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# DETERMINATION OF OPTIMAL PARAMETRIC VARIABLES FOR THE CONVERSION OF NON-EDIBLE NEEM OIL TO BIODIESEL

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## ABSTRACT

Biodiesel has become more attractive recently owing to its environmental benefits. Biodiesel fuel is a renewable, biodegradable, non-toxic, and essentially free of sulfur and aromatics. Biodiesel are produced from vegetable oils, animal fats/oil and west cooking oil. The process used to convert these oils to biodiesel is called transesterification in which oil or fats are reacted with alcohol in the presence of catalyst. The most important variables affecting the biodiesel conversion during the transesterification reactions are molar ratio of alcohol to vegetable oil, reaction temperature, reaction time and amount of catalyst. The biodiesels were characterized for their main fuel properties by its viscosity, density and colorific values among others. In this research, non-edible neem oil was used as biodiesel sources, the optimal parametric values for the conversion were investigated, the results show that, the optimal volume can be obtained at around 80 ºC, 63 minutes and 1 % wt of reaction temperature, reaction time and amount of catalyst respectively. The oil to methanol ratio were kept at 6:1 throughout the processes. The average viscosity, density and colorific values were found to be 3.72 $mm^2/s$, 869 g/L and 38301.4366 respectively. The research work could provide some hints on biodiesel conversion and will establish corresponding standards concerning the optimal parametric values of transesterification of non-edible vegetable oils.

**Keywords**: biodiesel, transesterification and renewable energy

## INTRODUCTION

Experts in field of energy predict that the global demand of energy would be about 10 times the present energy by the year 2050 (Chu & Meisen, 2011). Fossil fuels such as natural gas, coal, as well as nuclear power plants represent 80% of the world energy consumption. The problems with energy supply from fossil sources are related not only to global warming but also to environmental concerns such as air pollution, acid precipitation, ozone depletion, forest destruction, and radioactive substance emissions (Wai, Wang, & Lin, 2008). Nuclear power plants also face a lot of challenges related to the processing, disposing the used fuel as well as risk and security (ELECTIVES & No). Hence, there must be an alternative source of energy to prevent these effects; the potential solution is the use of renewable energy.

Among various renewable energies, biodiesel is considered as an alternative fuel that could productively solve the problem of fossil fuel in due future. Moreover, biodiesel offers several advantages compared to diesel fuel because of its renewable, non-toxic, biodegradable, superior lubricity and clean combustion properties (Karmakar, Karmakar, & Mukherjee, 2010). In recent years, green diesel produced from seed oils of several woody plants has been shown with a notable advantage over conventional feedstock across the world (Fan et al., 2016; Wang & Yu, 2012; Xue et al., 2009; Yu et al., 2017).

The techniques usually used for biodiesel fuel production are classified into four: direct, microemulsion, pyrolysis and transesterification reaction (Demirbas, 2009a, 2009b, 2011). However, transesterification reaction is the most used techniques (Fadhil, Sedeeq, & Al-Layla, 2019). Transesterification is the reaction in which triglycerides react with an alcohol in the presence of catalyst to produce biodiesel and by-product, glycerol (Demirbas, 2008a, 2008b). This reaction is mostly influenced by many factors such as: alcohol to oil molar ratio, reaction time, nature and amount of catalyst, reaction temperature, and the nature of feedstocks composition (Demirbas, 2009c). Conventionally, edible vegetable oils such as soybean, palm, sunflower and coconut oils have been widely used as a feedstock for production of biodiesel which contribute to over 95% of global biodiesel production (Helwani, Othman, Aziz, Fernando, & Kim, 2009). This process usually provides high-quality biodiesel fuel with less refining process. But, the high price of vegetable oil as well as its use in food restricts biodiesel production from these feedstocks (Gashaw & Lakachew, 2014). Recently, alternative feedstocks such as natural plant oils, animal fats, waste/used cooking oils, and non-edible feedstocks such as jatropha carcass, neem, and castor oils are used to produce biodiesel fuels, to compensate the high prices of biodiesel fuel and improve its development (Mata, Martins, & Caetano, 2010). Although, these feedstocks are of low prices, but the production and the refining processes of these low-quality feedstocks are difficult (Demirbas, 2010). However, several investigations have revealed the potential of biodiesel production through this low-quality feedstock by varying one parameter and keep the rest constant. In this study, three parameters (reaction temperature, reaction Time and Amount of catalyst) were optimized. And the properties of the produced diesel were investigated.

## MATERIAL AND METHODS

### Materials

### Chemicals

The chemicals used in carrying out the experiments are: Sodium hydroxides, Acetone [$CH_2(CH_2)_2O$, BDH Limited poole, England], Filter paper [12-5cm Grade Whatman, England], Methanol, Crude Neem Vegetable oil [Nigerian Institute of Research and Chemical Technology NIRCT, Zaria].

### Equipment

The list of equipment used for this research are: Magnetic stirrer with thermostatically controlled rotary hot plate [IKA CMAG HS10], Brookfield digital viscometer [Brookfield RVDV-1], Bomb Calorimeter [per 6100], Cheng Sang Vacuum oven [MA 0-30L].

## METHODOLOGY

### Transesterification

Three sets of parameters were optimized in three different experiments for the production of the biodiesel. The optimized parameters are; temperature, time and amount of catalyst

### Varying temperature of reaction

In this case 30 ml of the neem oil was measured using measuring cylinder and free heated to 80 ºC using hot magnetic stirrer with thermometer. Certain amount of sodium hydroxide (1 weight percent of the oil) was added to the methanol (6:1 oil to methanol ratio). The mixture was stirred until all the pellets dissolved into sodium methoxides. The sodium methoxide solution was then added to the pre-heated oil, and the reaction mixture was maintained at 80 ºC for 16 hrs for the proper settle of glycerol. Biodiesel was then separated from the glycerol using separatory funnel. The same procedure was repeated for other values of temperatures (70 ºC, 55 ºC, 40 ºC, 32 ºC).

### Varying amount of catalyst

The time and the temperature of the reaction were set respectively at 40 minutes and 70 ºC, the catalyst concentrations were varied from 2.0 wt % to 0.5 wt % in 0.5 % interval of the oil while oil to methanol ratio maintained at 6.1. The products of the reactions were left for 16 hrs for the proper settling of glycerol for each sample, and then the biodiesels were separated from the glycerol using separatory funnel in each case.

### Varying Time of Reaction

The catalyst concentration and the reaction temperature were respectively maintained at 1 % and 70 ºC because they were the optimal parameters from the above experiments. The reaction times were varied as 57 min, 60 min, 63 min, and 80 min respectively while oil to methanol ratio was maintained at 6.1 ratio. The products of the reactions were left for 16 hrs for the proper settling of glycerol for each sample, and then the biodiesels were separated from the glycerol using separatory funnel in each case.

### Washing of Biodiesel Product

The water washing step was used for the removal of the remaining catalyst, soaps, salts, methanol, or free glycerol from the produced biodiesel. In this case, 20 ml of water was poured gently on to the produced biodiesel and then stirred slowly to avoid foam formation: it should be noted that shaking is not advised at this stage. The mixture was left for 16 hours to settle for two phases (water impurity phase and biodiesel phase), the two phases were then separated using separatory funnel and the biodiesel layer was then heated to about 100 ºC for 1 hour to evaporate the remaining water molecules.

### Determination of Analytical properties

The calorific values were determined using per 6100 bomb colorimeters, the viscosities of the samples were investigated using Brookfield Digital viscometer and final, the density of the biodiesel was investigated.

## RESULTS AND DISCUSSION

### Effect of Reaction Temperature

The reaction temperature was varied from 32 ºC to 80 ºC (fig.1.1). The reaction was carried out for 60 minutes at 1.0 wt % catalyst with fixed oil to methanol ratio of 6:1. It was observed that, the biodiesel conversion was low at lower temperature and the volume attained maximum at 80 ºC. At high reaction temperature, the reactants acquire enough kinetic energy that increases the mass transfer rate among oil-methanol-catalyst phases resulting in maximum biodiesel conversion. The results show that the optimal volume can be obtained at around 80 ºC or above.

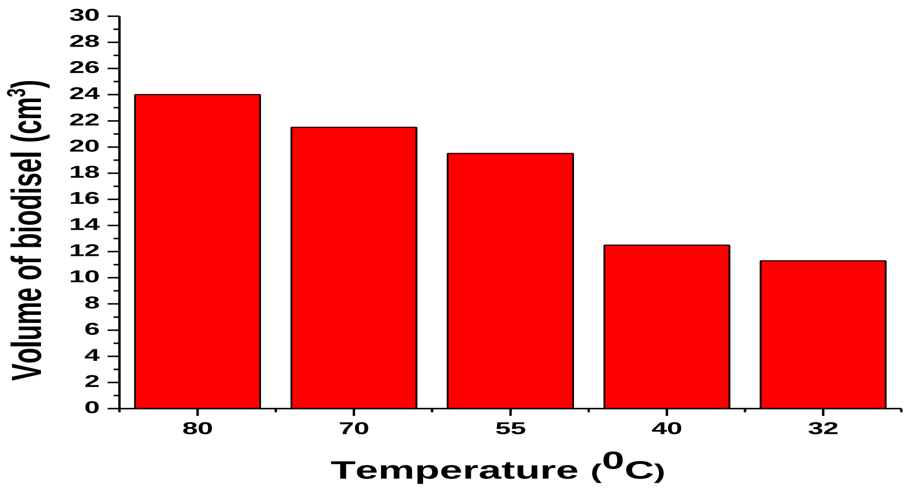


Fig. 1: Effect of temperature on biodiesel conversion

**Effect of Reaction Time:**

The effect of reaction time on biodiesel conversion was studied by varying the reaction time from 57 minutes to 80 minutes using 6:1 oil to methanol ratio and 1.0 wt% catalyst at 70 ºC (fig. 1.2). The results revealed that biodiesel conversion was low at the first 57 minutes, this is because the heterogeneous catalyst was not completely activated giving a low reaction rate. However, with further increase in reaction time, the conversion increased gradually and attained a maximum at 63 minutes. After that at 80 minutes, the biodiesel conversion was observed to be reduced. This behavior may be due to the reverse transesterification occurring between FAME and glycerol when the reaction time is prolonged. Hence, 63 minutes reaction time was considered as optimum time for biodiesel production in this case.

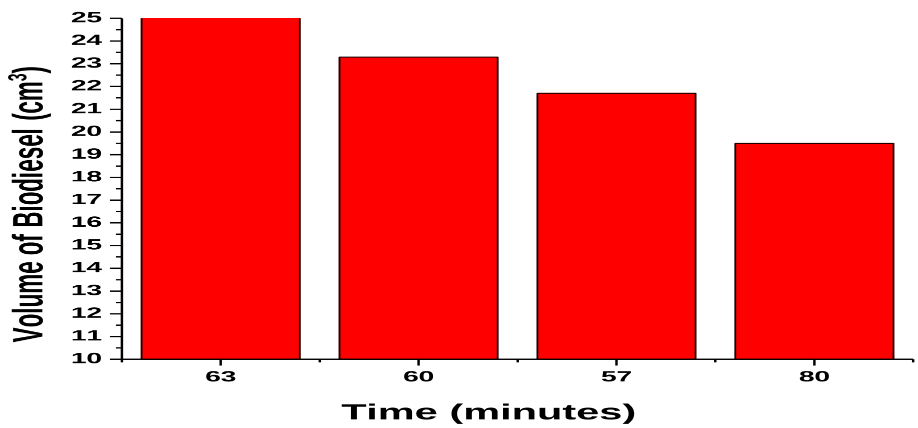


Fig. 1: Effect of Reaction Time on Biodiesel Conversion

**Effect of Catalyst Concentration:**

The effect of catalyst concentration on the conversion of biodiesel was studied by varying the amount of catalyst ranging from 0.5 – 2.0 wt% (fig. 1.3). The reaction was carried out at 70 ºC for 40 minutes with fixed oil to methanol ratio of 6:1. The investigation revealed that when the catalyst concentration was at 0.5 wt%, the biodiesel produced is 20 $cm^3$. Increasing the volume to 1.0 wt% yield a maximum volume of the biodiesel; by further increasing the catalyst amount beyond the optimum value, biodiesel products may be absorbed into the catalyst, which consequently reduce the conversion. The results show that the maximum conversion was obtained at 1.0 wt% of the catalyst While the increase in the catalyst amount be young 1.0 wt % lead to the decreased in the volume of the produced biodiesel.

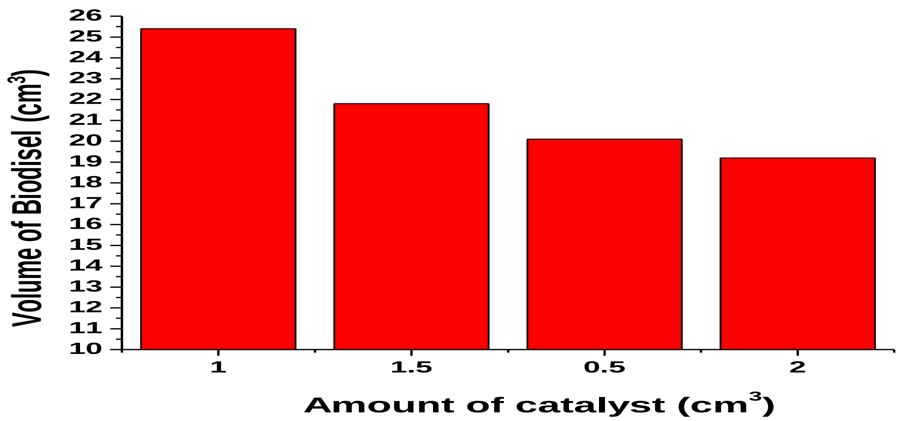

Fig. 2: Effect of Catalyst Amount on Biodiesel Conversion

**Analysis of physical properties of the produced biodiesel**

**Table 1: compares of Viscosities, Densities and colorific Values of biodiesel oils**

| Material property | Viscosity @ 40 ºC (mm$^2$/s) | Density @ 20 ºC (g/L) | Calorific Value (kj/kg) |
|---|---|---|---|
| 0.5 %wt, 57 min, 55 ºC | 3.75 | 871 | 36823.6341 |
| 1.0 % wt, 63 min, 80 ºC | 3.72 | 869 | 38301.4366 |
| 1.5 % wt, 60 min, 70 ºC | 3.71 | 865 | 39221.7324 |
| 2.0 % wt 80 min, 40 ºC | 3.71 | 864 | 40246.5438 |
| Neem oil | 4.98 | 933 | 34321.1231 |
| Diesel oil | 2.76 | 820 | 42200.0000 |

Viscosity is a measure of the internal friction of an oil to flow. Viscosity is the most important property of biodiesel since it affects the operation of fuel injection, particularly at low temperatures. High viscosity leads to low atomization of the fuel spray and hence less accurate operation of the fuel injectors. The viscosity value of the vegetable oil was found to be 4.98 mm$^2$/s, after transesterification the values varied between 3.75 to 3.71 mm$^2$/s. This may be as a result of reduction in glycerol during the transesterification process. It was also observed that the viscosity values in the table 1 decreases with increasing catalyst concentration. The reduction may be as a result of reduction in free fatty acid content with increasing the catalyst.

Density is another important property of biodiesel. It is the weight of a unit volume of fluid. It is also the ratio of the density of a liquid to the density of water. The relative density of biodiesels ranges between 848 and 901 g/L. Fuel injection equipment operates on a volume metering system; hence a higher density for biodiesel results in the delivery of a slightly greater mass of fuel. From table 1, it can be observed that, the density of the neem oil is 933 g/L, after transesterification the value was decreased to 871 g/L at 0.5 % amount of catalyst, the decrease is seemed to be a function of amount of catalyst, this phenomenon may be as a result of reaction between the catalyst and free fatty acid in which the catalyst convert the free fatty acid into soluble salt and the evaporation of the salt through the heating courses the reduction in mass of the oil as a result the density was decreased

Calorific 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. This is an important property of the bio-diesel that determines the suitability of the material as alternative to diesel fuels. The calorific value of vegetable oils and their methyl esters were measured in a bomb calorimeter according to ASTMD240 standard method. The gross calorific values of crude neem oil and neem biodiesels were found to be 34321.1231 and 38301.4366 kJ/kg, respectively, which are lower than 42200.000 kJ/kg for diesel. This could be due to the difference in their chemical composition from that of diesel or the difference in the percentage of carbon and hydrogen content as well as the presence of oxygen molecule in the molecular structure of neem oil and biodiesel.

## CONCLUSION

In this paper the optimal parametric variables for the conversion of neem vegetable oil to biodiesel were investigated, the parameters are: reaction time, reaction temperature and amount of catalyst. The biodiesel properties (viscosity, density and calorific values) were determined and compare with that of diesel oil. the results show that the optimal volume can be obtained at around 80 ºC, 63 minutes and 1 % wt of reaction temperature, reaction time and amount of catalyst respectively. The oil to methanol ratio were kept at 6:1 throughout the processes. The average viscosity, density and calorific values were found to be 3.72 $mm^2/s$, 869 g/L and 38301.4366 respectively. The produced biodiesels show good properties which can be used in diesel engine.

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