Production and Characterization of Biodiesel-Ethanol-Diesel Blend as Fuel in Compression-Ignition Engine

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ABSTRACT

Biodiesel was produced from Neem oil, which was extracted from Neem seed (Azadirachta indica) using Soxhlet extraction by transesterification method. Ethanol was produced from saw dust of Masonia (Masonia Altissama) wood by means of simultaneous saccharification and fermentation process. 7.25 liters of oil was recovered from 13.5 kg of crushed neem seeds. Using a DMA-35 meter, the sugar produced from the saw dust was measured to be 923.2 g and 157.9 ml of ethanol was distilled at 78°C. The oil, the biodiesel and the blends properties, such as acid value (AV), saponification value (SP), ester value (EV), iodine value (IV), free fatty acid (FFA), peroxide value (PV) and cetane number (CN), were investigated. Total bacterial count and identification of colony growth were conducted and the result shows that Staphylococcus aureus identified with the maximum growth rate of 7 days. The ethanol produced was blended with diesel in different proportions and 10% biodiesel was used as emulsifier to prevent phase separation of the ethanol and diesel. The fuel properties of the biodiesel-ethanol-diesel (BED) blends were also experimentally investigated. The properties determined were relative density, cloud point, pour point, flash point, viscosity and the calorific value. The experimental results of all the blends were compared with standard values to know the suitability of using BED blend in compression ignition engine. The results show that both the relative density and viscosity of the blends decreased as the ethanol content in the blends was increased.

Keywords: Biodiesel, Transesterification, Ethanol, Fermentation, Staphylococcus Aureus

I. INTRODUCTION

Energy is one of the most important resources to mankind for his sustainable development. Today, the energy crisis becomes one of the global issues confronting us. The decline of available oil reserves and more stringent environmental regulations have motivated the global interest in renewable energy sources. Yusuf et al. \cite{1}, reported that biodiesel is considered as an attractive alternative fuel to replace Diesel fuels. Biodiesel consists of a mixture of fatty acid alkyl esters (FAAE) that can be obtained from vegetable oils or animal fats, mainly by transesterification reactions. Fuels are of great importance because they can be burned to produce significant amounts of energy. Many aspects of everyday life rely on energy, in particular the transport of goods and people; and the main energy resources come from fossil fuels, such as petrol oil, coal and natural gas.

Fossil fuel contributes 80% of the world’s energy needs and most industries use diesel machines for the production process \cite{2}. In the transportation sector,
private vehicles, buses, trucks, and ships also consume significant amounts of diesel and gasoline. This situation leads to a strong dependence of everyday life on fossil fuels. However, the growth of the population is not covered by domestic crude oil production [3, 4]. Fossil oils are fuels which come from thermal decomposed marine life and microorganisms. Fossil fuel formation requires millions of years. Thus, fossil oils belong to non-renewable energy sources. Based on current technology, it is estimated that the fossil fuel resources will be consumed in only 65 more years. In addition, the emission produced by the combustion of fossil fuels also contributes to the air pollution and global warming [5, 6]. Hence, renewable and clean alternative fuels have received increasing attention for current and future utilization. Apostolakou et al. [7] reported that biodiesel as one of the promising alternatives to fossil fuel for diesel engines, has become increasingly important due to environmental consequences of using petroleum fuels in diesel engines and the decreasing petroleum resources. Biodiesel can be produced by chemically combining any natural oil or fat with an alcohol such as methanol or ethanol. Methanol has been the most commonly used alcohol in the commercial production of biodiesel [8]. Several researches on biodiesel have shown that fuel made from vegetable oil can be used properly in diesel engines. Hayyan et al. [9] found that the energy density of biodiesel is quite close to regular diesel.

Moreover, the use of ethanol blended with diesel was a subject of research in the 1980s and it was shown that ethanol–diesel blends were technically acceptable for existing diesel engines. The relatively high cost of ethanol production at that time meant that the fuel could only be considered in cases of fuel shortages. The commitment made by the USA government to increase bioenergy three-fold in 10 years has added impetus to the search for viable biofuels (U.S. DE, 1998). The European Union (EU) has also adopted a proposal for a directive on the promotion of the use of biofuels with measures ensuring that biofuels account for at least 2% of the market for gasoline and diesel sold as transport fuel by the end of 2005, and rose to a minimum of 5.75% by the end of 2010 [10].

II. MATERIALS AND METHODS

2.1 Materials
2.1.1 Neem seed
Neem is an evergreen and deciduous tree in dry areas and it has straight trunk and long spreading branches forming a broad round crown and hence grown as Avenue tree. Its bark is moderately thick, then height 40-60 feet, the girth is 35 - 74 inches and the tree matures in 10 to 15 years. All parts of the tree are bitter and medicinal, has religious significance. Fruits bearing in 4 - 5 years continues for about 100 years. A spacing of 8.0 x 8.0 m is recommended to establish 150 trees per hectare.

Neem seeds have considerable economic significance due to a variety of commercial usages and the quality of seed determines the commercial value. If one tonne of neem seed is processed, it gives 1.5 kg of Azadirchtin 200kg of neem oil and 780kg of neem cake can be obtained [11]. Honda and Kaul [11] reported that on an average, neem kernels contain between 2 and 3mg of Azadirachtin Per gram of kernel. Neem Kernels collected from different Agro eco zones, when analyzed for minerals were found to contain: calcium 0.2 – 0.4%, magnesium 0.29 – 0.46% and phosphorus content 0.24 – 0.38% and oil is around 40%.

2.1.2 Sawdust of masonia wood
Renewable energy sources, such as lignocellulosic biomass, are environmentally friendly because they emit less pollution without contributing net carbon dioxide to the atmosphere unlike fossil fuels. Large amounts of lignocellulosic wastes are continuously generated from the production of waste materials in an undeniable part of human society. Lignocelluloses may be grouped into different categories such as wood residues (including sawdust and paper mill discards), grasses, waste paper, agricultural residues
(including straw, stover, peelings, cobs, stalks, nutshell, non-food seeds, bagasse, domestic wastes (lignocelluloses garbage and sewage), food industry residues, municipal solid wastes and the like [12]. These wastes or residues can be considered as potential energy sources.

It is estimated that the total area of forest reserves in Nigeria is 10 million ha (25 million acres) which is about 10% of the total land area of the country [1]. Sawdust abounds in great quantities in Nigeria. It is found in many sawmill sites, waste dumps, and in burnt forms that pollute the land and the atmosphere. Sawdust can be obtained from sawn wood and probably other wood wastes. Sawdust can be wastes/residue from either hardwood or softwood or the mixture of both. Softwood and hardwood vary in the percentages of cellulose, hemicellulose and lignin (Table 1). The cellulose and the hemicellulose fractions are the polysaccharide complex in lignocellulosic materials. Cellulose and hemicellulose are not directly available for bioconversion because of their intimate association with lignin [13, 14, 15].

Table 1. Weight Percent of Cellulose, Hemicelluloses, and Lignin in Wood Biomass

<table>
<thead>
<tr>
<th></th>
<th>% Cellulose</th>
<th>% Hemicellulose</th>
<th>% Lignin</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hardwood</td>
<td>40 – 55</td>
<td>24 – 40</td>
<td>18 – 25</td>
</tr>
</tbody>
</table>

Source: Bailey and Ollis (1986)

Among lignocellulosic biomass, sawdust waste from the Masonia tree (Masonia Altissama) can be a useful feedstock to economically produce environmentally friendly biofuels. The Masonia tree is typically a savannah woodland tree species. The Masonia is a big tree, 10 - 15m tall, which can reach sometimes 25m and it is a deciduous tree. The trunk of Masonia tree makes excellent charcoal. It is a favored source of wood fuel. Its natural habitat stretches over Africa, south of the savannah, from the eastern part of Senegal to the north of Uganda. This stretch covers an area of over 5,000 km long and 400 – 750 km wide. The West African subspecies Masonia Altissama is found in the eastern end of the range of the distribution of the species and indigenous to southern Nigeria and south-western Ethiopia [13].

2.2 Methods

2.2.1 Samples collection

The Neem seeds were collected at the streets of Azare town, Bauchi, Nigeria, which was used as the major raw material for the production of the Neem oil. The sawdust was obtained from wood market of Muda Lawal, Bauchi, Nigeria, which is the major raw material for the production of the ethanol. The material was collected in sacks and transported to the Chemistry Laboratory of Abubakar Tafawa Balewa University, Bauchi, where the production was carried out.

2.2.2 Soxhlet extraction method

A Soxhlet extractor is a piece of laboratory apparatus invented in 1879 by Franz von Soxhlet. It was originally designed for the extraction of a lipid from a solid material. However, a Soxhlet extractor is not limited to the extraction of lipids. Typically, a Soxhlet extraction is only required where the desired compound has a limited solubility in a solvent, and the impurity is insoluble in that solvent. If the desired compound has a significant solubility in a solvent, then a simple filtration can be used to separate the compound from the insoluble substance [16].

A mass of 50g of crushed neem seeds were packed inside a thimble made from thick filter paper, which was loaded into the main chamber of the Soxhlet extractor. The Soxhlet extractor was placed onto a flask containing hexane (extraction solvent). The Soxhlet was then equipped with a condenser (Plate I). The solvent was heated to reflux. The solvent vapour travels up a distillation arm, and floods into the chamber housing the crushed neem seeds. The chamber containing the solid material slowly filled with warm solvent. Some of the desired compound was then dissolved in the warm solvent.
When the Soxhlet chamber was almost full, the chamber was automatically emptied by a siphon side arm, with the solvent running back down to the distillation flask. This cycle was allowed to repeat many times, at an interval of 8 hours for 5 days. During each cycle, a portion of the non-volatile compound dissolved in the solvent. After many cycles, the desired oil was concentrated in the distillation flask. After extraction the solvent was removed by means of a rotary evaporator, yielding the extracted neem oil. The non-soluble portion of the extracted solid remained in the thimble, and was later discarded. 7.25 liters was recovered from 13.5kg of crushed neem seeds.

The sugar concentrations in brix against time in hours are presented as shown in Figure 2.

3.1 Physico-chemical Analysis of the Blends
3.1.1 Percentage yield of ethanol
According to [17], 52.2g of sugar is present in 100g of hardwood sawdust. Therefore, in 2500g of hardwood there would be 1305g of sugar.
Thus, the sugar yield is:

\[
\text{Sugar Yield} = \frac{\text{Actual value}}{\text{Theoretical value}} \times 100\%
\]

\[
\text{Sugar Yield} = \frac{923.2}{1305} \times 100\% = 70.74\%
\]

3.1.2 Acid value
The acid value for the neem oil and biodiesel of the neem oil were found to be 1.866mgKOH/g and 0.7mgKOH/g of oil respectively. The values are within the limits specified by ASTM D664 and compared with the ones reported by [17], [18] and [19].

3.1.3 Saponification value
The results of the saponification values of the oil, biodiesel and the blends are presented in Table 2. The values obtained were compared with standard values reported by [20].

3.1.4 Ester value
The ester value was calculated by subtracting the acid value of the neem oil from the saponification value of the oil. The value was found to be 191.33 mg and the percentage of glycerol was computed to be 10.47%.

3.1.5 Iodine value
Iodine values for the oil, the biodiesel and the blends were found to be 116.0 gl/100g, 99.6 gl/100g, 79.5 gl/100g, 77.4 gl/100g, 75.0 gl/100g, 72.0 gl/100g, 71.5 gl/100g, 70.3 gl/100g and 68.8 gl/100g respectively as
presented in Table 2. The values are within the limit set by ASTM D 1959 (ISO 3961) standard.

**Table 2. Saponification Value, Cetane Number and Iodine Value of Oil, Biodiesel and Blends**

<table>
<thead>
<tr>
<th>Sample</th>
<th>Obtained SV (mg)</th>
<th>Standard value of SV (mg)</th>
<th>Iodine value (IV)</th>
<th>Cetane number (CN)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Neem Oil</td>
<td>297.2</td>
<td>200-305</td>
<td>116.0</td>
<td>56.03</td>
</tr>
<tr>
<td>Biodiesel</td>
<td>291.8</td>
<td>200-305</td>
<td>99.6</td>
<td>56.56</td>
</tr>
<tr>
<td>BED5</td>
<td>291.7</td>
<td>200-305</td>
<td>79.5</td>
<td>56.23</td>
</tr>
<tr>
<td>BED10</td>
<td>282.5</td>
<td>200-305</td>
<td>77.4</td>
<td>56.89</td>
</tr>
<tr>
<td>BED15</td>
<td>280.1</td>
<td>200-305</td>
<td>75.0</td>
<td>57.04</td>
</tr>
<tr>
<td>BED20</td>
<td>270.8</td>
<td>200-305</td>
<td>72.0</td>
<td>57.74</td>
</tr>
<tr>
<td>BED25</td>
<td>260.9</td>
<td>200-305</td>
<td>71.5</td>
<td>58.59</td>
</tr>
<tr>
<td>BED30</td>
<td>253.4</td>
<td>200-305</td>
<td>70.3</td>
<td>59.27</td>
</tr>
<tr>
<td>BED35</td>
<td>252.3</td>
<td>200-305</td>
<td>68.8</td>
<td>59.35</td>
</tr>
</tbody>
</table>

3.1.6 Free fatty acid

To see the relationship between AV and %FFA, the following equations were solved for common values:

\[
\frac{AV}{56.1} = \frac{(v - b) \times N}{W} \quad \text{And} \quad \frac{\%FFA}{28.2} = \frac{(v - b) \times N}{W}
\]

The percentage of free fatty acid is usually calculated in terms of oleic acid and 1000g of sample contains 282 g of oleic acid [20].

Now from the two equations above, the free fatty acid (FFA) was found as follows:

\[
\frac{AV}{56.1} = \frac{\%FFA}{28.2} \quad \text{Or} \quad AV = 1.99 \times \%FFA
\]

\[
\%FFA = \frac{AV}{1.99} = AV \times 0.503
\]

Therefore,

The %FFA for the oil and the biodiesel were found to be 0.94 and 0.35, respectively.

3.1.7 Cetane number

The cetane number of the biodiesel was found to be 56.56. Also, the values of the cetane number for the various blends were found as presented in Table 2. The standard values of cetane number of biodiesel as specified by the ASTM are 48-69. It was observed that, with increase in ethanol blends, there was a corresponding increase in cetane number from BED10 through BED15 to BED35. Therefore, the knocking tendency of the engine decreases with an increase in the percentage of ethanol in the blend. However, at 5% ethanol in the blend, there was no any change in cetane number; it was same 56.23 as the diesel cetane number, this is because, there is no any significant change with 5% blend as compared to diesel as it was also observed with specific gravity and calorific value.

3.1.8 Total bacterial count

The results of the daily total bacterial count are presented in Figure 3. The colony counts were taken for the period of 9 days in order to obtain maximum growth. It can be seen that for almost all the samples, colonies repeat themselves after day 7, which is an indication that the maximum growth rate was reached.

The results of the microbial count presented in Figure 3 gave a clear indication of degradable behavior of organisms present in the biodiesel. The number of colonies for all the samples increases with days and maintained constant count in the last three days of the examination. Increase of the colonies indicates life supporting behavior of the biodiesel. The control sample (nutrient agar) maintained zero growth throughout the days, while diesel sample gave a maximum of 22 counts. BED10 appears to have the maximum growth of 149 counts and the oil sample stopped at 40 counts. The results followed the trend of the results presented by [21], [22] and [23]. It should be noted that, studies have shown that biodiesel has a higher amount of microbial contamination, higher rate of microbially induced fuel degradation and higher rate of Microbially Induced Corrosion (MIC) of fuel system components compared to petroleum diesel [22, 23]. Therefore, majors should be taken to take care of the expected corrosion.
3.1.9 Relative density

Figure 4 shows the values of the relative densities of the blends at different temperatures, while Figure 5 shows the percentage differences in the relative densities of the blends with that of diesel.

3.1.10 Cloud point

The cloud points for the fuels are also presented in Figure 6. All the blends were found to have the same cloud point with that of diesel. The reason is that all the blends have diesel as a major component. Diesel was reported to have a cloud point of 50°C, while ethanol has a cloud point below 100°C [24]; therefore, the cloud point found for all the blends are the same with that of diesel.

3.1.11 Pour point

The presence of ethanol in the blends however, affected their pour point. Fuel blends BED5, BED10, BED15, BED20, BED25, BED30 and BED35 were found to have pour point of 4, -5, -10, -18, -30, -32 and -35°C, respectively (refer to Table 3). This low temperature obtained for all the blends is due to the fact that ethanol delayed the degree of coalescence of the blends despite the high degree of miscibility of ethanol and diesel.

3.1.12 Flash point

From Table 4, it can be observed that all the blends have a flash point lower (over 65% lower) than that of diesel. The temperatures obtained were all below the ambient temperature of about 280°C. Ethanol, which has a flash point below ambient temperature when blended with diesel that flashed at a temperature of 740°C, vaporized and supplied the vapor that was ignited by the test flame. The flash point gives the safe storage temperature for a fuel.
Table 3. Pour Point of Different Blends:

<table>
<thead>
<tr>
<th>Fuel blend sample</th>
<th>Pour point (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diesel</td>
<td>5</td>
</tr>
<tr>
<td>BED5</td>
<td>4</td>
</tr>
<tr>
<td>BED10</td>
<td>-5</td>
</tr>
<tr>
<td>BED15</td>
<td>-10</td>
</tr>
<tr>
<td>BED20</td>
<td>-18</td>
</tr>
<tr>
<td>BED25</td>
<td>-30</td>
</tr>
<tr>
<td>BED30</td>
<td>-32</td>
</tr>
<tr>
<td>BED35</td>
<td>-35</td>
</tr>
</tbody>
</table>

Table 4. Flash Point of Different Blends:

<table>
<thead>
<tr>
<th>Fuel blend sample</th>
<th>Flash point (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diesel</td>
<td>74</td>
</tr>
<tr>
<td>BED5</td>
<td>20</td>
</tr>
<tr>
<td>BED10</td>
<td>22</td>
</tr>
<tr>
<td>BED15</td>
<td>25</td>
</tr>
<tr>
<td>BED20</td>
<td>26</td>
</tr>
<tr>
<td>BED25</td>
<td>27</td>
</tr>
<tr>
<td>BED30</td>
<td>27</td>
</tr>
<tr>
<td>BED35</td>
<td>27</td>
</tr>
</tbody>
</table>

3.1.13 Viscosity

The measured viscosity of the blends decreased as the percentage of ethanol in the blends increased. The viscosity also decreased with increase in temperature. At 150°C, the viscosities of BED5, BED10, BED15, and BED20 blends were close to that of diesel (refer to Figure 7). The 20% blend was about 14% less viscous than diesel. The viscosities of the blends were however close to that of diesel when compared with other vegetable oils – diesel blends whose viscosities are usually very high [25, 26, 27, 28]. The reduction in the viscosity of the blends was mainly due to the presence of ethanol (with a very low viscosity) in the blends.

3.1.14 Calorific value

The calorific values for the fuels are presented in Table 5. The calorific values for BED5, BED10, BED15 and BED20 blends were 2, 3, 4 and 6% respectively less than that of diesel. This indicates that the ethanol-diesel blends have over 90% of the calorific value of diesel. The calorific values decreased as the percentage of ethanol in the blends increased. When compared to other vegetable oils as reported by [28] and [29], the calorific values of the tested fuels were quite high, which explains why ethanol-diesel blends have good potential for high heat release than other vegetable oils – diesel blends. The results of the current study on calorific values are similar to the ones earlier reported by [25].

![Figure 7. Viscosity of the Fuel Blend Samples at Different Temperatures](image)

Table 5: Calorific Value of the Fuels

<table>
<thead>
<tr>
<th>Fuel sample</th>
<th>Calorific Value (kJ/kg)</th>
<th>%difference compared to diesel</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diesel</td>
<td>45575.0</td>
<td>-</td>
</tr>
<tr>
<td>BED5</td>
<td>43711.4</td>
<td>4.089</td>
</tr>
<tr>
<td>BED10</td>
<td>43581.5</td>
<td>4.374</td>
</tr>
<tr>
<td>BED15</td>
<td>43477.9</td>
<td>4.601</td>
</tr>
<tr>
<td>BED20</td>
<td>43297.8</td>
<td>4.997</td>
</tr>
<tr>
<td>BED25</td>
<td>43221.0</td>
<td>5.165</td>
</tr>
<tr>
<td>BED30</td>
<td>43212.5</td>
<td>5.184</td>
</tr>
<tr>
<td>BED35</td>
<td>43163.7</td>
<td>5.291</td>
</tr>
</tbody>
</table>

IV. SUMMARY AND CONCLUSION

4.1 Summary

Biodiesel was produced from neem oil, which was extracted from neem seeds (Azadirachta indica), by
transesterification method and ethanol was produced from saw dust of Masonia (Masonia Altissima) by means of simultaneous saccharification and fermentation process, which can be described as a renewable energy source. The ethanol produced was blended with diesel in different proportion and 10% biodiesel was used as emulsifier to prevent phase separation of the blends. Physical and chemical properties (physico-chemical properties) of the oil, the biodiesel and the blends were determined, which included the percentage yield of oil and ethanol produced, acid value, saponification value, ester value, iodine value, free fatty acid, peroxide value, cetane number, relative density, cloud point, pour point, flash point, viscosity and the calorific value. Biodegradability analysis (total bacterial count) and identification of colony growth were also conducted. All the physico-chemical properties investigated were compared with diesel fuel.

4.2 Conclusion
From the experimental investigation the following conclusions may be drawn:

1) Biodiesel can be produced from neem oil in reasonable quantity.
2) Ethanol can also be produced from saw dust of Masonia wood in reasonable quantity.
3) Ethanol and diesel can be blended with biodiesel as emulsifier to serve as fuel with similar physical and chemical properties with diesel.
4) The acid value, saponification value, ester value, iodine value and free fatty acid of the neem oil and the biodiesel were found to be within the limits of ASTM standards.
5) The cetane number of the blends was found to be on increasing trends, which translates the possibility of the blends to reduce knocking tendency. Since, the knocking tendency of compression ignition engine decreases with an increase in the cetane number.
6) Relative densities at different temperature of all the blends are lower than that of diesel fuel.
7) All the blends were found to have the same cloud point with that of diesel. The reason is that all the blends have diesel as a major component.
8) Fuel blends BED5, BED10, BED15, BED20, BED25, BED30 and BED35 were found to have pour point of 4, -5, -10, -18, -30, -32 and -350C, respectively. This low temperature obtained for all the blends is due to the fact that ethanol delayed the degree of coalescence of the blends despite the high degree of miscibility of ethanol and diesel.
9) All the blends have flash points that are over 65% lower than that of diesel. The temperatures obtained were all below ambient temperature of about 280C. Ethanol, which has a flash point below ambient when blended with diesel that flashes at a temperature of 740C, vaporizes and supplies the vapor that easily ignites. Hence, reducing ignition delay of the fuel blends when tested in an engine.
10) The viscosity decreased as the percentage of ethanol in the blends increased. The viscosities also decreased with increase in temperature. At 150C, the viscosities of BED5, BED10, BED15, BED20, BED25 blends were close to that of diesel. The BED20, BED25 and BED30 blend is about 14% less viscous than diesel.
11) The calorific values for BED5, BED10, BED15, BED20, BED25 and BED30 blends were 2, 3, 4, 6, 7 and 9% respectively less than that of diesel. This indicates that the ethanol-diesel blends have over 90% of the calorific value of diesel.

V. REFERENCES

Conversion and Management 2009, 50, 2239-2249.


