Experimental Procedure and Discussion of Results

4.1 Production of biodiesel from soybean oil

The objective of this experiment is to validate the modification as well as the operation procedure. The soybean oil used due low FFA content

4.1.1 Raw Materials for Biodiesel Production

The raw materials for biodiesel production are

1) Vegetable oils.
2) Short chain alcohols.
3) And Catalyst material.

4.1.1.1 Vegetable oils

The oils most used for worldwide biodiesel production are rapeseed (mainly in the European Union countries), soybean (Argentina and the United States of America), palm (Asian and Central American countries) and sunflower, although other oils are also used, including peanut, linseed, safflower, used vegetable oils.

In this project used soybean because low FFA(less than 1%) this why no need for pre-treatment process and available Knothe G et al [32].

4.1.1.2 Short chain alcohols

Methanol is the most frequently used alcohol although ethanol can also be used.

In this project used methanol because ethanol fixed boiling solution with water, which could lead to complicating recovered and recycled in the process.

4.1.1.3 Catalyst material

The catalysts used for the transesterification of triglycerides may be classified as basic, acid or enzymatic.

Basic catalysts include sodium hydroxide (NaOH) and potassium hydroxide (KOH). Acid catalysts include sulfuric acid, sulfonic acids and hydrochloric acid.

In this project used sodium hydroxide (NaOH) because available.

4.2 Tools used in test

1- The Reactor.
2- Electrical heater.
3- Thermometer.
4- Flask titration.

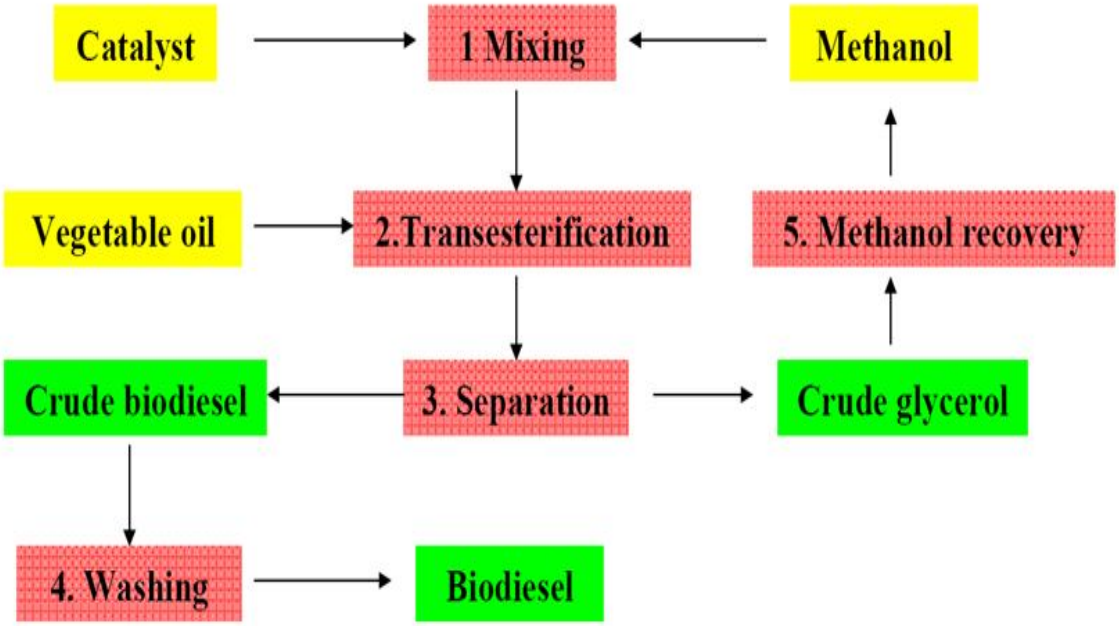

Figure (4-1) Procedures of producing biodiesel from fresh vegetable oil

4.3 Operations during the experiment

4.3.1 Heating of Water

In order to keeping temperature of oil during the reaction. The ideal temperature range is 60 ° C to 70 ° C. the reaction can take more time and will be gets loss in temperature of oil. For decrease these loss been heating oil.

4.3.2 Heating of Oil

In order to speed up the reaction, the oil must be heated. The ideal temperature range is 55°C to 65°C. Heating with electric elements is usually the easiest way to bring the oil up to temperature.

4.3.3 Titration

In order to determine the percent of free fatty acids (FFA) in the oil, a process called titration is used. The vegetable oil is first mixed with methanol. Next, a mixture of Sodium Hydroxide (NaOH) and water is added until all of the FFA has been reacted. This is confirmed by checking the pH of the mixture.

The following items are needed to perform an accurate titration:

- Two 50mL flasks
- One 5mL graduated pipette
- A 1mL dropper
- A 10mL dropper
- A mixture of NaOH and water in 0.1% concentration
- PH solution and color chart

The NaOH water mixture can be prepared by adding 1 gram of NaOH to 1000ml distilled water. The mixture will be more accurate if it is first made as a 1% solution (10grams NaOH to 1000ml water). Next, add 100mL of

the 1% solution to 900ml of distilled water. This will make a 0.1% NaOH solution.

4.3.3.1 The process for titration is as follows

1) Place 10mL of Methanol in a 50ml flask
2) Add 1mL of vegetable oil (mix the oil thoroughly prior to drawing 1ml)
3) Mix the oil with the Methanol using the squirting action of the dropper
4) Add the ph indicator solution (usually 3 drops, check instructions)
5) Place 15ml 0.1% NaOH (known as titrant) solution in a 50ml flask
6) Draw exactly 5ml of the NaOH solution into the graduated pipette.
7) add the 0.1% NaOH to the methanol/oil mixture one drop at a time. Mix the solution using a swirling action between the drops. Using the eyedropper to mix the solution may help if the oil forms drops in the bottom of the flask.
8) Continue to add 0.1% NaOH until a pH of 9 (blue-green color) is reached. This may require more than 5ml. Refill the pipette and continue.

From titration for 4.5 liters of vegetable oil the required amount of catalyst is 15.75 gram of NaOH.

4.3.4 Mixing of Methanol and Catalyst

The purpose of mixing methanol and the catalyst (NaOH) is to react the two substances to form Methoxide. The amount of Methanol used should be 20% of the volume of the oil. Methanol and NaOH are dangerous chemicals by themselves, with Methoxide even more so. None

of these substances should ever touch skin. Vapors should not be inhaled. Gloves, goggles and ventilation are required at all times when working with these substances.

NaOH does not readily dissolve into Methanol. It is best to turn on the mixer to begin agitating the Methanol and slowly pour the NaOH in. When particles of NaOH cannot be seen, the Methoxide is ready to be added to the oil. This can usually be achieved in 20 –30 minutes.

Figure (4-2) Mixing of Methanol and Catalyst

4.3.5 Draining of Glycerol

After the transesterification reaction, one must wait for the glycerol to settle to the bottom of the container. This happens because Glycerol is heavier then biodiesel. The settling will begin immediately, but the mixture should be left a minimum of eight hours to make sure all of the Glycerol has settled out. The Glycerol volume should be approximately 20% of the original oil volume. Figure (4-3) shows the difference in viscosity and color between the two liquids. The object is to remove only the Glycerol and stop when the biodiesel is reached. Glycerol looks very dark compared to the yellow biodiesel. The viscosity difference is large enough between the two liquids that the difference in flow from the drain can be seen.

Figure (4-3) Process separating glycerol from biodiesel

4.3.6 Washing of Fuel

The purpose is to wash out the remnants of the catalyst and other impurities. There are three main methods:

- Water washes only (a misting of water over the fuel, draining water off the bottom)
- Air bubble washes (slow bubbling of air through the fuel)
- Air/water bubble wash (with water in the bottom of the tank, bubbling air through water and then the fuel).

used method number one (water wash only). In washing tank added about 1.6 liters of water to biodiesel and mixing about 10 minutes then were left about one hours after that separated the water from the biodiesel ‘ process is repeated several times until become a pure water. After that extracted biodiesel.

Figure (4-4) Washing of biodiesel

4.3.7 Making of soap

For the work soap we did solution between water (40 milliliters) and sodium hydroxide (4grams) after that heated the glycerol up to 75°C until evaporatives the methanol. Then added the solution to glycerol then heated the mixing about 10 minutes then poured into molds and left for a period 24 hours.

Figure (4-5) Final product of soap

4.4 Transesterification (biodiesel reaction for oils)

The transesterification process can be summarized in the following steps:

1) Heat water to 70 ° C.
2) Add 4.5 liters of oil and heated to 60 ° C.
3) Titrate the oil (determine how much sodium hydroxide to add)
4) Mix the sodium hydroxide (15.75 gram of NaOH) and methanol (1.0 liters of methanol) to make methoxide.
5) Mix the methoxide with the oil (for about 30 minutes).
6) after 4 hours were separated glycerol for biodiesel.
7) Drain glycerol.
8) Wash biodiesel.
9) Make soap.

4.5 Reactor performance

Although the developed processor is fabricated using the available and cheap materials, but it achieved good performance in term of producing biodiesel in adequate quantity and shorter time. Also the operation procedure is worked effectively.

The overall performance is summaries as follows:

- Reaction temperature should be maintained at constant value by using controller which is not included in this processor this why hot water bath is used, if is replaced by heater with controller, then the water tank is eliminated.
- Using thermometers for checking the heat of the oil and the water is not effective.
- Using electrical motor for stirring with 400 rpm increased the output of biodiesel.
- Production of 4 liters of biodiesel from 4.5 liters of oil is considered high production yield (88%) compare to the simple techniques and materials used for fabrication. This yield can be maximized by using the optimum process parameters.
- Glycerin & soap are a bio-product produced in the production process which increases the feasibility of this processor and raises its economy.

4.6 Results and Discussion

4.6.1 Density

Relative density otherwise known as the specific gravity refers to the ratio of the density of a fuel to the density of water at same temperature with its other properties could be judged.

The value of density for test at a temperature of 30 ºC is presented in Table 4.1. From the results it appears that the test density was found 930 kg/m^3 for biodiesel. The standard density of biodiesel (@ 40 ºC 860-900 kg/m^3) but increase the temperature dropped the density.

4.6.2 Flash Point

Flash point is the lowest temperature corrected to a standard atmospheric condition at which application of a test flame Cause the vapor specimen to ignite under specific conditions of test.

The value of flash point for test is presented in Table 4.1. From the results it appears that the test flash point was found 169 ºC for biodiesel.

So within suitable limits because the standard biodiesel flash point is (≥130).

4.6.3 Kinematic Viscosity

The resistance to flow exhibited by fuel blends is expressed in various units of viscosity. It is a major factor of consequence in exhibiting their suitability for the mass transfer and metering requirements of engine operation. Higher the viscosity results low volatility and poor atomization of oil during injection in CI engine, that results in complete combustion and ultimately carbon deposit on injector nozzle as well as in the combustion chamber.

The value of viscosity for test is presented in Table 4.1. From the results it appears that the test viscosity was found (@ 30 ºC 10.40264151 mm^2/sec for biodiesel.

4.6.4 Heating Value

Heating Value of a fuel is the thermal energy released per unit quantity of fuel when the fuel is burned completely and the products of combustion are cooled back to the initial temperature of the combustible mixtures.

The value of heating value for test is presented in Table 4.1. From the results it appears that the test heating value was found (44.0328 MJ/kg) for biodiesel.

There is difference with standard because the reservoir cannot completely seal & process reading of the temperature of oil is not accurate.

4.6.5 Pour Point

Pour Point is the temperature where fuel becomes solid and can no longer be pumped. That temperature is a good bit colder than the cold filter plugging point. The value of pour point for test is presented in Table 4.1. From the results it appears that the test pour point was found (15) for biodiesel.

Table4.1. Fuel properties

property / Type Of fuel	Density Kg/m^3	Viscosity mm^2/sec	Flash point ºC	Pour point	Heat value MJ/kg
Petroleum diesel	@ 15 ºC 833-845	@ 40 ºC 1.3-48	52-80	-35 to 10	43-47
ASTM biodiesel	@ 40 ºC 860-900	@ 40 ºC 3.5-48	≥130	-15 to 15	40-43.33
Biodiesel from test	@ 30 ºC 930	@ 30 ºC 10.40264151	169	15	44.0328