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EFFECT OF TEMPERATURE VARIATION ON THE PRODUCTION OF BIODIESEL USING NEEM OIL

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Abstract

Biodiesel was produced from neem seed oil via a two-step process of esterification and transesterififcation reactions. The transesterification was carried out using CH_3ONa as catalyst with ethanol as the alcohol. The reaction temperature was varied between 30, 40, 50, 60, and 70^0C, while all other process parameters were kept constant. From the results obtained, a significant change in biodiesel yield (73-79%) from 30-50^{o}C temperatures was observed. At a temperature of 60^{o}C, a good yield of 94% was obtained which was observed at a temperature below the boiling point of the alcohol used. At 70^{o}C biodiesel yield of 67% was obtained; this indicates a drop in biodiesel yield. Further flash point of 149.6^0C indicated that the biodiesel produced is within the specification of ASTM D6751. Also, the high value of flash point indicated that the fuel is safe for handling as it exceeds the minimun ASTM requirement (130min). It is worthy to mention that other properties such as viscosity, pour point and cloud point etc investigated also presented good values which were within ASTM D6751. The formation of biodiesel was confirmed by FT-IR analysis. The conversion of the ester functional group into methyl esters in biodiesel verified the success of the reaction.

Keywords: Biodiesel; Transesterification; Neem Seed Oil; Ethanol; Sodium Methoxide; Washing.

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1. Introduction

Today, continuous increase in World energy consumption, fuel price hike, depletion of non-renewable fossil fuels and global warming effects are among the greatest challenges facing the globe *(*1*)*. Also environmental concerns have drastically increased globally over the past decade, particularly after the Earth Summit '92 (2). Thus, the most viable approach to meet this rising demand, decrease greenhouse gas emissions and minimize the effects of fossil fuels depletion is by exploring alternative renewable energy sources (3). Biofuels, particularly biodiesel is such a fuel that shows great potential to replace petro-diesel (4). Biofuels are commonly known to offer several advantages over fossil fuel such as sustainability, biodegradability, lower greenhouse gas emissions, regional development, social structure and agriculture development, and fuel security supply (5). Further, replacing petro-diesel with biodiesel fuel could reduce the accumulation of greenhouse gases such as CO_2 in the atmosphere (6). Also biodiesel fuel has been commonly found to offer similar engine performance to that of petro-diesel fuel, whilst reducing engine emissions of particulates, hydrocarbons and carbon monoxide (7). Biodiesel fuel is usually produced from virgin and used vegetable oils and animal fats (8). Presently several efforts are made to produce biodiesel from microalgae. Microalgae clearly offers a few advantages among others include: much higher biomass productivities than land plants (doubling times may be as short as 3.5 h), some species can accumulate up to 20–50% triacylglycerols, while no high-quality agricultural land is necessary to grow the biomass, and even no land at all, offshore microalgae farming could be a reasonable alternative (9).

A number of methods are currently available and have been adopted for the production of biodiesel fuel. There are four primary ways to produce biodiesel: direct use and blending of raw oils, micro-emulsions, thermal cracking, and transesterification. The most commonly used method for converting oils to biodiesel is through the transesterification of animal fats or vegetable oils (10).

Conventionally, the triglycerides of fats and oils are transesterified using short-chain alcohol such as methanol and ethanol in the presence of alkali catalysts. Also, acid catalysts are used for the transesterification reaction. Transesterification reaction is the most adopted process for biodiesel production. At the end of transesterification, biodiesel is mostly separated via gravitational settling or centrifugation. The crude biodiesel is then purified and dried to meet the stringent international standard specification provided by EN14214. The production of biodiesel using alkaline catalysts such as sodium and potassium hydroxides (NaOH and KOH), could provide higher biodiesel yield (>98%) (11). Thus, to the best of my knowledge less work has been done on the production of biodiesel using sodium methoxide. Global energy crisis due to depletion of petroleum resources and increased environmental problems have led to the search for an alternative biodiesel fuel, which should not only be sustainable but also environment friendly. The aim of this study is to produce biodiesel from vegetable oil using sodium methoxide (CH_3ONa) (12).

2. Materials and Methods

2.1. Materials

Sodium methoxide, Tetraoxosulphate IV acid, Ethanol, Neem seed oil, Sodium Sulphate and distilled water. Reactor, Water bath, Beakers, Separating Funnel, Evaporation apparatus, Thermometer and Conical Flasks

2.2. Methods

2.2.1. Transesterification Reaction for the Production of Biodiesel

Biodiesel fuel was produced using a batch reactor using palm oil and methanol at a molar ratio of 6:1. The reaction conditions choosing based on the literature reviewed are as follows; molar ratio of 6:1, stirring rate of 645 rpm and duration of 1hr. Then, the reaction mixture was transferred to a decanter and was kept overnight to settle glycerol reach phase (13).

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2.2.2. Evaporation Process

The biodiesel produced was sent into evaporation apparatus so as to remove the alcohol from the biodiesel by evaporation. The apparatus was set at 70^{o}C for at least 20 min with constant stirring to ensure proper evaporation of alcohol (14).

2.2.3. Washing

The crude methyl esters produced was purified with warm water at 60^{0} C and 10% H2SO4 and 90%.

2.2.4. Drying

The methyl ester layer was dried using anhydrous sodium sulphate and in air ventilated oven

2.2.5. Analysis

Analysis of biodiesel produced was based on the methods developed by American Standard for Testing Method (ASTM D6751) and European Union (EN 14214). Furthermore, methyl esters was analysed using (EN 14103), Total glycerol (ASTM D 6584), Water content (ASTM D 2709), Cetane number (ASTM D 613), Acid value (ASTM D 664), Flash point (ASTM D 93), viscosity (ASTM D 445), and Density EN ISO 3675.

3. Results and Discussions

Biodiesel was produced via the alkaline transesterification of neem seed oil using homogeneous catalyst of sodium methoxide (CH_3ONa). While reaction conditions of time, catalyst concentration, mixing ratio and mixing speed were kept constant, the effect of reaction temperature was investigated through varying the reaction temperature between, 30, 40, 50, 60, and 70^{0}C.

3.1. FT-IR Spectra of Neem Seed Oil

The FT-IR spectra in the mid-infrared region have been used to identify functional groups and the bands corresponding to various stretching and bending vibrations in the samples of oil and biodiesel. The position of carbonyl group in FT-IR is sensitive to substituent effects and to the structure of the molecule (15). The FTIR spectra of oil allowed to identify the typical bands of vegetable oils such as the one located at 1100 - 1170 cm correspondent to the vibrations of the C-CH-O group, the asymmetric stretching of C-O-C and C-C bond stretching. The intense peak located at 1748.66cm -1 corresponds to the carbonyl radical and is characteristic of the esters. The band at 2859.2cm is due to the symetric *stretching of* CH(-CH-) and at 2929.92cm that was correspondent to the assimetric stretch of CH(-CH-) saturated bonds that are abundant in vegetable oil (16).

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3.1.1. FT-IR Spectra of Neem Oil Biodiesel

To confirm the conversion of the fatty acid functional group into an ester, FT-IR analysis was conducted on the biodiesel produced.

The FT-IR spectra of the neem biodiesel have confirmed the conversion of the triglycerides to mono alkyl esters (biodiesel). The two intense peaks at 2928.89 and 2862.25cm^{-1} corresponding to a CH- strectch I found in both the oil and the biodiesel. However, the ester functional group seen in the oil was converted into methyl ester functional group in the biodiesel. This functional group transformation has confirmed the formation of biodiesel, as the major functional group is the methyl ester.

3.2. The Effects of Temperature on the Biodiesel Yield

Reaction temperature is another important factor that affects the yield of biodiesel. For example higher reaction temperature increases the reaction rate and shortened the reaction time due to the reduction in viscosity of oils. However, it found that increase in reaction temperature beyond the optimal level leads to decrease of biodiesel yield, because higher reaction temperature accelerates the saponification of triglycerides. Usually the transesterification reaction temperature should be below the boiling point of alcohol in order to prevent the alcohol evaporation. The range of optimal reaction temperature may vary from 50°c to 60°c depends upon the oils or fats used (17) From Table 3.1 and Figure 3.3, the reaction temperature had significant effect on the biodiesel yield. Moderate yield of 73-79% was observed for lower temperatues of 30-40^{0}C, despite such low temperature, the yeild obtained were due to the very high catalytic activity of metal methoxide catalyst systems, in addition to the reduced FFA content (achieved via acid pretreatment of oil). Optimal reaction yeild was observed at higher temperatures of 50-60^{0}C, respectively.

Table 3.1: Effect of reaction temperature on biodiesel yield (oil to alcohol ratio 1:3, tempt. 60^{0}C, catalyst conc, 1%w/w oil and stiring intensity of 350rpm

Reaction temeperature (^{0}C)	**Percentage yield of biodiesel (%)**
30	73
40	79
50	91
60	94
70	67

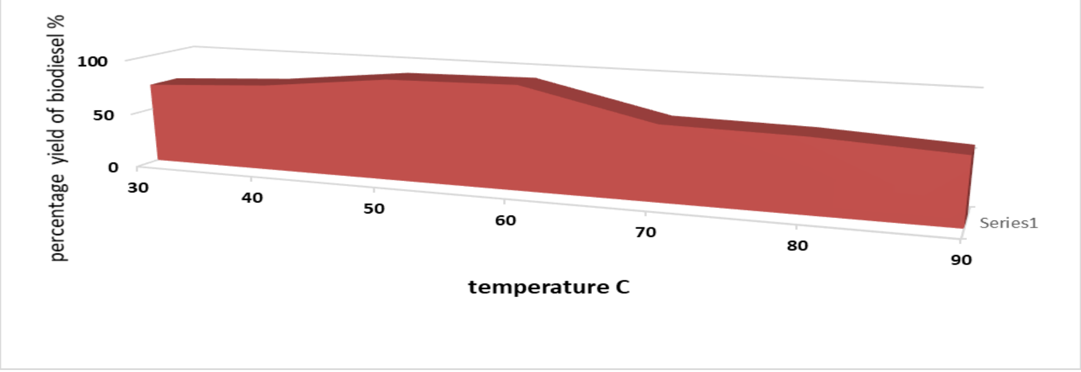

Figure 3.3: Effect of Temperature on Biodiesel Yield.

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Table 3.2: kinematic viscosity of Crude neem oil

Temperature(^{0}C)	**Mean time (sec)**	**Kinematic Viscosity(mm^2/s)**
30	1.946	5.48
35	1.666	4.69
40	1.432	4.03
45	**1.209**	**3.41**
50	**1.037**	**2.92**

3.3. Properties of Neen Seed Oil

3.3.1. Kinematic Viscosity

A gradual decrease in viscosity was observed in the methyl esters with temperature. The result agrees with theoretical claims which stated that viscosity decreases with an increase in temperature. Further, lower viscosity values were recorded for *neem* oil methyl esters. It was observed that the viscosity (5.7mm^2/s at 40^{0}C) of *neem oil* methylester generated was within the limit (2-6mm^2/s) specified by the American Society for Testing and Materials Standards (2003).

3.3.2. Density

The density of diesel oil is important because it gives an indication of the delay between the injection and combustion of the fuel in a diesel engine (ignition quality) and the energy per unit mass (18). The density of neem biodiesel at ambient temperature (25^{0}C) was found to be 0.87g /ml, which is close to the density of diesel (0.83g/ml). This showed the potential use of of neem seed oil biodiesel as an alternative fuel (See Table 3.3).

Table 3.3: Fuel properties of neem biodiesel (NBD)

Properties	**ASTM D6771- 02**	**NBD**
Viscosity at 40^{o}C (mm^2/s)	1.9-6	5.73
Specific gravity at 15^{o}C	0.95max	0.89
Pour point(^{o}C)	-35 to -16	-9
Cloud point		0
Flash point (^{o}C)	130min	149.6
Density (g/cm^3)	-	0.87
Heat of combustion mj/kg		47.12

3.3.3. Flash Point

Flash point is one of the most important characteristics of any fuel. Flash point is the temperature that indicates the overall flammability hazards in the presence of air; higher flash points make for safe handling and storage of biodiesel (19). The Flash Point of the neem oil biodiesel produced was 149.6^{0}C which above the ASTM D6751 minimum standards for biodiesel fuel of about 130^{0}C

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(Table 3.3). This makes the biodiesel sample safe for keeping and use (20). Fuels with lower flash point which tend to ignite at lower temperatures are highly dangerous if not stored and used properly. Most non-edible based seeds oils flash point are higher than fossil diesel (21).

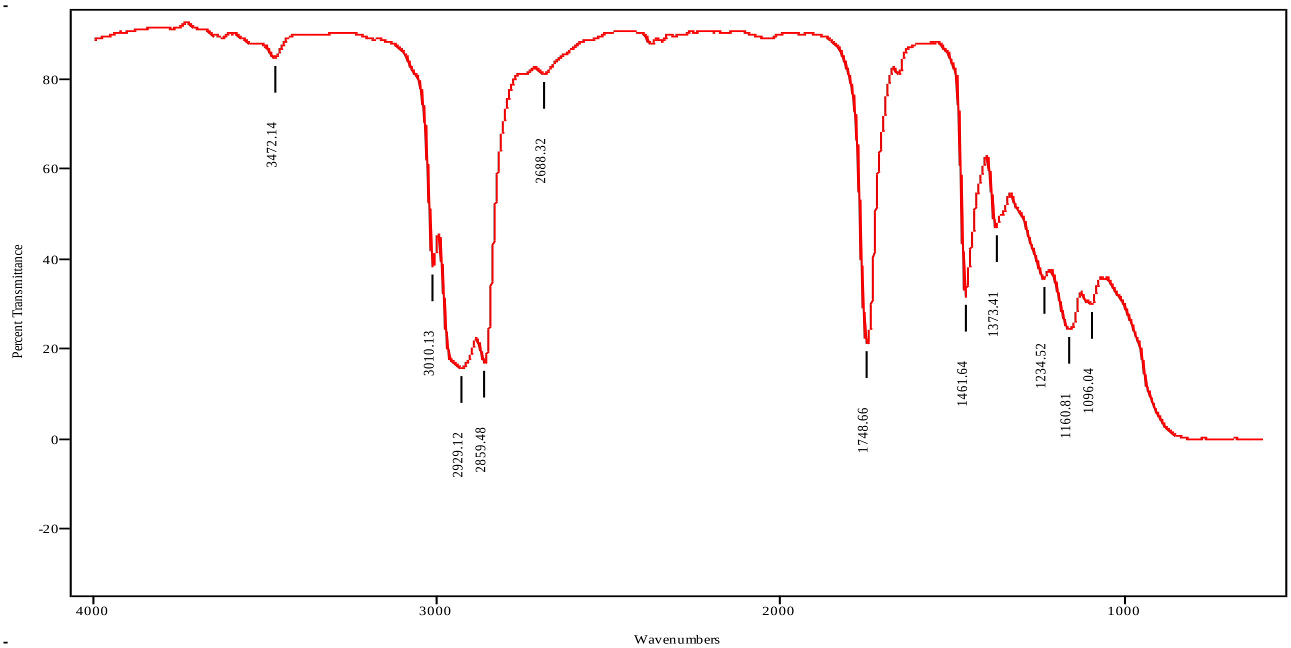

Figure 3.1: IR spectra of crude neem oil

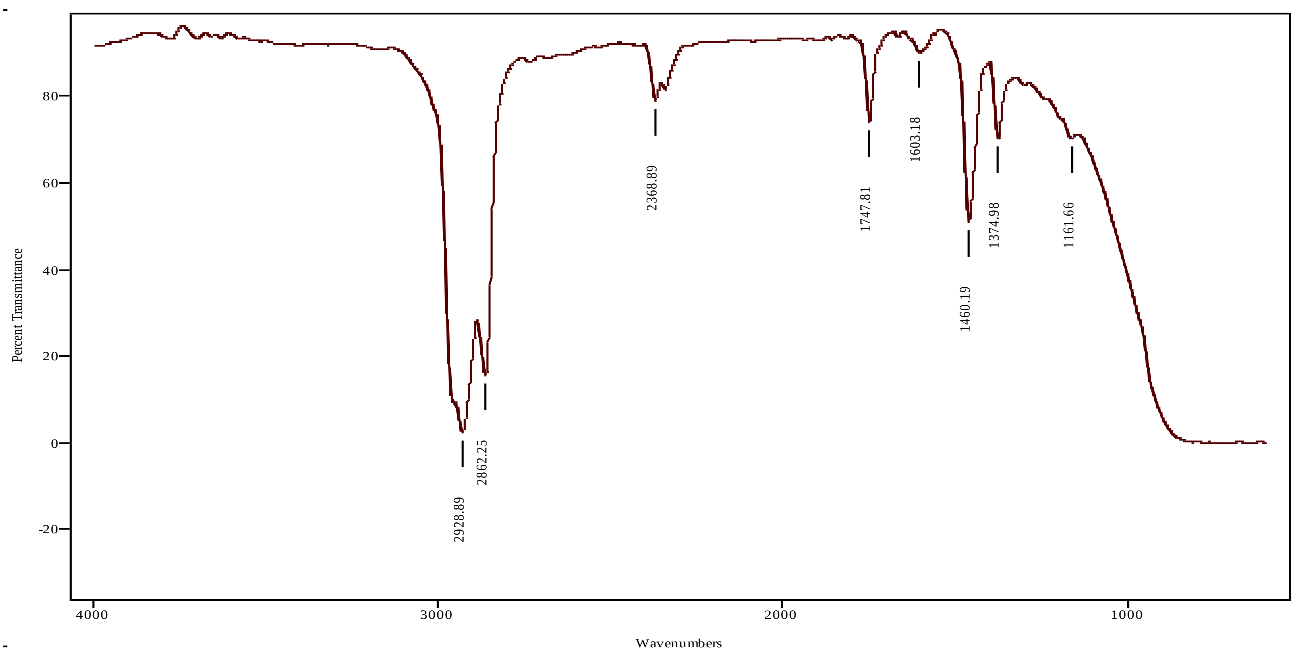

Figure 3.2: *FT-IR spectra of Neem oil biodiesel*

3.3.4. Pour and Cloud Point

The pour point is the lowest temperature at which the oil/fuel sample can flow. This property is related to the use of biodiesel in colder region (22). Neem oil biodiesel has a pour point of -9^{0}C and a cloud point of 0^0C. These values clearly indicate that the use of neem oil methyl esters in colder regions is limited. However, these values are also indicative of the high potential of this fuel as biodiesel fuel particularly in Northern Nigeria where temperature is always above 20^oC, a temperature at which the oil is fluid.

3.3.5. Heat of Combustion

The Heat Value of the biodiesel produced is 47.12MJ (Table 3.3). The heat of combustion of a fuel also known as heat of combustion is amount of energy a fixed quantity of the fuel releases when burnt with oxygen under specific conditions. The energy a fuel releases when it is burnt is used to drive the piston and eventually move the wheels of a vehicle. This means that the heat value of a fuel determines how far a vehicle containing a fixed amount of fuel can go. In order words, a fixed volume of a fuel with a high heat of combustion will travel farther than the same volume of another fuel with a low heat of combustion (3).

4. Conclusions and Recommendations

Biodiesel is fatty acid mono alkly ester. This research was carried out at temperature ranging from 30 – 70^0C and at a temperature of 60^0C; a good yield of 94% was obtained. The properties investigated present good values which were within the ASTM standard.

4.1. Conclusion

Biodiesel was produced from neem seed oil through a two-step process of esterification and transesterification reactions. The transesterification was carried out with CH_3ONa catalyst with ethanol as the alcohol. The reaction temperature was varied between 30, 40, 50, 60, and 70^0C, while all other process parameters were kept constant. From the results obtained, the results obtained showed a significant change in biodiesel yield (73-79%) from 30-50^oC temperatures. At a temperature of 60^oC, a good yield of 94% was obtained which was below the boiling point of the alcohol used. At 70^oC biodiesel yield of 67% was obtained; this indicates a drop in biodiesel yield.

The flash point of 149.6^0C indicated that the biodiesel is within the specification of ASTM. Also, the high value of flash point indicated that the oil is safe for handling as it exceeds the minimun ASTM requirement (130min). It is worthy to mention that other properties investigated also presented good values which are within ASTM D6751. The formation of biodiesel was confirmed by FT-IR analysis. The conversion of the ester functional group into methyl esters in biodiesel verified the success of the reaction.

4.2. Recommendations

1) Reasonable amount of neem seeds are present in this community but not utilized for any significant economic gains. Such seeds should be processed into biodiesel.
2) More research should be done on the potential of neem seed oil to serve as a viable substitute for fossil diesel.

3) Public sensitization should be done to educate the populace about the potentials of some of the seed or feedstock in their closest environment to serve as raw materials for biodiesel production.
4) The Government of Nigeria should work towards the industrial production of biodiesel for commercialization. This would help boost the economy and creat jobs.

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