20%, respectively, to sample 1. Accordingly, sample 3 yields the highest percentage at 50 °C and 2 hours. The bared graph indicates that the sample heated to 50 °C had a greater yield percentage while the sample heated to a higher temperature had a lower yield percentage. Additionally, it displays the proportion of yield that rises at lower temperatures and longer response times. According to a prior study, a longer mixing time yields a higher yield than a shorter one. Therefore, utilizing fried oil and methanol, 120 minutes of reaction time yielded good results compared to other reaction times used [6]. Secondly, producing biodiesel at a higher temperature can result in lower biodiesel production. According to Leung's previous research, a study employing 60 °C and a two-hour reaction period found that high temperature values can promote saponification, which can reduce the biodiesel output [9]. Other than that, the yield will be optimized when using the temperature reaction is under the boiling point of alcohol which is 50°C and below to avoid vaporization [9].

3.4 Result of Fourier Transform Infrared Spectroscopy (F-TIR)

Materials' chemical composition can be identified and characterized with the help of analytical techniques like Fourier Transform Infrared Spectroscopy. Its working theory is that various substances each have their own "fingerprint" that is created by the way molecules absorb different frequencies of infrared light. Chemical composition, forensic analysis, material analysis, and quality control can all be ascertained with F-TIR. Apart from that, an organic compound's chemical bonds and content are displayed using F-TIR. The spectra of each present molecule can also be presented via F-TIR. The transmittance spectrums of the four samples are mixed with the raw maize oil spectra in Figure 6. Additionally, it demonstrates that the contents of raw corn oil and the four other samples are nearly same. However, following the transesterification process, biodiesel has greater excess content due to the creation of bonds, methylene, and other compounds. Through Table 6, the compound and bonds are shown by the numbered labels.

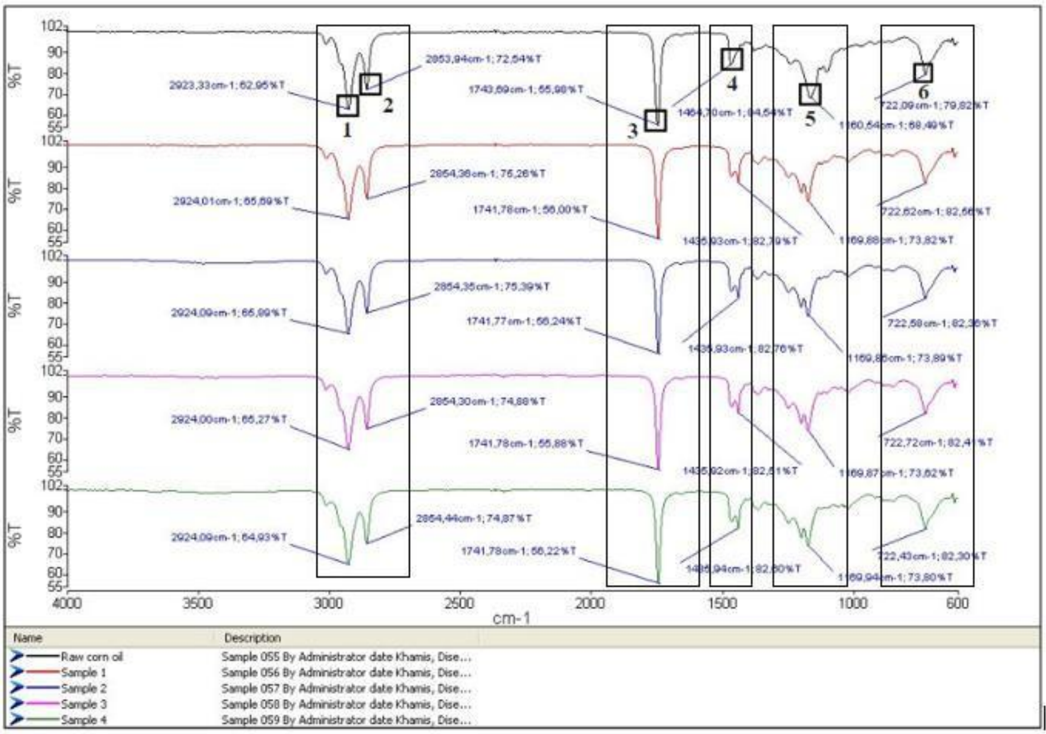

Fig. 6 Spectrum of all 4 sample with raw corn oil

Table 6 The classification of element follows the frequency of range biodiesel

Label	Frequency Range (cm^{-1})	Group	Compound Class
1	2880 - 2900	CH	Benzene, Alkene group
2	2652 - 2972	C-CH3	Propene, Alkene group
3	1700 - 1900	C=O $_{\text{stretching}}$	Ethyl ethanoate, methyl group
4	1405 - 1465	CH2	Alkane, methyl group
5	1175 - 1275	C-O-C	Cyclic olefin, Ester group
6	885 - 895	C=CH2	Propidine

Figure 6 presents that two methyl groups C=O and CH2 have been isolated through the use of methanol and triglycerides, which stretch between 1700 and 1405 cm^{-1}. Because of the C=O were stretching vibration of the carbonyl groups present in esters and triglycerides, this strong peak was situated at C=O and CH2. Then, a peak between 2880 and 2900 cm^{-1} demonstrates the symmetric and antisymmetric stretching vibrations of the groups CH, CH2, and CH3, respectively. On the other hand, the ester group bond exhibits the compound class of ester when stretched between 1175 and 1275 cm^{-1}. It is certain that the ester bond is present in the biodiesel's composition. Lastly, there is an alcohol detected at 2500 - 3200 cm^{-1} with OH group and the compound class is alcohol. Last but not least, the spectrum result from F-TIR analysis was be verified along [10]. It is found that it was discovered that every ingredient in the experiment existed, although the graph's trend varied [10]. This could be the result of various factors, such as the use of distinct techniques to identify the elements present in methyl ester, NaOH, and methanol.

4 Conclusion

Methanol was used as the excess reactant in the transesterification process of corn oil, while NaOH was used as a catalyst. Nonetheless, the goal of producing a percentage of biodiesel production between 72% and 75% was accomplished. Since the biodiesel production results closely adheres to the international standard, it can be approved within the given reaction time and temperature range. Overall, sample 3 had the highest biodiesel yield of the samples mentioned. For the density analysis, it was found that sample 3 is the best choice for the density value because the value is near 860 kg/cm^3. It shows that the lowest value of density is the best properties for ideal biodiesel quality. Then, the best value for kinetic viscosity is the highest one, which was sample 1 because it has the highest value of kinetic viscosity value. Therefore, a 2-hour reaction time at 50°C was the optimal parameter that could be optimized for the production of biodiesel. Hence, this study can improve renewable energy with less hydrocarbon emissions for a healthy future.

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