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OPTIMIZATION AND CHARACTERIZATION OF USED COOKING OIL FOR BIODIESEL PRODUCTION USING RESPONSE SURFACE METHODOLOGY

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ABSTRACT

This study focuses on optimizing and characterizing alkali-catalyzed biodiesel production from used cooking oil. Transesterification using potassium hydroxide (KOH) and methanol, followed by solvent-solvent extraction, yielded biodiesel. Physicochemical analysis of the used cooking oil revealed an acid value of 29 mgNaOH/g, free fatty acid (FFA) value of 14.5, and density of 0.91 g/cm^3. The high FFA content suggests the use of a heterogeneous catalyst. Optimization parameters included alcohol-to-oil ratio, catalyst concentration, reaction temperature, and time, employing Response Surface Methodology (RSM) based on Central Composite Design (CCD). Optimal conditions for biodiesel production were determined at a reaction temperature of 60°C, a reaction time of 60 minutes, 0.3g KOH catalyst concentration, and a 3:20 methanol-to-oil ratio, predicting a 100% yield. Physiochemical properties of the produced biodiesel indicated specific gravity and pH values of 0.891 and 7.60, respectively. Biodiesel blends (B100, B80, and B20) exhibited specific gravity and pH values of 0.891, 0.842, and 0.839, and 7.60, 7.81, and 5.5, respectively. Comparative analysis with diesel suggests the biodiesel's suitability for standalone or blended use in diesel engines. Characterization involved physicochemical analysis, Fourier Transform Infrared Spectroscopy (FTIR), and Thin Layer Chromatography (TLC). Overall, the optimized process presented a viable and efficient approach to producing biodiesel from used cooking oil with favourable fuel properties.

Keywords: Transesterification, Optimization, Thin Layer Chromatography, FT-IR Spectroscopy, Response Surface Methodology, Biodiesel

INTRODUCTION

In recent years the demand for renewable fuels has greatly increased, the alarming increase in the price of oil and the concern for the environment due to climate change induced by the use of fossil fuels, has placed renewable energy sources extremely important (Rosa *et al.*, 2013). Currently the most dominant resources for world energy supply are crude oil, coal and gas (IEA, 2015). However, the limited reserve of such fossil fuels prompts the consideration of alternative fuels from renewables. Most renewables do have environmental advantages over the conventional fuels, such as net greenhouse gas and pollution reduction (Ellabban *et al.*, 2014). The consumption of fossil fuels also releases greenhouse gases (GHGs) including oxides of sulphur (SO_x), nitrogen (NO_x), and carbon (CO_x) into the environment. Global warming and environmental hazards have become primary concerns by exploiting these energy resources (Priyanka *et al.*, 2021). These environmental advantages are additional points to strengthen the concept of replacing fossil fuels with renewable energy sources. In line with this, the International Energy Agency (IEA) renewable energy term market report 2016 indicated that the renewable energy share in the total world energy consumption is expected to have atleast 39% increment by 2021 (IEA, 2016). According to the Organization of the Petroleum Exporting Countries (OPEC). By 2040 world fuel oil demand will reach up to 109.4 million barrel per day from which, diesel fuel demand is expected at 5.7 million barrel per day (OPEC, 2016). However, this higher oil fuel demand is facing two major challenges, scarcity of the resource and negative environmental impact due to its use. Accordingly, many researchers are becoming interested in investigating alternative energy resources. Among such alternatives, biodiesel is getting more emphasis for some reasons. It can be produced from a wide variety of resources including used cooking oil, oily sludge from factories and waste animal fat (Olkiewicks *et al*., 2016). In addition, there are a number of technological choices to produce biodiesel based on the quality of the feedstock, giving possible alternatives to minimize overall production expenses (Ahvad and Marchetti, 2015). When it is compared to conventional petrol diesel fuel, biodiesel has no sulphur. It also produces less carbon monoxide, particulate matters, smoke and hydrocarbons and has more free oxygen than the conventional petrol diesel (Hasan *et al.*, 2017). Having such more free oxygen results in complete combustion and reduced emission (Fazal *et al.*, 2011). Biodegradability, higher flashpoint and inherent lubricity are other worth mentioning advantages of biodiesel over the conventional petro diesel (Knothe, 2008).

The use of edible vegetable oils and animal fats for biodiesel production has been of great concern because they compete with food materials the food versus fuel dispute (Pimental *et al.*, 2009; Srinivasan, 2009). There are concerns that biodiesel feedstock may compete with food supply in the longterm (Lam *et al*., 2009; Metzger, 2009). The new process technologies developed during the last years made it possible to produce biodiesel from recycled frying oils comparable in quality to that of virgin vegetable oil biodiesel with an added attractive advantage of being lower in price (Canakei, 2007; Chhetri *et al*., 2008; Refaat *et al*., 2008). Base-catalyzed transesterification involves stripping the glycerin from the fatty acids with a catalyst such as sodium or potassium hydroxide and replacing it with alcohol, usually methanol. The resulting raw product is then centrifuged and washed with water to cleanse it of impurities. This yields methyl or ethyl ester (biodiesel), as well as a smaller amount of glycerol, a

valuable by-product used in making soaps, cosmetics and numerous other products (Singh *et al*., 2006). This study evaluated the optimization of used cooking oil with heterogenous based catalyzed tranesterification using KOH as base and methanol as the alcohol by response surface methodology. And characterized using FTIR spectroscopy, physiochemical analysis and thin layer chromatography.

MATERIALS AND METHODS

Physiochemical Analysis of the Oil Sample

This analysis was carried out in order to assess the quality of the oil. This is to ascertain the acceptability, stability and other characteristics of the oil in terms of biodiesel production. (Usman *et al.,* 2013).

Determination of Acid Value

Acid value is the number of milligram of base required to neutralize the fatty acid in 1g of the fat/oil. 2.8g of the oil was weighed in a conical flask and dissolved in 50cm^3 of solvent mixture (ethanol and diethyl ether) to which 5 drops of phenolphthalein indicator was added. The solution was then titrated with 0.1M NaOH. The end point was pink in colour.

Acid value = $\frac{40\ x\ N\ x\ T}{M}$ (1)

Where 40 = molar mass of NaOH, N = normality of NaOH, T = titre value and M = mass of oil used.

Determination of pH

The pH was determined using a Jenway pH meter, model 3320. The pH value was then recorded.

Determination of Specific Gravity

A clean and dried density bottle was weighed and labelled (M_0). The bottle was then filled with distilled water and weight of the bottle taken and recorded as (M_1). The same bottle was emptied and dried and it was then filled with oil, weighed and the value obtained was recorded as (M_2).

The specific gravity was then calculated using the formula:

Specific Gravity = $\frac{M_2 - M_0}{M_1 - M_0}$ (2)

Where M_0= Weight of empty bottle, M_1= Weight of the bottle filled with distilled water and M_2 = Weight of the bottle filled with oil.

Determination of Free Fatty Acid

The free fatty acid of the oil was determined using the formula;

%FFA = $\frac{Acid\ Value}{2}$ (3)

Determination of Moisture Content

The moisture content of the oil was determined by weighing a clean and dried beaker (50ml) and then recording the value obtained as W_1. A specific amount of the oil was then put into the beaker and weighed again recording the new value as W2. It was then put into an oven and kept for 24 hours at 60°C. It was weighed again and the value obtained was recorded as W3.

The moisture content was calculated as:

Moisture content = $W_2 - W_3$. (4)

Where W_3= final weight obtained after 24hours, W_2=initial weight obtained

Optimization using Response Surface Methodology

Response Surface Methodology (RSM) based on a Central Composite Rotatory Design (CCRD) was used to optimize three process variables using `Design Expert Version 7.1.6'. The parameters varied are described in Table 2.1

Table 1: Parameter Varied using RSM

Parameters	Minimum Value	Maximum Value
Oil/Alcohol ratio	1:2	1:7
Time (min.)	19.09	220.91
Temperature (°C)	13.07	71.93

Biodiesel Production Process

The oil was first filtered to remove unwanted food chunks it was then fed to a reactor (a flat bottom flask with a stirrer connected to a reflux condenser on a hot plate) and specified amount of potassium methoxide solution was poured onto it after heating the oil for about 5 minutes to reduce the viscosity of the oil. The reaction took place for the desired time at the specified temperature. The reaction mixture was then allowed to cool and even up, resulting in the separation of two (2) phases. Fatty acid methyl ester (biodiesel) at the top and glycerol at the bottom. The mixtures were separated in a separatory funnel the biodiesel was collected in a sample bottle and the glycerol was also collected in a separate sample bottle. The biodiesel layer was washed with hot distilled water to about 30% of the weight of the oil, it was then subjected to centrifugation to further remove the glycerol and water remaining in the biodiesel.

Biodiesel Yield

The percent yield of Biodiesel produced in each case was calculated using the formula;

YOB = $\frac{\text{mass of biodiesel produced (g) x100\%}}{\text{mass of oil taken for reaction}}$ (5)

Where YOB= Yield of Biodiesel

Biodiesel Blend

The biodiesel was blended with diesel to obtain B50 which is 50ml of diesel and 50ml of biodiesel and also B20 which is 80ml of diesel and 20ml of biodiesel. The specific gravity and the pH of the various blends were obtained.

Fourier Transform Infrared Spectroscopy

Fourier transform infrared spectroscopy (FTIR) characterization techniques was carried out on the samples (the feedstock, biodiesel before washing and Biodiesel after washing) Fourier transform infrared spectroscopy was adopted for functional group analysis and possible molecular rearrangement expected for chemical changes by careful inspection of the spectra (Zhang *et al* 2014).

Thin Layer Chromatography (TLC)

Thin layer chromatography was carried out on the samples (feedstock and biodiesel). The TLC was performed by spotting the sample using capillary tube on precoated aluminium silica gel. Each plate was developed on suitable solvent system and then allowed to dry. The plates were visualized under wavelength (254 and 365nm).

Retention factor (RF) value was determined from the relation

RF = $\frac{Distance\ of\ centre\ of\ spot\ from\ starting\ point}{Distance\ of\ solvent\ front\ from\ starting\ point}$ (6)

Where RF = Retention factor

RESULTS AND DISCUSSION

Physiochemical Analysis

Acid value is the measure of mg of KOH required to neutralize FFA in 1g of the oil. The acid value of the oil feed as shown in Table 2 was 29 mgNaOH/g hence, this value indicates that there was free fatty acid present in the oil feed that need to be neutralized to obtain good biodiesel quality (Reda, 2014). The data obtained from acid value allowed us to compute the value of free fatty acids present in a given oil, the value of free fatty acids indicate the catalysis method to employ, if the value of FFA is lower than 3 homogeneous catalysis is preferred and if its greater than 3, catalysis use is heterogeneous because at value greater than 3 homogeneous catalysis lead to the formation of soap, which make separation between the biodiesel and the soap to be difficult (Dawudo *et al.,* 2014).

The value of FFA obtained from the oil that 14.5 mg NaOH/g allow us to make use prompted the adoption of heterogeneous catalysis method. Density has been described as one of the most basic and most important properties of fuel because of its correlation with cetane number, heating values and fuel storage and transportations (Alawu *et al.,* 2007). The density, 0.91 g/cm^3 obtained for the oil feed as shown in Table 2 was close to standard which is within the range of 0.87-0.9 g/cm^3 (Usman *et al.,* 2013). The moisture content which is the quantity of water contained in the oil was found to be 0.01 and the colour of the oil is Golden Brown this was determined visually. All the values obtained are within the ASTM standards.

Table 2: Physiochemical analysis of the oil sample

S/N	Parameters	Values Obtained
1	Acid Value	29mgNaOH/g
2	Free Fatty Acid	14.5mgNaOH/g
3	Specific Gravity	0.91
4	pH	6.8
5	Colour	Golden Brown
6	Moisture content	0.01%

Response Surface Methodology

Response Surface methodology was used in order to determine the ideal temperature, time and the ideal oil and alcohol molar ratio to be used for biodiesel production having said that these three factors are the major variables that affect biodiesel production. The factors oil/alcohol ratio, time (minutes) and temperature (degree celsius) were varied in the range 1:3-1.6, 60-180 minutes and 25-60°C respectively. The optimization gave twenty (20) responses which are shown in the table3.

Table 3: Parameters Varied by the RSM and results obtained for biodiesel yield

Run	Oil/Alcohol Molar Ratio	Time (minutes)	Temperature (°C)	Biodiesel yield (%)
1	1: 6.0	60	25	90
2	1: 6.0	180	60	93
3	1: 4.5	220.91	42.50	25
4	1: 4.5	120	42.50	90
5	1: 3.0	60	25	100
6	1: 3:0	180	60	25
7	1: 4.5	120	13.07	25
8	1: 4.5	120	42.50	90
9	1: 6.0	60	60.00	50
10	1: 4.5	120	42.50	90
11	1: 4.5	19.09	42.50	70
12	1: 7.0	120	42.50	40
13	1: 4.5	120	42.50	90
14	1: 4.5	120	71.93	25
15	1: 4.5	120	42.50	90
16	1: 3.0	180	25	20
17	1: 4.5	120	42.50	90
18	1: 3.0	60	60	100
19	1: 1.9	120	42.50	20
20	1: 6.0	180	25	25

Table 3 shows the yield of biodiesel obtained which was calculated using the formula in equation (5) after the experiment has been conducted, the 5th and 18th responses gave the highest yield of biodiesel of 100% was obtained at 25 and 60°C for 60 minutes each, while the 16th and 19th responses gave the lowest yield of 20% at 25 and 42.5°C for 120 and 180 minutes respectively. The results obtained suggested that time has an effect on the reaction.

Table 4 contains the actual values obtained after conducting the experiments, the values predicted by RSM and the residual values which is the difference between the actual values and the predicted values.

Table 4: Actual, Predicted and Residual values

S/N	Actual Values	Predicted Values	Residual Values
1	100.00	96.36	03.64
2	100.00	67.19	32.81
3	20.00	-02.15	22.15
4	25.00	25.19	-0.19
5	90.00	64.19	25.81
6	50.00	46.52	03.48
7	25.00	32.18	-07.18
8	93.00	71.02	21.98
9	25.00	39.06	-14.06
10	25.00	47.18	-22.18
11	70.00	96.74	-26.74
12	25.00	34.50	-09.05
13	20.00	42.38	-22.38
14	40.00	53.86	-13.86
15	90.00	88.96	01.04
16	90.00	88.96	01.04
17	90.00	88.96	01.04
18	90.00	88.96	01.04
19	90.00	88.96	01.04
20	90.00	88.96	01.00

Fourier Transform Infrared Spectroscopy

Since each different material is a unique combination of atoms, no two compounds produce the exact same infrared spectrum. In FTIR spectroscopy, analysts plotted the intensities at each individual frequency in order to quick sample identification.

Oil Sample Spectrum

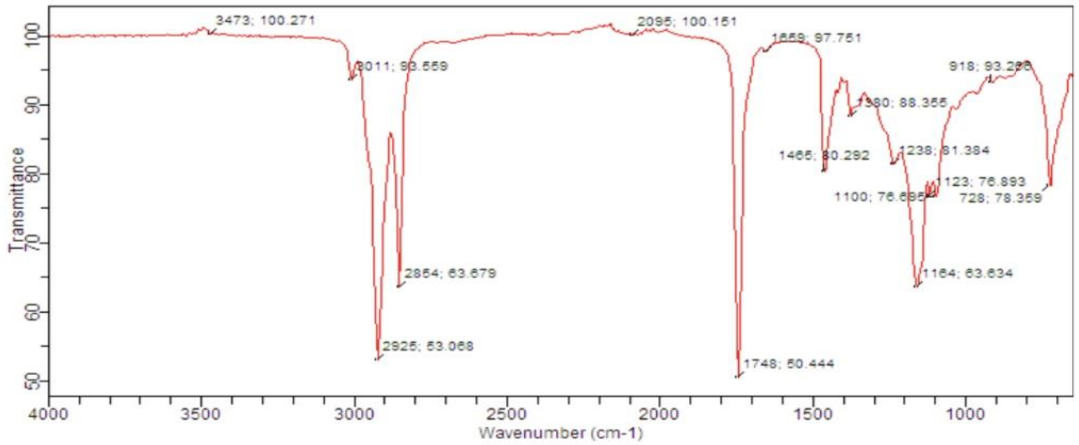

Figure 1: FTIR Spectrum of Oil Sample

Figure 1 represents the spectrum of the oil sample, the feedstock is a triglyceride and the main functional group in triglyceride is ester, in the finger print region we have absorptions at around 728cm^{-1} which indicates the presence C=C bending, 1100 cm^{-1}, 1164 cm^{-1}, 1238 cm^{-1}, 1123 cm^{-1} indicate the presence C-O stretching, 1380 cm^{-1}and 1469 cm^{-1} indicate the presence of C-H bending, and 1659 cm^{-1} which indicates the presence of C=C stretching. From the spectrum, a strong absorption at 1748 cm^{-1} indicates the presence of esters and confirms the presence of triglyceride. An absorption being medium to strong absorption around 2850-2975 cm^{-1} indicates the presence of alkane C-H stretching.

Biodiesel Spectrum

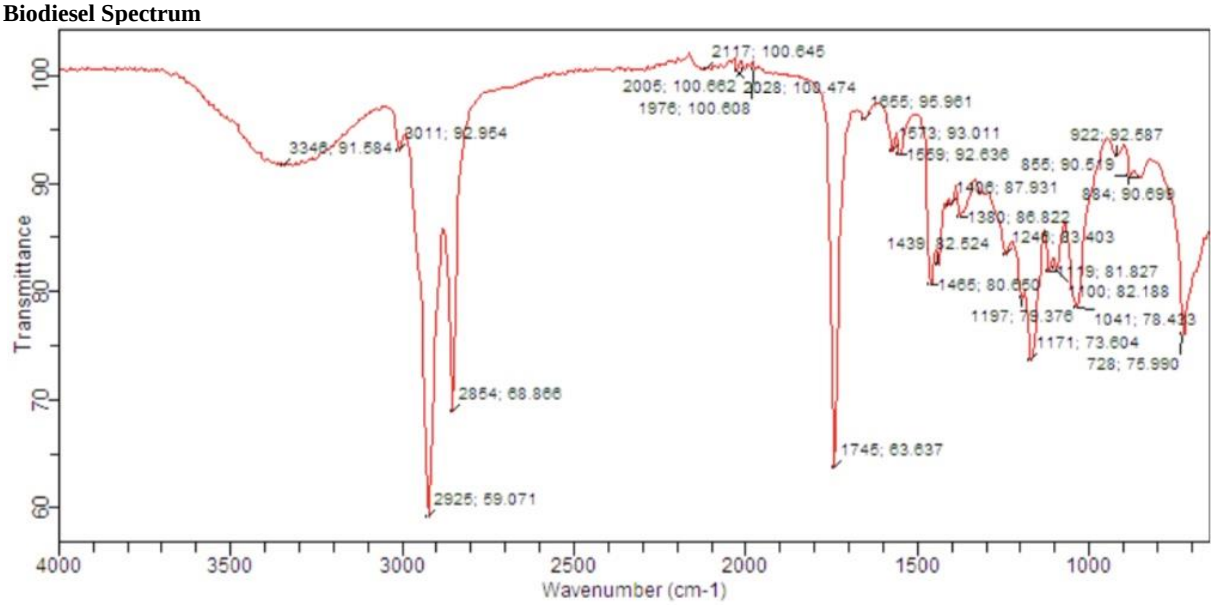

Figure 2: FTIR Spectrum of Biodiesel before washing

Figure 2 is a biodiesel FTIR spectrum, the main functional group in biodiesel is ester. The finger print region showed absorptions around 728 cm^{-1}, 884 cm^{-1}, 855 $cm^{-1}$and 922cm^{-1} which indicates the presence of C=C bending of alkene, 1041 cm^{-1},1100 cm^{-1} indicates the presence of 1197 cm^{-1} ,1171 cm^{-1} and 1246 cm^{-1} Indicates the presence of C-O stretching of ester. Vibrations around 1380 cm^{-1},1406 cm^{-1} ,1439 cm^{-1}, 1465 cm^{-1} indicates the presence of C-H bending of alkane. The spectrum shows absorptions around 1559 cm^{-1}, 1573 cm^{-1}, 1655 cm^{-1} and in the functional group region strong absorption at 1735-1750 cm^{-1} indicates the presence of ester. In the spectrum a strong absorption at 1748 cm^{-1} observed indicates the presence of esters and confirms the presence of the biodiesel. In the spectrum a medium to strong absorption around 2854-2925 cm^{-1} indicates the presence of C-H alkane. Furthermore, strong and broad absorption band around 3400-3700 cm^{-1} indicates the presence of alcohol and in the spectrum above strong and broad absorption band around 3346 cm^{-1} which is very close to the absorption band of alcohol indicated that there are other products other than the biodiesel, the sample was washed and subjected to a centrifuge machine to further remove impurities present in the sample, FTIR test was done again which gave rise to the sample in figure 3.

Washed Biodiesel Spectrum

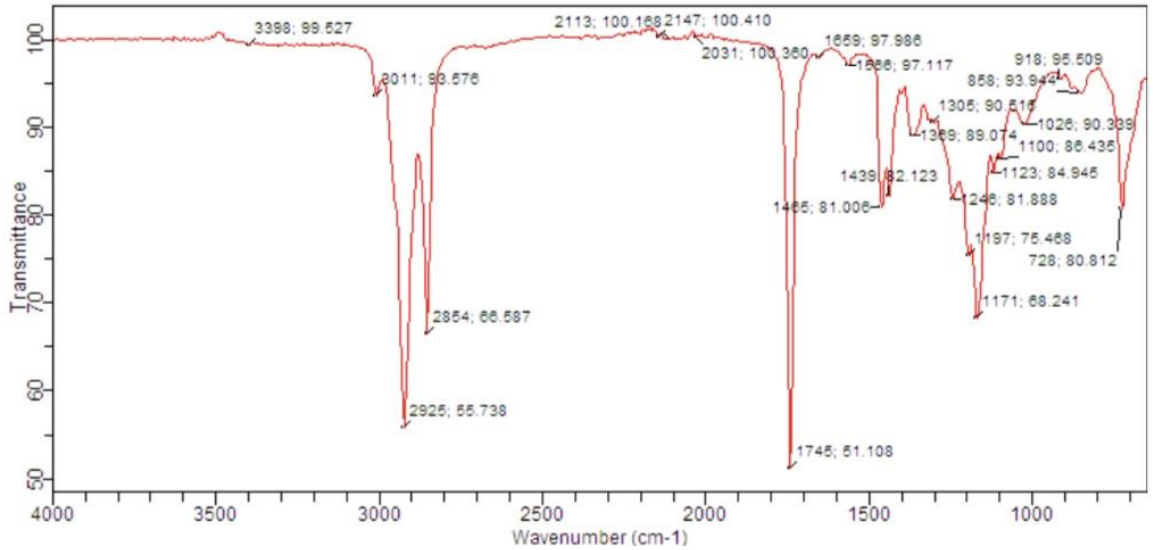

Figure 3: FTIR Spectra of Washed Biodiesel

In fig 3 is the washed biodiesel sample spectrum and a pure biodiesel is expected, the main functional group in biodiesel is ester. The finger print region shows absorptions around 858 cm^{-1} and 918 cm^{-1} which indicates the presence of C=C bending of alkene, 1026 cm^{-1}, 1100 cm^{-1}, 1123 cm^{-1}, 1171c m^{-1}, 1197 cm^{-1} and 1246cm^{-1} indicates the presence of C-O stretching of ester, 1305 cm^{-1}, 1369cm^{-1}, 1439 cm^{-1}, 1465cm^{-1} indicate the presence of C-H bending of an alkane. The functional group region shows absorptions at around 1566 cm^{-1}, 1659 cm^{-1} indicates the presence of C=C bending of an alkene. Strong absorption at 1735-1750 cm^{-1} indicates the presence of ester. In the spectrum, a strong absorption at 1745 cm^{-1} which indicates the presence of ester also confirms the presence of the biodiesel. Also, absorption at around 2011 cm^{-}

[1] indicates the presence of alkene C=C=C, a medium to strong absorption around 2850-2975 cm^{-1} indicates the presence of alkane, In the spectrum we have a medium to strong absorption around 2854-2925 cm^{-1} which confirms the presence of alkane, a weak and narrow absorption band around 3398 cm^{-1}indicates that the impurities that were present in fig 3.3 have been thoroughly washed.

Physiochemical Properties of Biodiesel Blends

The physiochemical properties (Colour, Specific gravity and pH) of biodiesel (B100) and diesel were compared as shown in Table 5. The specific gravity of B80 and B20 are found in the range of diesel standard range 0.81-0.86 while that of B100 was in the biodiesel standard range of 0.87-0.9, this indicates that used cooking oil biodiesel could be used in blends with diesel to power compression (diesel) engine. The pH of B100, B80 and B20 are 7.60, 7.81 and 5.5 respectively as shown in Table 6, which indicates that the blends of the biodiesel could prevent corrosion problems since the pH is near neutral.

Table 5: Physiochemical Properties of Biodiesel and Diesel

S/N	Properties	Biodiesel	Diesel
1	Colour	Golden Yellow	Golden Brown
2	Specific gravity	0.891	0.857
3	pH	7.60	8.56

Table 6: Specific Gravity and pH of Biodiesel blends

S/N	Sample	Specific Gravity	pH
1	B100	0.891	7.60
2	B80	0.842	7.81
3	B20	0.839	5.5

Thin Layer Chromatography (TLC):

After the sample has been applied on the plate, a solvent or solvent mixture (known as the mobile phase) is drawn up the plate via capillary action. Because different analytes ascend the TLC plate at different rates, separation is achieved. (Archana and Anubah, 2011). After completing the TLC, Ultraviolet light was used to view the spots on the plate and the spots were very much visible when viewed with shorter wavelength than longer wavelength, the difference between the retention factor of the spots indicates that's the two samples are different. Table 7 shows the retention factors of the samples.

Table 7: Retention factors of the sample

Sample	Retention Factor
Vegetable Oil	0.75
Biodiesel	0.87

CONCLUSION

In this project biodiesel is produced from used cooking oil. Optimization was used for the production of biodiesel, providing optimal conditions for the research conducted. The conducted experiment suggests that carrying out the transesterification reaction for 60 minutes within a temperature range of 25°C- 60°C using 3g of alcohol and 20g of oil, will yield 20ml of biodiesel, which is equivalent to the grams of oil used. The biodiesel was characterized using physiochemical, spectroscopic, chromatographic properties.

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