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SYNTHESIS OF BIODIESEL FROM VEGETABLE OIL

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Abstract-*Vegetable oils and animal fats can be transesterified to biodiesel for the use as an alternative diesel fuel. Biodiesel production is a very modern and technological area for researchers nowadays because the price of petroleum is increasing day by day and it has much environmental advantages. The increasing awareness of the depletion of fossil fuel resources and the environmental benefits of biodiesel fuel has made it more attractive in recent times. I select this it as my undergraduate research topic because it is economical, renewable, non-toxic, and biodegradable; it reduces dependency on Crude Oil and supports the farmers for growing vegetables which we used as our raw material. Different studies have been carried out using different oils as raw material and different alcohols as well as different catalysts. In my work I select soybean oils as raw material and three different types of catalysts such as a combination of sodium hydroxide and potassium hydroxide as double base catalyst; sodium hydroxide as single base catalyst and sulfuric acid as acid catalyst. The ratio of alcohol to oil is the most important parameter in the production of biodiesel. An excess of alcohol is required to drive the reaction to the right. In the experiments I select the ratio of alcohol to oil is 5:1 in case of alkali-transesterification and 30:1 in case of acid-transesterification. The effects of the reaction time, rate of mixing and the reaction temperature during the experiments were studied properly. The properties of the produced biodiesel are almost near to the ASTM standard.*

Key words: Biodiesel, Trans-esterified, Vegetable oil, Biodegradable, Soybean oil, Catalysts, Reaction time.

1. INTRODUCTION

The American Society for Testing and Materials (ASTM) defines biodiesel fuel as mono-alkyl esters of long chain fatty acids derived from a renewable lipid feedstock, such as vegetable oil or animal fat. Biodiesel fuel consists of the simple alkyl esters of fatty acids, is presently making the transition from a research topic and demonstration fuel to a marketed commodity. Annual US production in 2001 has been estimated at 57–76 million liters [1], with European production more than 10 times that size [2]. It is predicted that, in the US alone, production will reach 1.3 billion liters annually by 2011 [2].Countries can use their own natural resources for the production of biodiesel. Biodiesel can use the existing distribution infrastructure of the fossil diesel. An alcoholic of vegetable oils is an important reaction to produce fatty acid alkyl esters which are excellent substitutes for diesel fuel and valuable intermediates in oleo-chemistry [3][4]. Biodiesel is obtained from both vegetable oils such as sunflower oil, canola oil, soybean oil, palm oil, rapeseed oil, peanut oil, cotton seed oil and animal fats such as beef tallow, and lard.

Biodiesel can be obtained also from other sources such as waste cooking oil, algae, and greases [5]. Biodiesel can be produced by several methods such as direct use or blending, micro-emulsion, thermal cracking (pyrolysis) and trans-esterification. Among them trans-esterification is the most common method for producing biodiesel which includes acid-catalyzed processes, base catalyzed processes, lipase-catalyzed processes, non-ionic base-catalyzed processes, and heterogeneously catalyzed processes [6][7]. Biodiesel causes less damage to the environment than fossil diesel and is an alternative fuel in order to reduce carbon dioxide emissions. It is derived from a renewable, domestic resource, thereby relieving reliance on petroleum fuel imports. It is biodegradable and non-toxic. Compared to petroleum-based diesel, biodiesel has a more favorable combustion emission profile, such as low

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emissions of carbon monoxide, particulate matter and unburned hydrocarbon.

Biodiesel production is a very modern and technological area for researchers due to the relevance that it is winning every day because of the increase in the petroleum price and the environmental advantages. In this work it is made a review of the alternative technological methods that could be used to produce this fuel. Different studies have been carried out using different oils as raw material, different alcohol (methanol, ethanol) as well as different catalysts, homogeneous ones such as sodium hydroxide, potassium hydroxide, sulfuric acid and supercritical fluids, and heterogeneous ones such as lipases.

The homogeneous alkaline transesterification is the preferred process by the industry in order to produce biodiesel. Usually the homogenous alkaline catalyst is faster and less expensive compared with homogenous acid catalyst or other catalysts. Strong bases such as NaOH and KOH are the most used catalysts. For a basic catalyst, either sodium hydroxide (NaOH) or potassium hydroxide (KOH) should be used with methanol or ethanol as well as any kind of oils, refine, crude or frying. In this process it is better to produce the Alcoxy before the reaction to obtain a better global efficiency. The alcohol–oil molar ratio that should be used varies from N ¼ 1:1–6:1. However N ¼ 6:1 is the most used ratio giving an important conversion for the alkali catalyst without using a great amount of alcohol. The types of alcohol are usually methanol and ethanol. The last one has fewer safety problems because it is less toxic.

The amount of catalyst that should be added to the reactor varies from 0.5% to 1% w/w [8][9], but some authors prefer advice any values between 0.005% and 0.35% w/w [10] should be used. The last but not least important variable is the reaction temperature. The standard value for the reaction to take place is 60°C, but depending on the type of catalyst different temperatures will give different degrees of conversion, and for that reason the temperature range should be from 25°C to 120°C

The reason why there is a great interest in the alkali process is it is more efficient and less corrosive than the acid process, making it a preferred catalyst to be used in industries. Kinetics studies have been made by Freedman [11], Noureddini and Zhu [12] and Darnoko and Cheryan [13].

Acid catalyzed system is the second conventional way of making the biodiesel. The idea is to use the triglycerides with alcohol and instead of a base to use an acid—the most commonly used is sulfuric acid [9] [14][15] and some authors prefer sulfonic acid [16]. This type of catalyst gives very high yield in esters but the reaction is very slow, requiring almost always more than one day to finish. The molar ratio of alcohol to vegetable oil is one of the most important factors that influences the conversion of vegetable oil to its ester. For the stoichiometric reaction, 3 moles of alcohol (96 g of methanol) are required per mole of oil (885 g of soybean oil). In reality, the molar ratio should be higher than the theoretical ratio to drive the reaction to completion. Formo (1954) advocates the use of a large excess of methanol (15 to 35 moles per mole of fatty acid) when using sulfuric acid as a catalyst. He noted that after refluxing for several hours ester yields of 95% or higher can be obtained [17]. Freedman and Pryde found that a 30:1 molar ratio of methanol to soybean oil with 1% sulfuric acid gave good conversion after 44 h of heating at 60°C [11]. The actual conversion percentage was not stated. They emphasized that if vegetable oil has more than 1% free fatty acid, the acid catalyst will be much more effective than the alkali catalyst. Freedman et al. (1984) compared molar ratios of 6:1, 20:1, and 30:1 for acid catalysis with methanol. The molar ratios of 6:1 and 20:1 gave unsatisfactory ester conversion after 18 h of reaction time. The highest conversion with acid catalyst was measured at the 30:1 molar ratio.

In this study, to investigate the effect of molar ratio, five different molar ratios, 3.3:1, 3.9:1, 6:1, 20:1, and 30:1, were selected. Each reaction was run for 48 h with 3% sulfuric acid catalyst at 60°C. Ester conversions reached 98.4% at the 30:1 molar ratio. The specific gravity of the ester decreased with increasing molar ratio. This was probably due to a decrease in residual triglycerides.

Another important factor that affects the conversion of vegetable oil to its ester is the reaction temperature. Liu (1994) noted that heating is required for faster reaction and the reaction time may vary from a few minutes to several hours for acid catalysts [18]. For this experiment, three different reaction

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temperatures, 25°C, 45°C, and 60°C, were selected. The highest reaction temperature, 60°C, was selected because it is near the boiling point of methanol (65°C). Each reaction was run for 48 h with 3% sulfuric acid catalyst and a 6:1 molar ratio of methanol to oil. The changes in ester formation and the specific gravity of the methyl ester. As can be seen, the ester conversion increased almost linearly with increasing reaction temperature. Ester conversions were 8.3%, 57.2%, and 87.8% at 25°C, 45°C, and 60°C, respectively..

2. EXPERIMENTAL

2.1 Mixing of methanol and catalyst

First of all, 7 gm. (KOH+NaOH), 7.5 gm. NaOH, 8 ml sulfuric acid was measured in scale. A small lightweight plastic bag was used during measurement. Then, the (KOH+NaOH) and NaOH catalyst was mixed with 160 ml methanol and 8 ml sulfuric acid with 430 ml methanol in three separate sturdy, heat proof glass bottles with narrow neck. The solution was mixed constantly to quick dissipate the heat given off by the reaction. The mixing process took about 15 minutes.

2.2 Transesterification

After mixing the methanol and catalyst as described before, vegetable oil (800 ml for (KOH+NaOH) and NaOH catalyst and 350 ml for sulfuric acid catalyst) was poured into the container and the container was heated to about 50℃ to 60℃ (120-140°F) by the electric cooker. The temperature was kept below 60℃ (140°F) since methanol could boil at 65 ℃ (148.5°F) and be lost. Care was taken to avoid sparks or open flames near reactor, as methanol vapor was explosive. Then the oil was poured into the conical flask that was kept on magnetic stirrer. The heated oil was stirred well; the methanol-catalyst mixture was added to the oil carefully. The reaction will start immediately; the mixture rapidly transforms into a clear golden liquid. Stirring was continued for an hour by the magnetic stirrer while keeping the temperature at ~ 60℃.Then the mixture was allowed to settle overnight. The system was kept closed to the atmosphere to prevent loss of methanol during the reaction. The reaction took about 18 hours to complete using alkali catalyst and 50 hours to complete using acid catalyst.
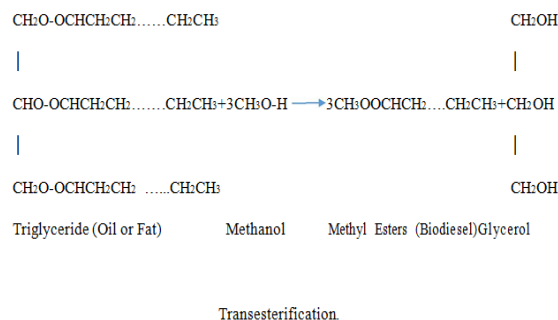
$$CH_2O-OCHCH_2CH_2......CH_2CH_3 \qquad\qquad CH_2OH$$
$$|\qquad\qquad\qquad\qquad\qquad\qquad\qquad\qquad\qquad |$$
$$CHO-OCHCH_2CH_2.......CH_2CH_3+3CH_3O-H \longrightarrow 3CH_3OOCHCH_2....CH_2CH_3+CH_2OH$$
$$|\qquad\qquad\qquad\qquad\qquad\qquad\qquad\qquad\qquad |$$
$$CH_2O-OCHCH_2CH_2 ......CH_2CH_3 \qquad\qquad CH_2OH$$

Triglyceride (Oil or Fat)　　　Methanol　　　Methyl Esters (Biodiesel)Glycerol

Transesterification.

Figure 1: Process of Transesterification

2.3 Separation

As soon as the reaction was completed, the mixture was kept for settling and screwed the cork tightly. The mixture was allowed to settle 18 hours. After settling, there was two phases in the bottle with a clear interface. Dark-colored glycerol byproduct was at the bottom, with crude biodiesel was on top. The biodiesel varies in color depending on the oil used, but is usually pale yellow. The top layer was carefully removed into a clean PET bottle. It was confirmed to not inadvertently mix up the glycerol layer with the biodiesel.

2.4 Crude biodiesel washing

The crude biodiesel still contains contaminants such as soaps, excess methanol, residual catalyst, and glycerol. It was purified by washing with warm distilled water to remove residual catalyst or soaps. The washing procedure was effective because the residues are more readily dissolved in water. When the two types of liquid were mixed into a homogenous emulsion, the residues were transferred from a biodiesel-phase into a water phase. The two liquids were eventually separate into two phases; thus, the residues were washed from crude biodiesel with water. The washing procedure involved using two 1-liter PET bottles in succession as follows: A small hole in the bottom corner of each bottle is pierced and the holes were securely covered with duct tape. 200 ml crude biodiesel was poured in the biodiesel into one of the wash bottles. 0.4 liter distilled water was heated. Then it was poured into the crude biodiesel. Stirring was continued until oil and water are well mixed and appear homogenous. Then the mixture was allowed to settle for two hours or more. The water was drain off from the bottom of the bottle by removing the duct tape from the hole.

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The flow of water was stopped with finger when the biodiesel had begun to flow out the hole. Then the biodiesel was transferred to the second washing bottle, fresh water was added and again washed. The first bottle was cleaned and the duct tape is replaced. This procedure was repeated for three times.
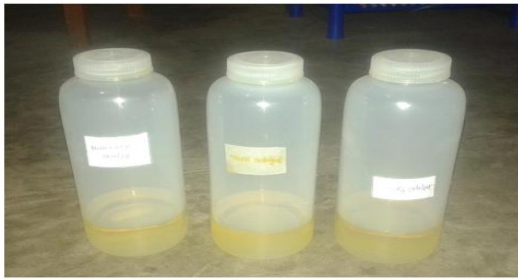
Biodiesel after washing.

Fig 2: Preserved in bottle after proper purification

3. RESULT AND DISCUSSION

3.1 Measurement of calorific value

Calorific value is the amount of heat energy released when unit quantity of burn completely and product of combustion are cooled back to STP. The unit of calorific value for fuel is J/kg. Calorific value of the biodiesel can be determined by "Bomb Calorimeter". A bomb calorimeter is a type of constant-volume calorimeter used in measuring the heat of combustion of a particular reaction. Bomb calorimeters have to withstand the large pressure within the calorimeter as the reaction is being measured. Electrical energy is used to ignite the fuel; as the fuel is burning, it will heat up the surrounding air, which expands and escapes through a tube that leads the air out of the calorimeter. When the air is escaping through the copper tube it will also heat up the water outside the tube. The change in temperature of the water allows for calculating calorie content of the fuel. In more recent calorimeter designs, the whole bomb, pressurized with excess pure oxygen (typically at 30atm) and containing a weighed mass of a sample (typically 1-1.5 g) and a small fixed amount of water (to saturate the internal atmosphere, thus ensuring that all water produced is liquid, and removing the need to include enthalpy of vaporization in calculations), is submerged under a known volume of water (ca. 2000 ml) before the charge is electrically ignited. The bomb, with the known mass of the sample and oxygen, form a closed system - no gases escape during the reaction. The weighed reactant put inside the steel container is then ignited. Energy is released by the combustion and heat flow from this crosses the stainless steel wall, thus raising the temperature of the steel bomb, its contents, and the surrounding water jacket. The temperature change in the water is then accurately measured with a thermometer. This reading, along with a bomb factor (which is dependent on the heat capacity of the metal bomb parts), is used to calculate the energy given out by the sample burn. A small correction is made to account for the electrical energy input, the burning fuse, and acid production (by titration of the residual liquid). After the temperature rise has been measured, the excess pressure in the bomb is released. The crucible of the calorimeter was fitted with the known muss of fuel and then it was ignited. This heated the surrounding water, and the initial and final temperature was recorded by using thermometer. The calorific was determined using heat balance. Heat given by the fuel is equal to heat gained by the water.[19]
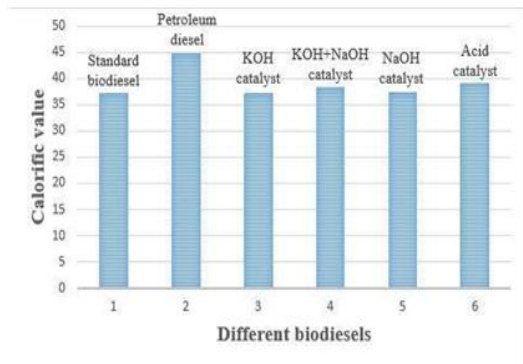
LHS: Mass of fuel ×calorific value.
RHS: Mass of water ×specific heat of water ×change in temperature.
Then, LHS=RHS.

Table 1: Showing different Calorific values

Parameter	Standard biodiesel	Petroleum diesel	Biodiesel using KOH catalyst	Biodiesel using (KOH+NaOH) catalyst	Biodiesel using NaOH catalyst	Biodiesel using H_2SO_4 catalyst
Calorific value	37.27 MJ/kg	44.8 MJ/kg	37.218 MJ/kg	38.321 MJ/kg	37.438 MJ/kg	39.075 MJ/kg

Table 2: Bar Graph showing Comparative values of calorific values.

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Comparison of calorific value of produced biodiesels.

Viscometer.

Fig 3: Viscometer

3.2 Measurement of viscosity

The viscosity of a fluid is a measure of its resistance to gradual deformation by shear stress. For liquids, it corresponds to informal concept of "thickness"; for example, honey has a much higher viscosity than water. The dynamic viscosity of a fluid expresses its resistance to shearing flows, where adjacent layers moves parallel to each other with different speeds. On the other hand, the kinematic viscosity is the ratio of the dynamic viscosity to the density of the fluid. It is expressed by [20]. One of the main purposes of the transesterification reaction was to reduce the viscosity of vegetable oil in order to achieve the properties that are more suitable for its functions fuel. The standard range of viscosity for biodiesel in ASTM D6751 is 1.9 - 6.0 mm^2/sec at 40°C. A viscometer is an instrument used to measure the viscosity of a fluid. For liquids with viscosities which vary with flow conditions, an instrument called a rheometer is used. Viscometers only measure under one flow condition. In general, either the fluid remains stationary and an object moves through it, or the object is stationary and the fluid moves past it. The drag caused by relative motion of the fluid and a surface is a measure of the viscosity. The flow conditions must have a sufficiently small value of Reynolds number for there to be laminar flow. At 20.00 degrees Celsius the dynamic viscosity (kinematic viscosity x density) of water is 1.0038 mPa·s and its kinematic viscosity (product of flow time x Factor) is 1.0022 mm^2/s. These values are used for calibrating certain types of viscometers.
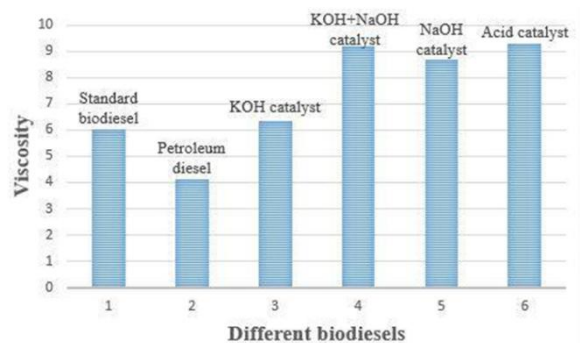
Table 5: Comparative analysis of synthesis biofuel viscosity to different Biofuel

Parameter	Standard biodiesel	Petroleum diesel	Biodiesel using KOH catalyst	Biodiesel using (KOH+NaOH) catalyst	Biodiesel using NaOH catalyst	Biodiesel using H$_2$SO$_4$ catalyst
Viscosity	1.92~6 mm^2/s	1.9~4.1 mm^2/s	6.34 mm^2/s	9.2 mm^2/s	8.7 mm^2/s	9.3 mm^2/s

Table 6: Bar Graph showing Comparative values of biofuel viscosity

Comparison of viscosity of produced biodiesels.

3.3 Measurement of flash point

The flash point of a chemical is the lowest temperature where enough fluid can evaporate to

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form a combustible concentration of gas. The flash point is an indication of how easy a chemical may burn. Chemical with higher flashpoint is less flammable than chemical with lower flash points. [21] The flash point is the lowest temperature at which vapors of the material will ignite, given an ignition source. The flash point not to be confused with the auto ignition temperature (the temperature at which the vapor ignites without an ignition source) or with the fire point (the lowest temperature at which the vapor will keep burning after having been ignited and the ignition source has been removed). The fire point is always higher than the flash point because at the flash point the vapor may cease to burn when the ignition source is removed. Neither the flash point nor the fire point dependent on the temperature of the ignition source, which is much higher. [22]


Flash point measuring device.

Fig 4: Biodiesel Flash point measuring Device
(Bomb Calorimeter)

Table 3: Different Flash point values compared to standard Biofuel

Parameter	Standard biodiesel	Petroleum diesel	Biodiesel using KOH catalyst	Biodiesel using (KOH+NaOH) catalyst	Biodiesel using NaOH catalyst	Biodiesel using H_2SO_4 catalyst
Flash point	93℃	52~96℃	53℃	69℃	56℃	58℃

Table 4: Bar Graph showing Comparative values of flash point
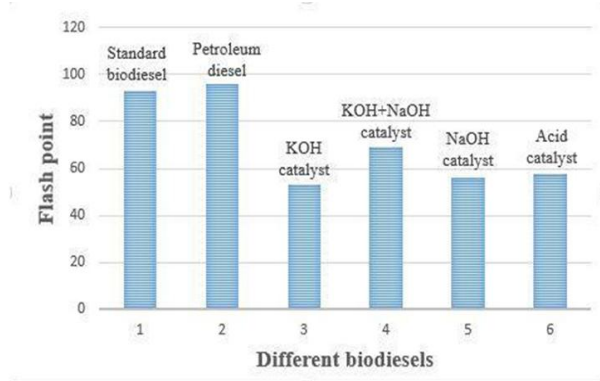
Comparison of flash point of produced biodiesels.

Here lower calorific value was not measured as the vapor could not be stored. So the calorific we measured is higher calorific value. The higher calorific value of the synthesis fuels are 38.321 MJ/kg, 37.438 MJ/kg and 39.075 MJ/kg which are near to the value of the standard biodiesel but the petroleum diesel's higher calorific value is 44.8 MJ/kg.. Therefore, the synthesis fuel is less efficient then the petroleum diesel. The kinematic viscosity of the synthesis fuels are found 9.2 mm^2/s, 8.7 mm^2/s and 9.3 mm^2/s which are not between the range of standard biodiesel because of temperature factor. The synthesis fuels are higher viscous then petroleum diesel. The flashpoint of the synthesis fuels are measured as 69℃, 56℃ and 58℃.

4. CONCLUSION

Continuous production of biodiesel from vegetable oils is very important because it is renewable, degradable, non-toxic and economical. Biodiesel has also much environmental advantages. As it is produced from vegetable oils, so production of biodiesel inspires the farmers to grow more soybean oils, palm oils and so on. Thus it affects our total economy. Moreover it reduces dependency on crude oil and it is good for engine. Biodiesel has become more attractive recently because of its environmental benefits and the fact that it is made from renewable resources. Biodiesel is briefly defined as the mono-alkyl esters of vegetable oils or animal fats. Biodiesel

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is the best candidate for diesel fuels in diesel engines. It burns like petroleum diesel as it involves regulated pollutants. So it necessary to implement the use of biodiesel over the current petroleum and gasoline because of all the merit and advantages it brings forth to the table. In comparison to petroleum and gasoline, biodiesel beats its competitors in all categories of toxic substance emissions and poses close to no threat to the environment. What's more, instead of increasing the carbon dioxide levels in the atmosphere, the overall production and use of biodiesel consumes more carbon dioxide than it emits, thus making it a valuable tool in preventing global warming.

Not only does petroleum diesel harm our environment through emissions of toxic substances, but it also has negative effects on us physically. Many health problems and illnesses have been traced back to emissions from petroleum diesel. These emissions have been related to many cases of cancer, cardiovascular and respiratory disease, asthma and infections in the lungs. By using biodiesel in the place of petroleum diesel, not only will we be helping the environment with a much better alternative, but we would be significantly reducing many health risks. The fact that most biodiesels are domestically produced means that by using more of it, the market of biodiesel would actually stimulate the economy, reducing a country's dependence on foreign oil imports. Also, the implementation of biodiesel is extremely easy and requires little or no modifications to the typical diesel engine, making it a very easy and smooth transition.

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