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Evaluation Of Conversion Efficiency And Product Characterization For A Mini Biodiesel Production Plant

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Abstract*— In this paper, evaluation of conversion efficiency and product characterization for a mini biodiesel production plant is presented. Specific details presented in the work include; the working principle of the plant, the procedure for the preparation of the Waste Vegetable Oil (WVO) feedstock, titration procedure, procedure for preparation of the Methoxide solution, description of the transesterification reaction, the procedure for purification of the biodiesel and the procedure for drying of washed biodiesel. The procedure for product characterization is also presented which includes; approach for the determination of the biodiesel acid value, biodiesel density, biodiesel specific gravity, biodiesel viscosity, biodiesel flash point and biodiesel cloud point. Six equal volumes of WVO methanol and sodium hydroxide were processed in the mini biodiesel production plant for periods of 60, 65, 70, 75, 80 and 85 minutes respectively, representing six batches of experimental runs. The effect of transesterification time on the yield show that highest biodiesel yield (46.47 kg) was obtained from the reaction that lasted 80 minutes. Based on these findings, a reaction time of 80 minutes was selected as the optimum reaction time. The highest yield was obtained at 60 °C, therefore representing the optimum temperature for the production of biodiesel from the plant. The optimum methanol/oil ratio was taken as 5:1 due to the highest yield observed. Similarly, 54.6 kg of WVO undergoes transesterification reaction to yield 46.47 kg of biodiesel. Hence the conversion efficiency was calculated as 85.10 %. Also, a flash point of 205 °C was measured for the waste vegetable oil but upon conversion it fell to an acceptable value of 134 °C. Finally, a cetane number of 55.38 was determined for the biodiesel which falls within the acceptable range. This property signifies a good measure of fuel ignition delay and combustion quality.

Keywords*— Product Characterization, Titration, Biodiesel Production Plant, Transesterification Reaction, Conversion Efficiency, Biodiesel

1.0 INTRODUCTION

Over the years, the global demand for energy continues to rise due to increase in population (Van Ruijven, De Cian and Sue Wing, 2019; Ahmad and Zhang, 2020; Avtar, Tripathi, Aggarwal and Kumar, 2019). On the other hand, the negative impact of fossil fuel on the environment and human health has prompted research into renewable energy (Ebhota and Jen, 2020; Holechek, Geli, Sawalhah and Valdez, 2022). Biodiesel has been identified as one of the most important and relevant alternative fuel due to its usability in diesel engines (Paulauskiene, Bucas and Laukinaite, 2019; Jagtap, Pawar andLahane, 2020; Erol, D., Yeşilyurt, Yaman and Doğan, 2023). It is a clean fuel and environmentally friendly fuel produced from the transesterification reaction involving triglycerides and alcohol (Jogarao and Swarna Kumari, 2019; Ramos, Dias, Puna, Gomes and Bordado, 2019). Biodiesel can

thus be used in the transportation industry and for private generation of electricity.

To achieve local production of biodiesel, it is essential that biodiesel production plants are constructed and properly evaluated to ascertain the efficiency of the plant and also to properly characterise the produced diesel product from the plant. Accordingly, in this paper, evaluation of conversion efficiency (Salehi, Karbassi, Ghobadian, Ghasemi and Doustgani, 2019) and product characterization for a mini biodiesel production plant is presented (De Paola, Mazza, Paletta, Lopresto and Calabrò, 2021; Hoseini, Najafi, Ghobadian, Mamat, Ebadi and Yusaf, 2019). Specific details presented in the work include; the working principle of the plant, the procedure for the preparation of the Waste Vegetable Oil (WVO) feedstock, titration procedure, procedure for preparation of the Methoxide solution, description of the transesterification reaction, the procedure for purification of the biodiesel and the procedure for drying of washed biodiesel. Particularly, in this study, the optimum reaction time, the optimum reaction temperature, the optimum methanol/oil ratio and optimum catalyst concentration are determined.

In addition, the physiochemical properties of the produced biodiesel are also determined (Singh, Sharma, Soni, Inda, Sharma, Sharma and Jhalani, 202; Yaşar, 2020). Accordingly, the procedure for product characterization is also presented which includes; approach for the determination of the biodiesel acid value, biodiesel density, biodiesel specific gravity, biodiesel viscosity, biodiesel flash point and biodiesel cloud point (Binhweel, Bahadi, Pyar, Alsaedi, Hossain and Ahmad, 2021; Onyezeka, Nnaji, Omotugba and Garba, 2020).

In all, the development of affordable mini biodiesel plant will bring a relief to the fuel industry by employing the use of waste vegetable oil for the production of biodiesel. It will also provide a cleaner fuel that offers little or no hazard to the environment and human health at large. This low-cost solution will provide a cheaper way of obtaining biodiesel which can be used in the transportation industry and for powering diesel engine generators

2. METHODOLOGY

2.1 Working Principle of the Mini Biodiesel Production Plant

2.1.1 Preparation of the Waste vegetable oil (WVO) feedstock

Waste vegetable oil (WVO) collected from local restaurants is fed into the WVO tank through a strainer in order to filter food debris from the oil. The oil is titrated in order to determine the amount of catalyst needed to make biodiesel. This is done by making use of an electronic pH meter to check the pH of the WVO. The oil is then heated to 80 °C in order to get rid of any moisture present in the oil. After heating, the oil is transferred to the reactor by means of the pump. The reactor is filled to 70% capacity then the oil is allowed to cool to 55 °C.

2.1.2 Titration Procedure

The essence of conducting the titration is to ascertain the amount of catalyst required to convert waste vegetable oil to biodiesel. The following equipment were used for carrying out the titration: scale, beakers, measuring cylinders, syringes, pipettes, dropper bottle, and methanol storage bottle. In addition, the following materials were used: 1 liter of Isopropyl alcohol, pH indicator solution (Phenolphtalein), 4 liters of distilled water.

The first step involves creating a 0.1% lye catalyst solution, this is done by measuring 1 gram of potassium hydroxide, dissolving the 1 gram of catalyst into 1 liter of distilled water. The next step is to measure 10 ml of Isopropyl alcohol using a 10ml syringe and pouring it into a 50 ml beaker. A couple of drops of the 1% phenolphthalein was added to the alcohol solution. 1 ml of WVO sample was measured using a 1 ml syringe and dispensed into the beaker containing the Isopropyl alcohol. The beaker was be swirled around until it dissolved in the Isopropyl alcohol. A stirring rod was used to stir the solution while the catalyst solution was dispensed into it. A cloudy white color appeared initially after which signs of pink are expected to emerge. Once pink color was observed, the catalyst solution was added drop by drop until the pink color remains for 30 seconds. At this point the results were measured and the titration amounts were recorded.

The amount of catalyst solution in grams or ml taken to complete the titration was recorded, indicating the amount of lye catalyst needed in addition to the base catalyst for processing the WVO.

2.1.3 Preparation of the Methoxide Solution

Methoxide solution was made in the methoxide tank by pouring methanol into the tank and mixing it with the potassium hydroxide (KOH) catalyst until complete dissolution of the KOH pellets. The obtain the optimum methanol /oil ratio, the following ratios 3:1, 5:1, 8:1 and 10:1 were tested. The quantity of the KOH was varied in the following order; 0.5, 1.0, 1.5 and 2.0 w/w oil to ascertain the optimum catalyst concentration. The pump was turned on and the methanol and KOH mixture recirculated within the methoxide tank until the KOH completely dissolved. The resulting mixture was then transferred into the reactor, where the transesterification reaction took place.

2.1.4 The Transesterification Reaction

This is the reaction that gives birth to biodiesel and glycerol. After the methoxide solution was transferred into the reactor, the pump and valves were controlled in such a way that allowed for recirculation and mixing of the methoxide and WVO present in the reactor. The reaction temperature was alternated between 40, 45, 50, 55, 60 and 65 °C to ascertain the optimum temperature. This was done by switching on the electric heater installed in the reactor which has a thermostat. The reaction was carried out for the following mixing times; 60, 65, 70, 75, 80 and 85 minutes after which the pump was switched off and all the valves closed. The resulting mixture was then allowed to settle for 8 hours to ensure complete separation of the glycerol and biodiesel produced from the reaction. The funnel shaped bottom of the reactor allows the denser glycerol to settle by

gravity at the bottom. The glycerol was drained out through the pipe connected to the bottom of the reactor. The biodiesel produced was washed to remove impurities and residual alcohol in order to enhance its suitability in engines.

2.1.5 Purification of the Biodiesel

The purification of the biodiesel was done in order to remove some impurities left in the biodiesel after the transesterification reaction. This was done by washing the biodiesel. The method for washing the biodiesel used in this work is the mist washing method which involves gently agitating deionized water with the biodiesel. A shower head was mounted at the top of the reactor tank containing the crude biodiesel. The shower hose was connected to a warm water source and the water was sprayed in tiny droplets over the biodiesel until the tank was 90% full. The water settled to the bottom of the reactor after 2 hours. The water containing excess methanol, catalyst and other impurities was drained out into a container. This process was repeated until the water became very clear indicating complete washing.

2.1.6 Drying of Washed Biodiesel

After the biodiesel was washed with water, despite draining the wash water that settles at the bottom of the reactor tank, some moisture was still left in the biodiesel. The presence of water can affect the diesel engines negatively and also cause difficulty in ignition. Hence, the biodiesel was heated to a temperature of 100 °C for about 1 hour by means of the electric heater in the reactor tank. After this was done, the biodiesel was pumped out into a storage tank, ready for use in any diesel engine.

2.2 Conversion Efficiency of the Plant

After making the WVO undergo transesterification reaction, this results in a yield of biodiesel. Therefore, the conversion efficiency of the developed biodiesel plant can be obtained as follows;

$$\frac{Biodiesel\ yield\ (kg)}{WVO\ quantity\ (kg)} \times 100\% \qquad (1)$$

2.3 Characterization of the Biodiesel produced from waste vegetable oil (WVO)

2.3.1 Method for determination of biodiesel acid value

Titration: Titration is done in order to obtain the amount of FFA present and how much extra lye is needed to complete the biodiesel reaction. The following items were used for carrying out the titration: 1/1 Ethanol/Diethyl Ether mixture and standard solution of KOH with concentration of 0.008285 M. Phenolphtalein indicator is used to show a color change when the end point is reached.

Procedure : 50mL of Ethanol/Diethyl Ether solvent was poured in a 250 mL Erlenmeyer flask using a graduate beaker and 5 drops of phenolphthalein were added. Some drops of KOH solution were added in order to neutralize the solvent until the color changed to pink. 10 g of biodiesel was measured and added to the neutralized solvent. The titration was repeated until the color change was noticed.

The acid value is given as;

$$acid\ value\left(\frac{mgKOH}{g\ sample}\right) = \left[\frac{volume\ KOH(mL) \times N\ KOH\left(\frac{mmol}{mL}\right) * 56.1\left(\frac{mg}{mmol}\right)}{sample\ weight\ (g)}\right] \qquad (2)$$

2.3.2 Determination of biodiesel density

The device for measuring the biodiesel density is a hydrometer. The hydrometer is an air-filled graduated glass tube, which has a weight that measures the biodiesel density.90 ml of biodiesel sample was added to graduated beaker and the room temperature is noted. The hydrometer was dropped inside the beaker containing the biodiesel and the hydrometer was observed for stop of its movement. The value in the hydrometer scale was read and recorded.

2.3.3 Determination of biodiesel specific gravity

After the reading of the density of biodiesel was recorded, the density of a similar volume of water was taken at the same temperature with the biodiesel sample. Thereafter the specific gravity $(S.G)$ of biodiesel was calculated using the formula:

$$S.G = \frac{Density\ of\ Biodiesel}{Density\ of\ water} \qquad (3)$$

2.3.4 Determination of biodiesel viscosity

An Ubbelohde viscometer was used to determine the kinematic viscosity of biodiesel. The viscometer is a U-shaped glassware with a reservoir on one side and a measuring bulb with capillary on the other.

The procedure started with charging the viscometer with biodiesel by putting it into the large tube. Next, a clean dry rubber tubing was connected to the timing tube and Air tube, the viscometer was placed in a bath of water in order to control the temperature. The capillary is kept vertical while the temperature of the biodiesel comes to the temperature in the water bath.

Next, the rubber tubing connected to the air tube was sealed off and gently suctioned to the timing tube until the biodiesel reached 5mm above the upper timing mark. The biodiesel is held at same level by sealing the tube.

Next the air tube is released to allow the biodiesel to fall away from the bottom of the capillary tube. The timing tube is released and the biodiesel is allowed to flow. The flow time for the bottom of the meniscus is measured as it passes from the top edge of the top mark line to the top edge of the mark line below it. The time taken for the liquid to pass through the two calibrated marks indicates the value of the viscosity.

2.3.5 Determination of biodiesel flash point

The flash point is the minimum temperature at which the vapors in a sample are inflamed by an attached ignition source. ASTM D 93 is the normal method for evaluating biodiesel's flash point.

The flash point was determined when a stirred container holding the biodiesel sample was heated and a flame was carried on the sample's surface. This was done until the vapor ignited and a flash was observed indicating that the temperature of the sample had reached its flash point.

2.3.6 Determination of biodiesel cloud point

The cloud point is the temperature at which a cloud of wax crystals first appears in the biodiesel sample upon cooling. Hence, it serves an index for the lowest temperature of the biodiesel's usefulness under certain applications. The standard procedure for measuring the cloud point is ASTM D 2500. This was done by pouring the biodiesel sample in a container and cooling it under controlled conditions. While this was done, the biodiesel was inspected for the appearance of a haze. Once the haze was observed, the temperature was noted and recorded.

3. RESULTS AND DISCUSSION

3.1 Effect of Transesterification Time on Biodiesel Yield

Six equal volumes of waste vegetable oil (WVO), methanol and sodium hydroxide were processed in the mini biodiesel production plant for periods of 60, 65, 70, 75, 80 and 85 minutes respectively, representing six batches of experimental runs. The effect of transesterification time on the yield was determined from Table 1.

Table 1: Biodiesel yield from varying transesterification reaction time

Experimental Conditions	1st run	2nd run	3rd run	4th run	5th run	6th run
KOH concentration (kg)	0.273	0.273	0.273	0.273	0.273	0.273
Reaction temperature	55	55	55	55	55	55
Reaction time (min)	60	65	70	75	80	85
WVO quantity (kg)	54.6	54.6	54.6	54.6	54.6	54.6
Methanol quantity (kg)	10.92	10.92	10.92	10.92	10.92	10.92
Biodiesel obtained (kg)	40.09	42.28	43.25	45.78	46.47	45.15
Glycerol obtained (kg)	13.26	13.47	13.69	13.54	13.38	12.85
Losses (kg)	12.443	10.043	8.853	6.473	5.943	7.793

Highest biodiesel yield was obtained from the reaction that lasted 80 minutes (Table 1). Losses were recorded in all experimental runs due to excess methanol, residual catalyst and emulsion removed during the washing phase of the production process.

The biodiesel was produced in six different batches from waste vegetable oil and the physiochemical properties of the samples taken from each of these batches were obtained using the methods earlier mentioned. Graphs of Biodiesel yield (kg) against Reaction time (min) and Glycerol yield (kg) against Reaction time (min) are shown in Figure 1 and Figure 2 respectively.

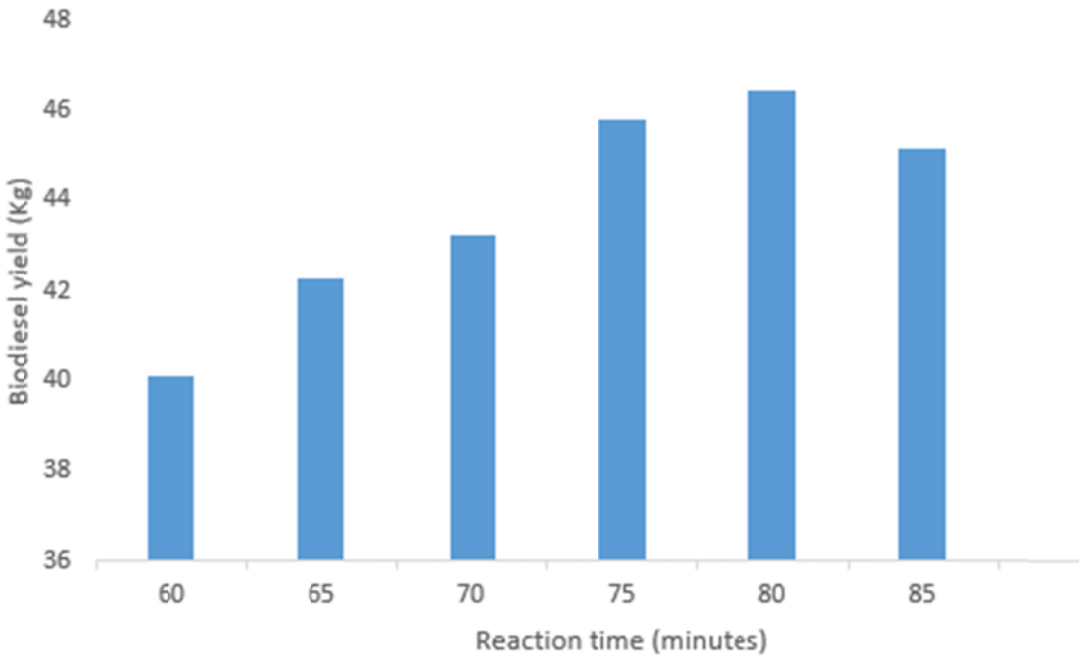

Figure 1: Biodiesel yields for different reaction times.

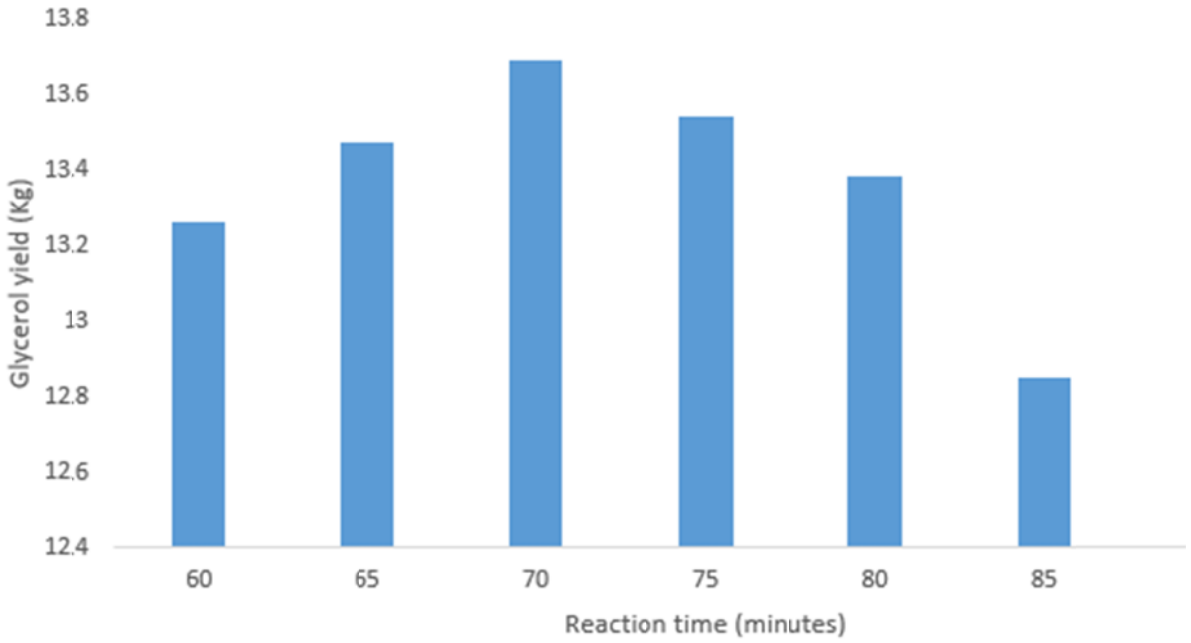

Figure 2: Glycerol produced at different reaction times

The results in Figure 1 show that biodiesel yield increases as reaction time increases until optimum biodiesel yield of 46 kg is obtained from the transesterification reaction which was allowed to last for 80 mins. If further time is used there will be a reduction in Biodiesel yield. Hence for optimum biodiesel yield, the reaction time is 80 minutes.

Also, Figure 2 shows the result of plotting glycerol yield (kg) against reaction time (kg). The chart shows that the highest glycerol yield (13.69 kg) was obtained at a reaction time of 70 minutes while at a reaction time of 85 minutes the lowest glycerol yield (12.85 kg) was produced. Based on these findings, in the next stage of the experiment adopted a reaction time of 80 minutes was selected as the optimum reaction time.

3.2 Effect of Variation of Reaction Temperature on Biodiesel Yield

In order to ascertain the influence of reaction temperature of the yield of biodiesel, four samples of waste vegetable oil with equal mass of 20 Kg were selected. The methanol to oil ratio was kept constant at 5:1, the reaction time was kept constant at 80 minutes and the reaction was carried out at the following temperatures, 40, 45, 50, 55, 60 and 65 °C respectively. The corresponding biodiesel yield as shown in Table 2 were measured and recorded upon completion of the reactions.

Table 2: Biodiesel yield obtained from varying reaction temperature

Reaction Parameters	1st Run	2nd run	3rd run	4th run	5th run	6th run
Temperature °C	40	45	50	55	60	65
WVO Quantity(Kg)	20	20	20	20	20	20
Methanol Quantity (Kg)	4	4	4	4	4	4
Reaction time (minutes)	80	80	80	80	80	80
Biodiesel yield (%)	78.64	79.08	81.20	83.55	84.37	83.82

As shown in Table 2, an increase in biodiesel yield with an increase in temperature was observed. This behaviour was noticed at temperatures of 40, 45, 50, 55 and 60 °C thereafter a decline in the yield was observed at 65 °C. The highest yield was obtained at 60 °C, therefore representing the optimum temperature for the production of biodiesel from the plant. This result greatly correlated with the studies conducted by several researchers (Olagunju and Musonge, 2017). With this realization, the reaction temperature was adopted for the next stage of experiment.

3.3 Effect of Methanol-Oil ratio on Biodiesel yield

To investigate the effect of methanol/oil molar ratio on the biodiesel yield, the following parameters were kept constant;

i. catalyst concentration at 2 % (w/w of oil)
ii. reaction time was kept constant at 80 minutes
iii. Reaction temperature at 60 °C
iv. Oil quantity at 20 Kg

The methanol/oil molar ratio was changed as 3:1, 5:1, 8:1, 10:1, 12:1 and 15:1 representing six sets of experimental runs. The results in Table 3 show that with an increase in molar/oil ratio from 3:1 to 5:1 an increase in biodiesel yield was observed. At 5:1 methanol/oil ratio a biodiesel yield of 83.87% was obtained. Surprisingly, an increase in the ratio from 5:1 to 8:1 produced a yield of 79.24% indicating a reduction of about 5% yield.

Table 3: Effect of variation of methanol/oil ratio on biodiesel yield

Reaction Parameters	1st Run	2nd Run	3rd Run	4th Run	5th Run	6th Run
WVO Quantity (kg)	20	20	20	20	20	20
Reaction time (minutes)	80	80	80	80	80	80
Reaction temperature (°C)	60	60	60	60	60	60
Catalyst concentration (%)	2	2	2	2	2	2
Methanol/ Oil ratio	3:1	5:1	8:1	10:1	12:1	15:1
Biodiesel Yield (%)	76.50	83.87	79.24	72.16	70.83	69.62

Subsequent increase in methanol ratio from 8:1 to 10:1 further reduced the biodiesel yield to 72.16 %. The least biodiesel yield was recorded at 15:1 methanol/oil ratio Therefore the optimum methanol/oil ratio was taken as 5:1 due to the highest yield observed. It was deduced that the biodiesel yield could be elevated by addition of excess methanol to an extent. The molar ratio of alcohol to oil is the most significant variable affecting the product yield. Stoichiometrically, the transesterification requires three moles of alcohol and one mole of oil to obtain three moles of biodiesel and one mole of glycerol. However, in practice transesterification is an equilibrium reaction which requires excess alcohol to push the reaction toward completion. Additionally, a higher molar ratio of alcohol negatively affects the yield because of its interference the glycerol separation due to the increase in solubility. When glycerol is kept in solution, it pushes the equilibrium to the left hand side, causing a reduction in biodiesel yield. (Barnwal and Sharma 2005). For the next stage the methanol/oil ratio of 5:1 was adopted.

3.4 Effect of Catalyst Concentration

To study the impact of catalyst concentration on biodiesel yield, other variables such as temperature, reaction time, methanol/oil molar ratio, were kept constant. Potassium hydroxide an alkaline catalyst was selected at different concentrations; 0.5, 1, 1.5 and 2.0, 2.5, and 3.0wt. % for the triglyceride. 0.5 wt.% gave off 76.40% biodiesel yield and 1.5 wt.% yielded 81.25% biodiesel as shown in Figure 3. For 2.0 wt.% a biodiesel yield of 72.68% was observed indicating a reduction of biodiesel yield due to the higher concentration of catalyst which negatively affected the formation of biodiesel. On the other hand, 1 wt.% catalyst concentration gave the highest yield of 86.31% representing the optimum catalyst concentration. The reduction in yield with increase in catalyst concentration above 1wt.% could be attributed to the formation of soaps particles observed during the reaction.(Predojevic, 2008).

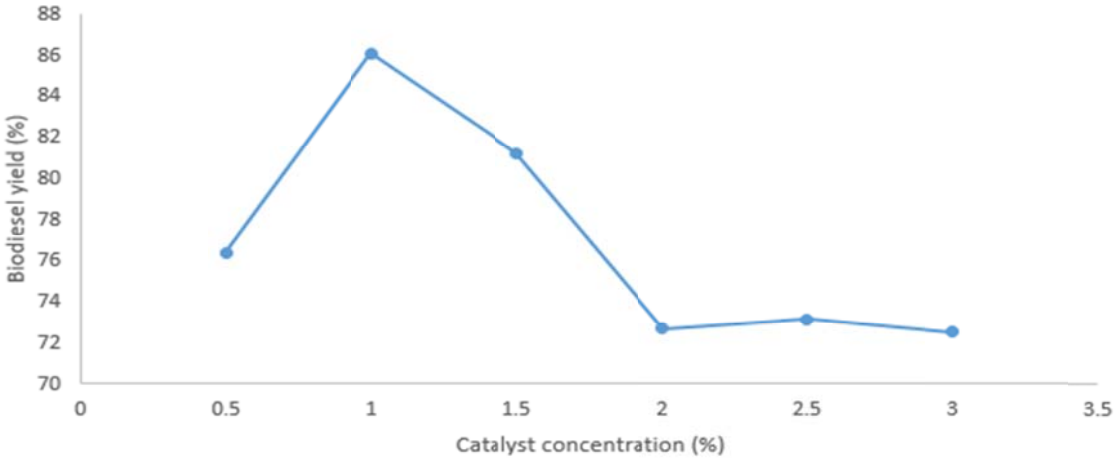

Figure 3: Effect of catalyst concentration on the biodiesel yield.

3.5 Conversion Efficiency of the Plant

As can be seen in Table 1, 54.6 kg of WVO undergoes transesterification reaction to yield 46.47 kg of biodiesel. Hence the conversion efficiency was calculated as 85.10 % using Equation 1. Again, with a flow capacity of 1.2 m^3/hr, density of oil as 910 kg/ m^3, acceleration due to gravity of 9.81 m/s^2, a head of 1.2 m. The pump power loss was calculated as 0.00357 kW. The power lost by heating elements was determined as 0.24 kW. Hence the total power loss in the mini plant is calculated as the sum of the heat loss due to pump and due to the heating elements. This resulted in a power loss 0.2436 kW. The efficiency of the mini biodiesel production plant was thus calculated as 88.67 %.

3.6 Product Characterization

Table 4 contains the results of the physical and chemical properties of WVO and WVO biodiesel. The density of the waste vegetable oil was determined as 920 Kg/m^3 which was reduced to 886 Kg/m^3 after conversion. The viscosity of the WVO at 40°C was determined as 7.15 mm^2/s which was decreased to 6.2 m m^2/s which represents an acceptable value according to ASTM specification. This property of the sample is necessary for the adaptability to diesel engines especially during low temperatures.

Table 4. Properties of WVO and WVO Biodiesel as compared with biodiesel specification

Parameters	Waste Vegetable oil	Biodiesel	ASTM Standard
Acid Value (mg KOH/g Oil)	2.80	0.57	≤ 0.5
Flash Point (°C)	205	134	130-170
Density at 20°C (Kg/m^3)	920	886	860-900
Viscosity at 40°C (mm^2/s)	7.15	6.2	1.9-6.0
Cloud Point °C	46	3	-3 to 12
Cetane Number	40.57	55.38	48-65
Moisture Content %	0.048	0.026	0.05 max

An acid value of 2.80 mg KOH/g Oil was determined for the WVO but after conversion it reduced to 0.57 mg KOH/g Oil which is a little above the ASTM specification as seen in Table 4. A flash point of 205 °C was measured for the waste vegetable oil but upon conversion it fell to an acceptable value of 134 °C (Atadashi *et al*., 2013). This indicates the minimum temperature with which the vapor of the biodiesel can ignite. This property is very important for safety especially when storing and transporting the product. A cetane number of 55.38 was determined for the biodiesel which falls within the acceptable range. Angelovic *et al*. (2014) in their study on determination model for cetane number of biodiesel confirmed the cetane number as one of the most important fuel quality indicators. This property signifies a good measure of fuel ignition delay and combustion quality.

4. CONCLUSION

Procedure for evaluation of a mini biodiesel production plant is presented. The study considered a mini biodiesel production which is designed to generate diesel from feedstock comprising of waste vegetable oil (WVO). The evaluation considered the biodiesel yield from a given volume of WVO and hence the WVO to biodiesel conversion efficiency of the plant is obtained.

The procedure for product characterization is also presented. In the study, approach for the determination of the biodiesel acid value, biodiesel density, biodiesel specific gravity, biodiesel viscosity, biodiesel flash point and biodiesel cloud point are presented and the listed parameter values were determined for sample biodiesel produced from the mini biodiesel production plant

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