Biodiesel Production using \textit{Helianthus annuus} (Sunflower) Seed Oil by Trans-
Esterification Method

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INTRODUCTION

The rising demand for energy amidst depleting petroleum reserves and fluctuating oil prices has stimulated the research into alternative forms of energy such as biofuels [1]. The interest in biofuels doesn’t lie solely in their ability to provide alternative sources of energy to petroleum and natural gas, but also their environmental safety, affordability, ready availability from vegetable and animal waste, ability to be blended with existing petroleum fuels, and amenability with existing internal combustion engines [1].

Particular advantages of biodiesel that makes it of interest to researchers include its ability to function safely in all conventional diesel engines while offering similar performance and engine lifetime as petroleum diesel. It is also non-toxic, non-flammable, reduces smoke, fumes and other tailpipe emissions. Biodiesel possesses superior properties to petroleum diesel, by having a better flashpoint, being biodegradable, low in Sulphur and aromatic compounds and is generally a cleaner fuel compared to petroleum diesel since it is oxygenated [2].

Suggested means of reducing the net cost associated with biodiesel production is the purification and commercialization of its by-product (glycerol), as well as the use of fats and oil wastes, form restaurants as triglyceride sources. However, the high amount of free fatty acids presents in these waste samples and the fact that free fatty acids can’t be esterified by alkaline alcoholysis is a limitation to the implementation of the latter approach [3].

The main methods for producing biodiesel include dilution, micro-emulsion, pyrolysis, and trans-esterification of vegetable oils. These methods mainly serve (among other functions) to reduce the viscosity of the fluid, making it more suitable as a fuel, compared to the raw vegetable oil [4]. Of these methods, trans-esterification has been the most utilized due to its ability to produce fuel (esters) with technical properties similar to that of petroleum diesel. The main factors known to affect the trans-esterification process are temperature and pressure of the reaction, choice of catalyst, duration of reaction, and the triglyceride alcohol mole ratio. Generally, the glyceride/alcohol mole ratio used is 6:1-30:1[2].
Trans-esterification involves the reaction of vegetable oils (e.g. sunflower oil) or fats with an alcohol in the presence of a catalyst to produce biodiesel and glycerol. The triglyceride is converted stepwise to diglyceride, monoglyceride and finally glycerol [5]. The use of fuels from bio-resources has been widely acknowledged as a panacea to the global twin-problem of the fuel crisis and environmental pollution informed by over-dependence on fossil fuels [6].

Biofuel can be broadly defined as any sort of fuel that is made from organic matter (Biomass). The most common biofuels are biodiesel and bio-alcohols, including bioethanol and biobutanol also called biogasoline [7]. The concept of biofuel is not new. Rudolph Diesel was the first to use a vegetable oil (peanut oil) in a diesel engine in 1911[8-9]. The use of biofuels in place of conventional fuels would slow the progression of global warming by reducing sulfur and carbon oxides and hydrocarbon emissions [10]. Because of economic benefits and more power output, biodiesel is often blended with diesel fuel in ratios of 2:5 or 20% [11]. The higher the ratio of biodiesel to diesel the lower the carbon dioxide emission [12, 13].

Biodiesel is a mixture of Fatty Acid Methyl Esters (FAMEs) which is produced from renewable sources [14]. However, fats and oils are often used interchangeably referring to the feedstock employed in biodiesel production. The raw materials used for the production of biodiesel can be crude, refined or waste such as frying oils/fats [15]. The feedstock can also be classified as plant-derived, animal-derived, microbial or waste materials [8]. According to [16] identified more than 300 oil-bearing plants/trees that can be utilized to make biodiesel.

The most popular plant-derived oils used for biodiesel production are canola, coconut, cottonseed, groundnut, jatropha, karanji, olive, palm, peanut, rapeseed, safflower, soybean, and sunflower oils [2,8,17]. The choice of feedstock depends on where the biodiesel is being produced and used which could meet norms of internationally accepted ASTM standards. Parameters such as saponification number, iodine value and cetane number of fatty acid methyl esters of the oil, also, play an important role in the selection of feedstock for biodiesel production [16].

Another important factor in biodiesel production is the fatty acid composition of the source of oil or fat. Oils containing higher levels of saturated fatty acids than unsaturated fatty acids (have one or more double bonds) may solidify and clog the fuel lines during the winter condition [18]. The transesterification process involves the reaction of triglyceride (oil or fat) with an alcohol in the presence of a catalyst, usually a strong base such as sodium or potassium hydroxide. The alcohol reacts with the fatty acids forming the mono-alkyl ester called biodiesel and glycerol [19] Methanol or ethanol are commonly used alcohols in the process. Methanol produces methyl esters, commonly referred to as Fatty Acid Methyl Ester-FAME, and ethanol produces ethyl esters, commonly referred to as Fatty Acid Ethyl Ester-FREE [20].

Sunflower “seeds” are a fruit, the inedible wall (husk) surrounding the seed that is in the kernel. The great importance of sunflower lies in the excellent quality of the edible oil extracted from its seeds. It is highly regarded from the point of view of nutritional quality, taste and flavour. Moreover, after oil extraction, the remaining cake is used as a livestock feed. It must be noted that sunflower oil has a very low content of linoleic acid, and therefore it may be stored for long periods. Sunflower adapts well to adverse environmental conditions and does not require specialized agricultural equipment and can be used for crop rotation with soybean and corn. The oil yield of current hybrids is in the range of 48–52 % [21].

**MATERIALS AND METHODS**

The substrate used is Sunflower seed oil. The Substrate was collected at a local market in Yola North, Adamawa State, Nigeria.

**Equipment**

Industrial sieve (of 70 µm pore), Beaker, Retort stand, Dropper, Thermometer, Hot plate, 250ml bottom flask, weighing pan, separating funnel, 50ml and 250ml conical flask, Magnetic stirrer, Syringe, Burette, Hand gloves, pH meter, Spatula, weighing balance.

**Chemicals/reagents**

Methanol, Distilled water, Sodium hydroxide pellets, Potassium hydroxide pellets, Phenolphthalein and Tetraoxosulphate (IV) acid.

**Removal of particulates**

Sunflower seed oil may contain solid particles such as sand, sticks, and debris. Larger particles were removed through sedimentation by allowing the particles to settle at the bottom of the container. Small particles present in the oil were removed through filtration, using the industrial sieve of 70 µm pore diameter.

**Removal of water from the oil**

The Sunflower seed oil was heated to 120°C for 20 minutes using hotplate which allowed vaporization of water in the oil into the atmosphere.

**The trans-esterification processes**

**Test batch**

Two hundred (200) gram (g) of Sunflower seed oil and 40 gram (g) of methanol (i.e. 20% by volume of oil) for each of the oil was utilized in the test batch production. 200 gram (g) of Sunflower oil was pre-heated to a steady temperature of 65°C using a magnetic heater/stirrer. With the aid of the measuring cylinder, 40gram (g) of methanol was measured and poured into separate beakers. 0.8g of NaOH pellet was measured using the weighing balance and added to the methanol in each of the beakers. The content of the beakers was then stirred vigorously using magnetic stirrer until the NaOH was completely dissolved in the methanol. The mixture formed is called sodium Methoxide. The Methoxide was poured into the beaker containing the preheated oil. The content of the beaker was then be stirred with the magnetic stirrer at a steady speed and temperature of 55°C. Then heating and stirring were stopped after 2 hours and the product was poured into a separating funnel to be mounted on a clamp stand. The mixture was allowed to settle down overnight. The separating funnel was opened at the bottom to allow the glycerin at the bottom to run off after which the biodiesel was collected in beaker for storage.

**Final production batch**

The test batch just described above was performed to have a firsthand experience of the reaction methodology and its attendant products. It serves as a means of ensuring that the end products can be successfully obtained from the reactants. Based on the successful results obtained from the test batch reaction, the final production batch was carried out using the same volume and/or mass of reactants and the separation of the products was carried out as already described for the test batch production. The
procedure will be replicated three times and average biodiesel yield, as well as glycerol yield, was measured.

**Settling and Washing**

The mixture was poured from the beaker into a separating funnel bottle for settling and the lid was screwed on tightly. The reaction mixture was allowed to stand overnight to allow phase separation to occur by gravity settling. The Sunflower biodiesel at the top was carefully decanted leaving the glycerol at the base. Thorough washing of the biodiesel was carried using distilled water.

**RESULTS AND DISCUSSION**

The process of trans-esterification of Sunflower Seed Oil (SSO) gave a yield of 176.6g biodiesel and 16.17g glycerol, 47.98g of the total reacting masses could not be accounted for. The results stated are averages of three different experimental runs. Detailed results for each of the experimental runs are as presented in Table 1 as was recorded in the course of carrying out the laboratory work. Table 2 contains the fuel properties of the biodiesel produced and the biodiesel standards mandated by the American Society for Testing and Material (ASTM) standard. The properties such as density, kinematic viscosity, dynamic viscosity, water content, acid value, flash point and pour point were determined. The percentage yield of biodiesel from Sunflower oil was 88.31%. This value was higher when compared to that of rapeseed oil which is 58.10% [22].

The density of the Sunflower oil biodiesel was 0.79g/cm³, which is within the range of ASTM value for diesel oil (0.75 – 0.84). Kinematic viscosity of the Sunflower oil is 3.59 mm²/s which was found within the limits of American Society for Testing and Material (ASTM) (1.9 – 6.0 mm²/s) for biodiesel fuel standard. The dynamic viscosity of Sunflower oil was found to be 6.58 x 10⁻³ kgm⁻¹s⁻¹. The water content of Sunflower was found close to ASTM value but varies with 0.01% mass. The acid value for sunflower oil was found to be 0.36 mg/KOH/g which is a little bit higher compared to American Society for Testing and Material (ASTM) (0.1) for biodiesel fuel standard. The flashpoint for Sunflower is 159 which were higher than the ASTM value (130min). Pour point of the Sunflower oil is -19 °C which was found to be lower when compared to that of American Society for Testing and Material (ASTM) (-15-10) for biodiesel fuel standard.

It is important to note that the benefits accrued to the use of Crude Sunflower oil (CSFO) for biodiesel production are far greater than the disadvantage traceable to their usage as feedstocks. The assessment of biodiesel produced highlights the potential of biodiesel as a direct substitute for synthetic fuel for diesel engines.

**REFERENCES**


**Table 1. Sunflower oil trans-esterification experiment.**

<table>
<thead>
<tr>
<th>Experimental conditions</th>
<th>1st run</th>
<th>2nd run</th>
<th>3rd run</th>
<th>Average</th>
</tr>
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<tbody>
<tr>
<td>Reaction temperature (°C)</td>
<td>55</td>
<td>55</td>
<td>55</td>
<td>55</td>
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<tr>
<td>Reaction time (MIN)</td>
<td>120</td>
<td>120</td>
<td>120</td>
<td>120</td>
</tr>
<tr>
<td>Oil quantity (G)</td>
<td>200</td>
<td>200</td>
<td>200</td>
<td>200</td>
</tr>
<tr>
<td>Methanol quantity (G)</td>
<td>40</td>
<td>40</td>
<td>40</td>
<td>40</td>
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<tr>
<td>NAOH concentration (G)</td>
<td>0.8</td>
<td>0.8</td>
<td>0.8</td>
<td>0.8</td>
</tr>
<tr>
<td>biodiesel obtained (G)</td>
<td>178.45</td>
<td>179.20</td>
<td>172.25</td>
<td>176.67</td>
</tr>
<tr>
<td>Glycerol obtained (G)</td>
<td>16.26</td>
<td>16.10</td>
<td>16.19</td>
<td>16.17</td>
</tr>
<tr>
<td>Losses (G)</td>
<td>46.1</td>
<td>45.5</td>
<td>52.36</td>
<td>47.98</td>
</tr>
<tr>
<td>Biodiesel yield (%)</td>
<td>89.22</td>
<td>89.60</td>
<td>86.12</td>
<td>88.31</td>
</tr>
</tbody>
</table>

**Table 2. Characterization of sunflower seed oil biodiesel.**

<table>
<thead>
<tr>
<th>Property</th>
<th>ASTM Standard</th>
<th>Sunflower Seed Oil Biodiesel</th>
<th>Unit</th>
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<tbody>
<tr>
<td>Density at 50°C</td>
<td>0.75±0.04</td>
<td>0.79</td>
<td>g/cm³</td>
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<tr>
<td>Kinematic Viscosity</td>
<td>1.9-6.0</td>
<td>3.59</td>
<td>mm²/s</td>
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<tr>
<td>Dynamic viscosity</td>
<td>-</td>
<td>6.58 x 10⁻³</td>
<td>kgm⁻¹s⁻¹</td>
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<tr>
<td>Water content</td>
<td>0.05 max</td>
<td>0.04 max</td>
<td>g/KOH/g</td>
</tr>
<tr>
<td>Acid value</td>
<td>0.1</td>
<td>0.36</td>
<td>mg KOH/g</td>
</tr>
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<td>Flash point</td>
<td>130min</td>
<td>159</td>
<td>°C</td>
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<tr>
<td>Pour point</td>
<td>-15 to 10</td>
<td>-19</td>
<td>°C</td>
</tr>
</tbody>
</table>

**CONCLUSION**

Biodiesel produced from Sunflower seed oil was characterized for its fuel properties using American Society for Testing and Material (ASTM) Standard method for biodiesel fuel quality assurance. The results obtained shows that the Sunflower seed oil could be considered as a viable raw material for biodiesel production. This is because its biodiesel meets the standard specification of the American Society for Testing and Material (ASTM). The trans-esterification reaction of sunflower oil with methanol gave higher yield 88.31% of biodiesel. Biodiesel produced from this study is environmentally-friendly and suitable as a direct substitute for synthetic fuel for diesel engines.


