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The Effects of Temperature and Catalyst Concentration on Transesterification of Oil Extracted from *Ceiba Pentandra* (Silk Cotton) Seed Oil

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ABSTRACT

The effect of temperature and catalyst concentration on transesterification of *Ceiba pentandra* (Silk cotton) seed oil was studied using two step, acid esterification and alkali-catalyzed transesterification process with varying catalyst concentrations (0.25, 0.50, 0.75, 1.0, 1.25, and 1.50 % w/v) and methanol/oil ratio of 3:1, 6:1, 9:1 and 12:1 at temperatures 50, 55, 60 and 65 °C for 1 hour. The results obtained showed optimal percentage oil yield of 92.9 % at 65 °C after 1 hour. The results also indicate that properties of the biodiesel: specific gravity ($0.88 \pm 0.01 \text{ g/cm}^3$), carbon residue ($0.02 \pm 0.01 \%$), acid value ($0.10 \pm 0.01 \text{ mgKOHg}^{-1}$), sulphur content ($0.02 \pm 0.03 \%$), flash point ($170 \pm 1.00 \text{ }^\circ\text{C}$), pour point ($25.00 \pm 0.50 \text{ }^\circ\text{C}$) and viscosity ($4.70 \pm 0.14 \text{ cSt}$) were within ranges of ASTM biodiesel standard specification. Maximum yield of biodiesel was obtained at 1.0 % catalyst concentration, hence it is recommended that 1.0 % of the catalyst be used for transesterification of *Ceiba pentandra* seed oil.

Keywords: *Ceiba pentandra*, biodiesel, transesterification, specific gravity, flash point

1.0 INTRODUCTION

One of the most important elements that affect world economy and politics is sustainability of petroleum resources, and it is also the main source of world energy supply. The demand for energy around the world is increasing rapidly due to large population excessive use of fuels, limited reservoir and instabilities in petrol supplying countries (Demirbas, 2008). It is in recognition to this challenge, that the Federal Government of Nigeria initiated different policies and programmes aimed at providing sufficient energy to its citizens. Example of this policy is the 2003 national energy policy. However, increasing concern on the persistent effect of pollution on the environment due to the wide spread combustion of fossil fuel, which has threaten wild and human live and impacted the environment negatively has cause a rethink on the need for renewable energy. Global warming, one of the world problems today is known to be caused by product of combustion which include carbon monoxide (CO), sulphur dioxide(SO₂) and Nitrogen oxide (NO_x). Hence the Nigerian government in 2015 came up with the Nigerian Renewable Energy and Energy Efficiency Policy

(NREEEP) which was to be implemented by the Energy Commission of Nigeria.

Researches on green fuel have a remarkable part in the world's technological advancement. According to Barwal and Sharma (2005), bioethanol, biogas and biodiesel are among the vast greener fuels that has gain wide applications in most of the developed world. Tranesterification of vegetable oil into biodiesel for instance, gained special consideration in Malaysia, Indonesia, United State of America and Europe. In the current competitive global economic, there is need for developing nation to strengthen effort towards diversifying energy sources by exploiting the endowed renewable resources. Nigeria as a developing country is enriched with vast oil bearing seeds capable of been profitably transesterified to biodiesel, but production and application of biodiesel is still at a low stage.

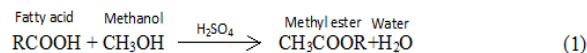
Biodiesel is the mono-alkyl esters of long chain fatty acids derived from renewable feedstocks, such as vegetable oil (edible and nonedible) through transesterification, for use in compression ignitions

(Mether *et al.*, 2007). The production of biodiesel is influenced by factors which include the type and concentration of catalyst used as well as reaction temperature. Action of catalyst and temperature depend on the chemical composition of the feedstock. This study looks at the impact of variable catalyst concentration and temperature on the oil of *Ceiba pentandra* seed grown in Nigeria.

2.0 MATERIALS AND METHODS

2.1 Sampling and Sample Treatment

The seeds of *Ceiba pentandra* (Silk cotton) were bought from Paskari market in Katsina State, Nigeria. The seeds were cleaned to remove dirt and other foreign materials such as ticks and sand before being dried. The seeds were then mechanically dehulled to remove the seed coat, milled into fine powder and oil was extracted from the sample seed using Soxhlet extractor using n-hexane as solvent.



2.3.2 Alkaline Transesterification

The esterified oil was transesterified to mono-esters of fatty acid using NaOH as catalyst. The 100 g of esterified oil sample was poured into conical flasks (500 cm³) and heated to 45 °C and NaOH (1g) was added to 100 cm³ methanol to form sodium methoxide, the mixture was poured into the oil in the reactor flask. The mixture was heated and stirred at constant speed to ensure uniform agitation for 1 h at 60 °C then the mixture was transferred into a separating funnel and allowed to stand for 6 h as it separates into layers of biodiesel and glycerol. The lower layer that contained impurities and glycerol was drained and methyl ester remains in the upper layer. The methyl esters were washed with warm distilled water to remove impurities such as soap, catalyst and glycerol. The excess methanol and water in the ester phase were removed by heating the methyl ester to 100 °C. After which the biodiesel was weighed.

Percentage oil yield of each sample was determined using equation 2:

$$\% \text{ Oil yield} = \frac{B-A}{W} \times 100 \quad (2)$$

where:

W is the weight of the sample in (g)

2.2 Physicochemical analysis of the *Ceiba Pentandra* Oil

The oil extracted from *Ceiba pentandra* seed was analyzed for its percentage oil yield, free fatty acid and acid value according to methods of AOAC (1998).

2.3 Production of Biodiesel

The production of biodiesel involved two steps: acid esterification and transesterification.

2.3.1 Acid Esterification

Given that the yield of biodiesel decreases considerably if the free fatty acid (FFA) value is greater than 2 % (Canakci and Van Gerpan, 2001) and the high free fatty acid (FFA) in the cotton seed oils, sulphuric acid (H₂SO₄) was used to reduce the FFA to less than 2 % before transesterification reaction:

B is the weight of the empty thimble + sample before extraction

A is the weight of the thimble + sample after extraction.

The optimal value of each parameter involved in the process was determined. Factors affecting the process such as methanol to oil molar ratio, catalyst (NaOH) concentration, reaction temperature and rate of agitation were studied. The variables affecting transesterification such as catalyst concentration (0.25, 0.5, 0.75, 1.0, 1.25, and 1.50 % w/v of oil), methanol/oil molar ratio (3:1, 6:1, 9:1 and 12:1), temperature (50, 55, 60 and 65 °C), rate of agitation (150, 225, 300 and 375 rpm) and reaction time were studied over a wide range of time (20, 30, 40, 50, 60, 65, 70, 80 minutes) to get higher conversion under optimal reaction condition

3.0 RESULTS

3.1 Effect of Catalyst Concentration

The effect of catalyst on Silk seed oil yield using NaOH as catalyst is presented in Figure 1. From Figure 1, the highest yield for the biodiesel fuel was observed at a catalyst concentration of 1.0 % with the yield of 92.90 % for silk seed oil at reaction time of 75 min. respectively.

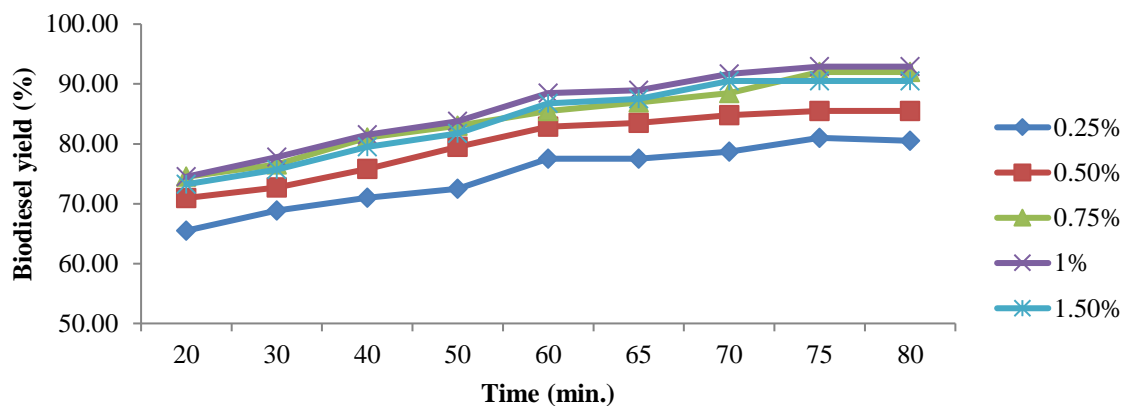


Figure 1: Effect of Catalyst Concentration on Yields of Biodiesel from Silk Cotton Seed Oil

3.2 Effect of Methanol/Oil Molar Ratio

The effect of varying methanol to oil molar ratio on biodiesel yield is presented in Figure 2. The highest

yield for the silk seed oil was at a molar ratio of 9:01 as shown in Figure 2

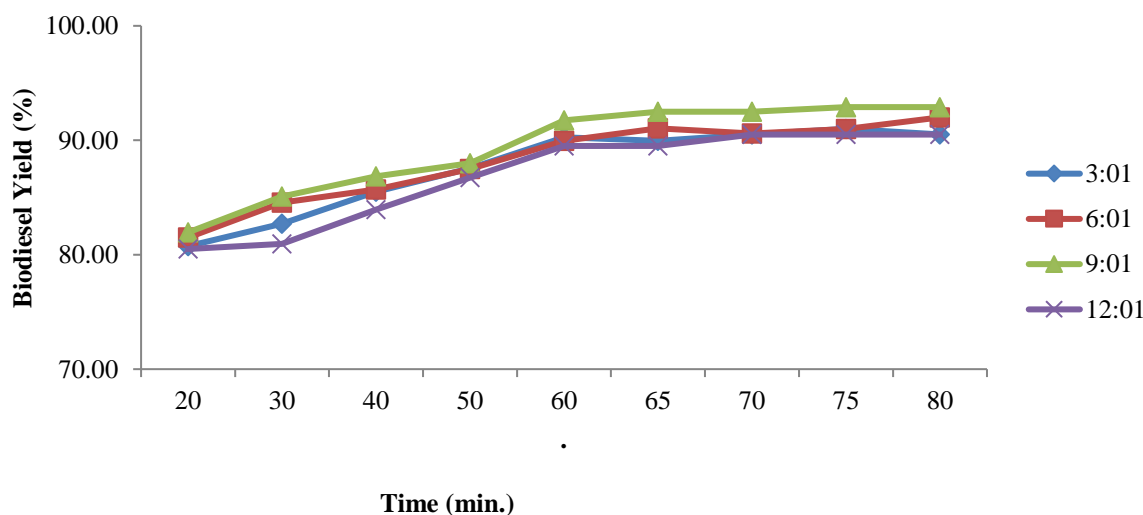


Figure 2: Effect of Molar Ratio on Biodiesel Yield from Silk Cotton Oil.

3.3 Effect of Temperature

Figure 3 present the result of the effect of temperature on biodiesel production. The optimal yield of biodiesel from the oils under investigation was achieved at a reaction temperature of 65 °C gave a yield of 92.90 % silk. The result indicates that the

transesterification reaction could proceed within the temperature range, but the reaction time to complete the reaction varied significantly with reaction temperature.

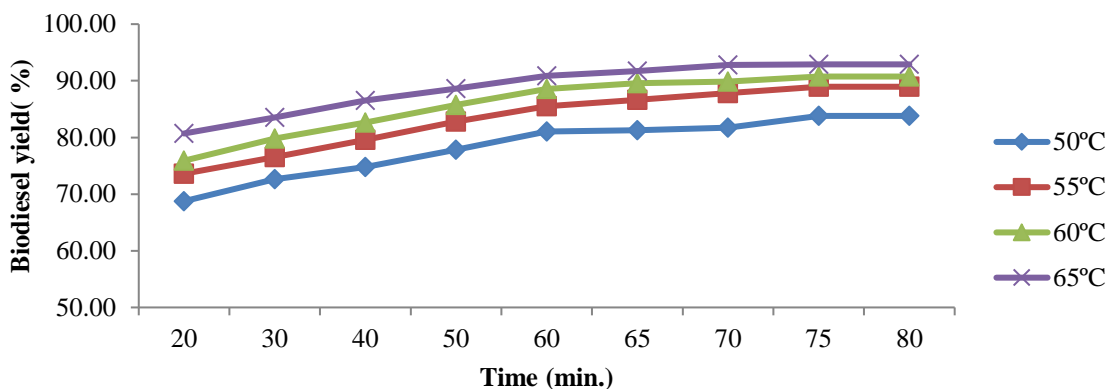


Figure 3: Effect of Temperature on Biodiesel Production

3.4 Effect of Stirring

The effect of stirring speed on biodiesel yield is presented in Figures 4. Percentage yields for the oil increase with increase in stirring speed. The highest

yield (92.90 %) of biodiesel was obtained at 375 rpm at methanol/ oil ratio 9:1, catalyst 1.0 % and temperature 65 °C respectively.

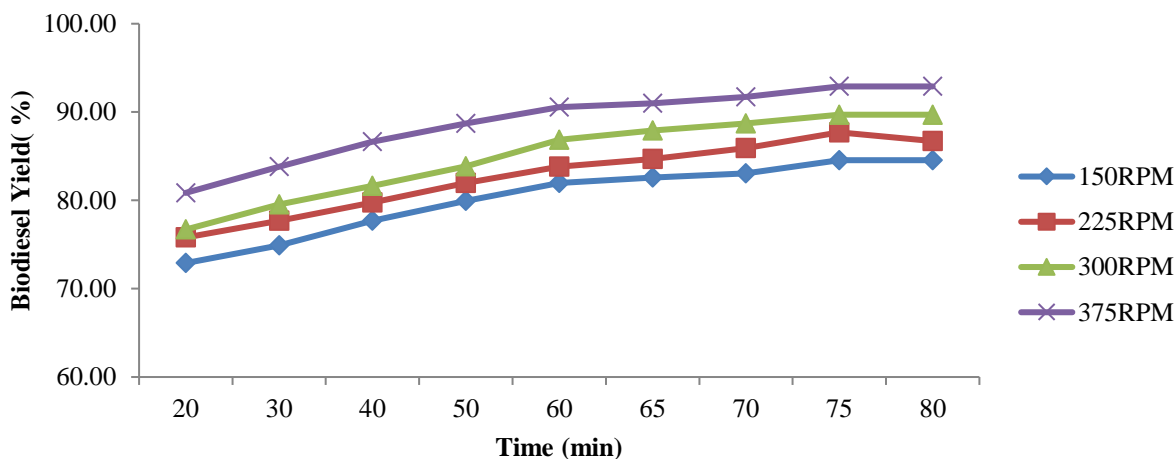


Figure 4: Effect of Stirring Speed on Biodiesel Yield

Table 1: Selected Fuel Properties of Silk Methyl Ester

Fuel Parameters	Silk methyl ester (SIME)
Specific gravity(g/cm ³)	0.88±0.01
Carbon residue (%)	0.02±0.01
Acid value (mgKOHg ⁻¹)	0.10±0.01
Sulphur content (%)	0.02±0.03
Flash point (°C)	170.0±1.00
Pour point (°C)	25.0±0.50
Viscosity (cSt)	4.70±0.14
Diesel index	27.13±0.48
Aniline (°C)	96.3±2.24
Calorific Value (MJ/kg)	41.48±0.40

4.0 DISCUSSION OF RESULTS

Figure 1 showed the effect of catalyst concentration and changes in biodiesel yield at different catalyst concentrations. Other parameters like temperature, methanol, reaction time and stirring rate were kept constant. From the results, high biodiesel yield was observed when 1.0 % NaOH was used to transesterify the oil sample. A yield of 92.90 % at reaction time of 80 mins was obtained from the seed oil. These results are in agreement with the result obtained by (Dorado *et al.*, 2004). It was observed in the course of the experiment that addition of excess amount of catalyst (NaOH) lead to the formation of emulsion which increased the viscosity of the mixture and lead to formation of gel, it was also observed that excess catalyst reduced the total yield of biodiesel.

Figure 2 presented the effect of methanol /oil molar ratio on Silk oil yield using NaOH as catalyst with concentration of catalyst, temperature and agitation rate kept constant. From Figure 2, the highest yield (92.90 %) was obtained at 75min and molar ratio of 9:1. This result indicate increased ester conversion and ensures complete reaction (Leung and Gu, 2006). The result obtained for Silk oil showed that at higher molar ratio 9:1 more methanol was required for achieving maximum yield in transesterification of silk oils.

The effect of temperature on Silk oil yields using NaOH as a catalyst is presented in Figure 3. Optimal yield (92.90%) of biodiesel from the silk oil was obtained at a reaction temperature of 65 °C. The transesterification reaction could proceed within the temperature range (50-65 °C) studied, but reaction time for completion of reaction varies significantly with reaction temperature. According to Leung and Gu (2006), increase in biodiesel yield could be due to decrease in viscosity of oil as reaction temperature increases. This condition increases the contact between oil and the methanol, thereby causing a better conversion of triglycerides. However when temperature is above 65 °C, product yield begins to decrease. This is because higher temperature can cause evaporation of methanol and also induce saponification reaction of triglycerides which can result in consumption of large amount of catalyst and methanol before certain level of conversion is achieved (Dorado *et al.*, 2004). Mixing is very

important in achieving good contact between the methanol/ alcohol solution and oil during transesterification reaction. The effect of rate of agitation on transesterification reaction was carried out under optimal conditions (9:1 methanol/oil, 1.0% catalyst, 65 °C) for Silk oil at different stirring speed. While the rate of stirring was varied, it was observed from Figure 4 that the ester yield for the oil studied increase with increase in rate of stirring. The highest yield of biodiesel (92.90 %) was obtained at 375 rpm. The effect of rate of agitation in transesterification reaction was carried out under optimal conditions (9:1 methanol/ oil, 1.0 % catalyst, 65 °C) at different stirring speeds. It was observed from Figures 1, 2, 3 and 4 that the ester yield for the studied oil increases with increase in the rate of stirring. From the results, it was observed that the optimal biodiesel yield at various experimental conditions was achieved at 9:1 methanol to oil molar ratio, 375 rpm and 65 °C respectively.

4.1 Fuel Properties of Biodiesel Produced from Silk Methyl Ester (SIME)

Results of the fuel properties of the produced biodiesels are shown in Table 1.

The density of petroleum products is usually expressed as a specific gravity and it is measured at a specific temperature commonly 20 °C (Dunn *et al.*, 1996). It is an important property in diesel engine performance, since fuel injection operates on a volume metering system (Song, 2000). The value of specific gravity obtained for Silk methyl ester was $0.88 \pm 0.01 \text{ g/cm}^3$ and is similar to 0.88 g/cm^3 and 0.86 g/cm^3 reported for cotton seed and sunflower oils respectively (Wawata, 2008; Barnwal and Sharma, 2005). The specific gravity obtained in this study conform to (ASTM D287, 1999) standard specification of 0.86 to 0.90 g/cm^3 for biodiesel fuel. Carbon residue content obtained was $0.02 \pm 0.01 \%$. This value is lower than the American Society for Testing and Materials (ASTM) limit of 0.05 %. This is an indication that the fuel can burn completely with little or no emission of carbon monoxide which is a potential Greenhouse gas. The complete combustion of the fuel reduces carbon deposits on the nozzle of diesel engine, (Zheng *et al.*, 2000). Acid value is a measure of the acid constituent in fuel; it is a good indicator of the quality of the fuel. The common acid constituents are free fatty acid which has high tendency for corrosion and also a symptom of water

in the fuel (Gbadge and Raheman, 2005). The results obtained in Table 1 shows acid value of 0.10 ± 0.1 mgKOHg⁻¹. This value was also within the ASTM limit, which indicates that they have low tendency of causing wear in storage tank and fuel system.

Sulphur content of the silk methyl ester was found to be 0.020 ± 0.03 %. This value was lower than the specified standard value of 0.05 % prescribed by ASTM. The result was lower than the 0.087 % reported by Bapjai and Tyagi (2006). Higher level of sulphur is not desirable in fuel as it contribute to the emission of greenhouse gases and acid rain. Biodiesel produced from silk methyl ester was expected to be clean and environment friendly than petroleum diesel. Biodiesel have been widely acknowledged as sulphur free fuel, hence is regarded as environmentally friendly.

Flash point is a property for determining the flammability of fuel (Gerpen *et al.*, 2004). Flash point is the lowest fuel temperature at which application of ignition source causes the vapour of the fuel sample to ignite under the prescribed test condition. The result obtained in Table 1 was 170 ± 1.00 °C. The value is within the range of 100 to 170 °C for biodiesel fuel (NREL, 2009). The high value indicate that the biodiesel produced is essentially free from methanol as even small amount of methanol can reduce the flash point and affect fuel pump seals (Gerpen *et al.*, 2004). According to Bapjai and Tyagi (2006), biodiesel with flash point greater than 93 °C is classified as nonflammable and nonhazardous from the perspective of storage and fire hazard, therefore biodiesel is much safer than petroleum. Pour point is the temperature at which the amount of wax from biodiesel is sufficient to gel the fuel. The result in Table 1 showed silk methyl ester had pour point of 25 ± 0.50 °F. The results suggest that the value is within the range of 5-50 °F recommended for biodiesel (ASTM D287, 1991). Biodiesel can be handled and stored safely without solidifying, but when the fuel is to be used in cold area, this would have to be preheated or blended with additives to reduce pour point (Hassan and Sani, 2006).

Viscosity measures the resistance of flow for a liquid due to internal friction of one part of a fluid over another. It affects the atomization of fuel upon injection into the combustion chamber, thereby

resulting in the formation of engine deposits. The efficient functionality of diesel engine depends on the kinematic viscosity of the fuel to be used. High viscosity is a major problem of using vegetable oil as fuel for diesel engine (Wang *et al.*, 2006). The result of viscosity from the study in Table 1 shows that the viscosity of Silk is 4.70 ± 0.14 cSt. The value for silk methyl ester lies within ASTM range of 1.9-6.0 cSt and is similar to 3.8-4.1 obtained for soya methyl ester (Bapjai and Tyagi, 2006).

Aniline point is the minimum temperature at which equal volume of anhydrous aniline and diesel fuel mix together. High aniline point indicates high diesel index and a very good ignition quality while low aniline point signifies poor ignition quality. The result of the Aniline point from the study in Table 1 is 96.30 ± 2.24 °C. The sample analysed have higher aniline point which indicate that the biodiesel fuel will have a short ignition delay and greater combustibility. The ASTM D611 (2001), specified minimum aniline value of 50 °C, therefore diesel engine will operate better on fuel with aniline number above 50. The low level of aniline number (below 50) will result in difficulty when starting, cause noise and emission of smoke. Calorific value is known to measures the amount of heat transferred to the combustion chamber during combustion and it also indicate the available energy in fuel (Demirbras, 2008). The results of calorific values of biodiesel obtained from this study is 41.48 ± 0.40 MJ/kg. This value is low and indicate that the biodiesel is an oxygenated fuel and the presence of oxygen in biodiesel molecule aids complete combustion of fuel in the engine.

5.0 CONCLUSION

The physicochemical properties of oil extracted from the seed of *Ceiba petandra* showed that the oil possesses suitable properties for biodiesel production. The samples gave high percentage of oil yield. Also, most properties of the oil conform to the ASTM standard. This implies that the seed oil is good raw material for biodiesel production and can be used in diesel engine.

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