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The Effect of Process Variables on the Transesterification of Refined Cottonseed Oil

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***Abstract*— This research work focuses on the production of biodiesel from refined cottonseed oil via alkaline-transesterification process using sodium hydroxide catalyst and methanol. Optimization was carried out using a 2^3 factorial design experiment, three variables namely mole ratio of methanol to oil, catalyst concentration and reaction temperature were studied at both high and low levels. The results obtained during the characterization of refined cottonseed oil include; Saponification Value (189.0 mgKOH/g), Acid Value (1.02 mgKOH/g), Free Fatty Acid (0.51%), Iodine Value (111.2 g/100g), Specific Gravity (0.920), Refractive Index (1.468). These results show the suitability of the oil for the production of biodiesel. Production yield and purity of biodiesel were used to verify the optimization. Results obtained showed that mole ratio of methanol to oil and catalyst concentrations were the most important variables affecting the yield of biodiesel. Optimum methyl ester yield of 96.23 % and a concentration of 99.54 % were gotten at a temperature of 40 ^oC, mole ratio of methanol to oil of (6:1) and catalyst concentration of 0.5 wt% at a constant reaction time of 90 minutes.**

***Index Terms*—Transesterification, Process variables, Energy source, Cottonseed oil**

I. Introduction

ENVIRONMENTAL concerns and depletion in petroleum resources have forced researchers to concentrate on finding renewable alternatives to conventional fossil fuels in general and in particular, diesel.

In today's world, petroleum is clearly the most important energy source, providing more than half the world's power, as well as being the basic raw material used in the manufacture of fertilizers, synthetic fibers, plastics, and synthetic rubber. As such, increasing environmental concerns and depletion in petroleum resources have forced researchers to concentrate finding renewable alternatives to conventional diesel fuels [1]. In Nigeria, concerns about dwindling supplies, its unstable and rising cost, and environmental problems have motivated researchers to extensively seek for an alternative, renewable energy sources. Among these alternative sources, vegetable oils have gained considerable attention since they are derived from renewable resources, can be domestically produced, and are not as harmful as petroleum to the environment [1]. However, the use of these oils is restricted to diesel engines, since they contain free fatty acids (FFAs), phospholipids, sterols, water, odorants, and other impurities. In addition, vegetable oil's high viscosity, about 11-17 times higher than diesel fuel, affects the flow properties of the fuel, such as spray atomization, consequent vaporization, and air-fuel mixing in the combustion chamber, leading to an adverse effect on the combustion process [2].

In Nigeria, it was reported by the Energy Commission of Nigeria in 2005 that Nigeria's fossil led economy is under severe pressure to diversify as the flaring of enormous amounts of natural gas has increased the level of CO_2 in the atmosphere, contributing to global warming [3].

Biodiesel is a variety of ester-based oxygenated fuels derived from natural, renewable biological sources such as vegetable oils. Its name indicates, use of this fuel in diesel engine alternate to diesel fuel. Biodiesel operates in compression ignition engines like petroleum diesel thereby requiring no essential engine modifications. Moreover it can maintain the payload capacity and range of conventional diesel [3-6]. It can be produced from renewable vegetable oils/animal fat and hence improves energy security and economic independence [6]. Various vegetable oil such as palm oil, soybean oil, sunflower oil, rape seed oil and canola oil have been used to produce biodiesel [7]. This study is concerned with the optimization and characterization of biodiesel from cotton seed.

II. Materials and Method

A. *Materials*

Refined cottonseed oil used in this experiment was purchased from Biodiesel Company and its physico-chemical properties are presented in Table 1. Chemicals such as methanol and sodium hydroxide (catalyst) used was manufactured by Aldrich Co. Ltd England with the methanol having a purity of 99.5 %.

B. *Experimental Design*

A 2^3 factorial experimental design was used to determine the optimum conditions, three variables were studied at both high and low levels with the response been methyl ester

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yield. The high level of alcohol/oil mole ratio was 1:8 and the low level was 1:6. The high level of temperature was chosen at 60 °C and the low level at 40 °C. The high level of catalyst concentration was chosen at 1.0 % and low level was 0.5 % sodium hydroxide catalyst by weight of cottonseed oil. A constant reaction time of 90 minutes and constant agitation were maintained throughout the experiment.

C. *Method*

Transesterification reactions were carried out in 500 ml flask. The reactor was filled with 100 g of refined cottonseed oil. Sodium hydroxide catalyst was dissolved in methanol and then added to the reactor. The mixture was heated to selected temperature. After the end of the reaction, the mixture was cooled to room temperature and transferred to a separating funnel. The two layers (biodiesel and glycerol) were separated by sedimentation. The methyl ester phase (biodiesel) was washed with hot distilled water and drying was done by heating the biodiesel to a temperature above 100 °C to remove water molecules.

TABLE I
SOME PROPERTIES OF REFINED COTTONSEED OIL

Properties	Refined cottonseed oil
Specific Gravity at 25 °C	0.921
Refractive Index at 40 °C	1.468
Iodine Value, g iodine/g oil	111.2
Saponification Value mg KOH/g oil	189
Acid Value, mg KOH/g oil	1.02
Free fatty acid value	0.51

Determined according to AOCS standards

TABLE II
REACTION CONDITIONS, PRODUCTION YIELD, METHYL ESTER CONCENTRATION AND METHYL ESTER YIELD.

Run	A	B	C	D	E	F
1	1:6	40	0.5	96.67	99.54	96.23
2	1:8	40	0.5	81.70	98.73	80.66
3	1:6	60	0.5	92.83	99.21	92.10
4	1:8	60	0.5	82.97	99.01	82.15
5	1:6	40	1.0	92.74	99.53	92.30
6	1:8	40	1.0	88.98	99.01	88.10
7	1:6	60	1.0	83.06	98.75	82.02
8	1:8	60	1.0	86.05	99.84	85.91

A= Mole ratio of oil to methanol, B= Reaction temperature (°C), C= Concentration of sodium hydroxide (wt %), D= Production yield (wt %), E= Methyl ester concentration (wt %), and F= Methyl ester yield (wt %)

III. RESULT AND DISCUSSION

The experimental results which were obtained from 8 runs according to the experimental design are shown in Table II. From the results obtained, catalyst concentration and molar ratio of oil to methanol were the most important factors affecting the methyl ester yield while temperature had little significant effect on methyl ester yield.

A. *Effect of Oil to Methanol Molar Ratio*

The methyl ester yield decreased with increasing molar ratio from 6:1 to 8:1. This can be explained on the basis of the reactant (oil) concentration in the reaction mixture. By increasing alcohol to oil molar ratio, the amount of alcohol was increased, therefore the cottonseed oil and catalyst concentrations were diminished, which reduced the rate of reaction. Boocock *et a*l. [8] reported a similar trend. At higher reaction temperature, a similar trend of ester concentration (ester concentration decreased with an increase in alcohol to oil molar ratio) was observed. As alcohol to oil molar ratio was increased, the polarity of the system was also increased. As a result, the rate of the reaction as well as the % conversion was increased. Leung and Guo, [9], observed that further increase of alcohol content does not increase the yield of biodiesel but instead increases the cost of alcohol recovery.

B. *Effect of Catalyst Concentration*

Sodium hydroxide catalyst was used at 0.5 wt% and 1.0 wt% in this work. It can be observed that the methyl ester yield decreases with increase in catalyst concentration. This can be due to the formation of soap by the oil and excess sodium hydroxide used. Freedman *et al* [10] found that the sodium methoxide would be more effective because mixing of sodium hydroxide with methanol produce little amount of water which inhibit the formation of end product (Biodiesel) due to the hydrolysis reaction.

C. *Effect of Reaction Temperature*

In this work, the effects of reaction temperature on production and methyl ester yield decreases with increase in temperature. This can be as a result of higher solubility of reactants because higher temperature reduces the separation of methyl ester and glycerol phase. Also, a higher reaction temperature of 60 °C that was chosen is close to the boiling point of methanol which can lead to evaporation of some of the methanol during transesterification process. As a result, lower methyl ester yield was observed at 60 °C as compared to that obtained at 30 °C leaving all other variables constant under the same reaction conditions. Based on the result obtained, a temperature slightly above room temperature (40 °C) was considered to be the optimum temperature. Eevera *et al.*, [11], observed that longer reaction time leads to the reduction of end product (biodiesel) due to the reversible reaction of transesterification resulting in loss of esters as well as soap formation.

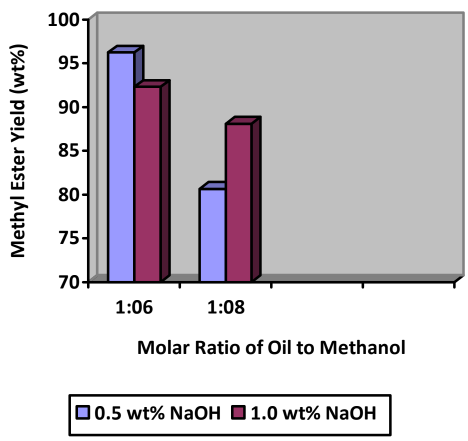

Fig 1. Effect of molar ratio of oil to methanol on the methyl ester yield at 90 minutes

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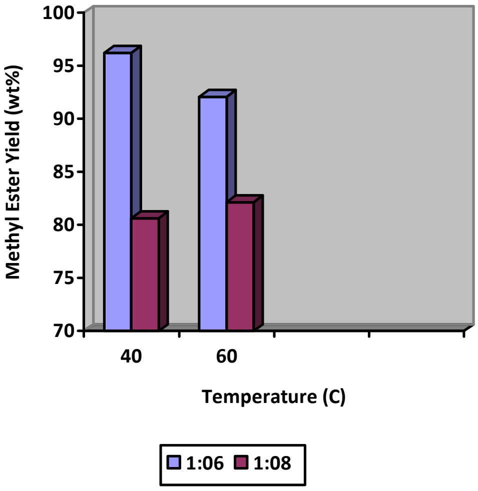

Fig 2. Effect of reaction temperature on methyl ester yield at 90 minute

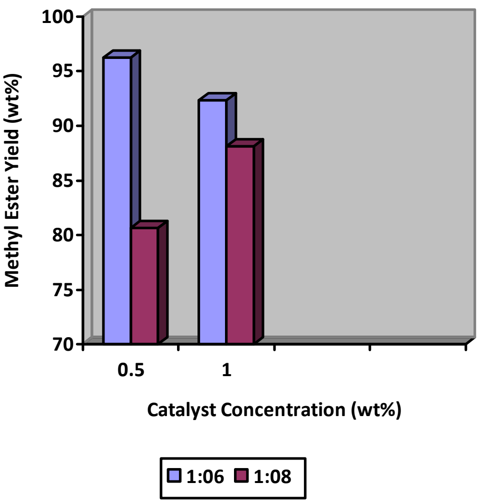

Fig 3. Effect of catalyst concentration on methyl ester yield at 90 minute

IV. Conclusion

Based on the experimental results, it was concluded that the optimal conditions for the transesterification of refined cottonseed oil are as follows: oil to methanol molar ratio, 1:6; temperature, 40 °C; catalyst concentration, 0.5 % at constant reaction time and agitation of 90 minutes and 300 rpm respectively. This optimized condition was validated with the actual methyl ester (biodiesel) yield of 96.23 % and a methyl ester concentration of 99.54 %. Characterization of biodiesel produced show that it can serve as a replacement for petroleum diesel.

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