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Optimization of Biodiesel Production from Seeds of Cotton and Calabash Via In Situ Transesterification using CaO as Catalyst

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ABSTRACT

Utilization of fossil fuels results to toxic emissions that adversely affect the environment. In order to arrest this challenge it is pertinent to rely more on cleaner fuels. This study evaluates the potential of producing clean and affordable fuel, biodiesel, in line with United Nations Seventh Sustainable Development Goal. Cotton and calabash seeds were used to produce biodiesel in this study. The seeds were mixed at different ratios and the catalyst concentration was controlled at 1% for two hours under a temperature of 60°C. About 70% of the cost of producing biodiesel rests on feedstock alone. To reduce cost, cheap and underutilized seeds were chosen for this study. In addition, the biodiesel was produced via a technique, in situ transesterification, which lowers production cost. In this process the oil from the seeds were extracted and converted to biodiesel in a single step. The highest yield of 61.33% was obtained at a ratio 1:5 of the cotton to calabash seeds respectively. The physicochemical parameters analysed for are: saponification value (210.38 mg KOH/g); iodine value (59.14 mgI_2/100g); acid value (1.68 mg/g); free fatty acid (0.84); cetane number (58.93); ester value (208.7); high heating value (39.91 MJ/kJ) and specific gravity (0.87). The results of the properties were compared with standards and this shows that the biodiesel produced has very good properties.

***Keywords*:** biodiesel, in situ transesterification, underutilized seeds, physichochemical analysis: iodine value, cetane number.

Introduction

Transportation of goods and services influences the world's economy and transportation relies mostly on energy from petroleum fuels.[1] The 1970s oil crisis gave rise to the search for clean and sustainable fuels that are better than fossil fuels.[2] Continuous utilization of fossil fuels leads to toxic emissions.[3] The burning of fossil fuels releases carbon dioxide which influences the increase in the heat concentration on the earth.[4] In Nigeria, depletion of fossil fuels, environmental concerns and ever-changing prices has necessitated the search for alternative by researchers.[5] One of such alternatives is biodiesel.

Biodiesel is an alkyl ester of long chain fatty acid that is usually obtained from alkyl glycerides of animal fats and vegetable oils.[6] The worldwide concern about air pollution from combustion engines has led to the search for oxygenated fuels like biodiesel which can replace petro-diesel.[7] Biodiesel can be used in diesel engines without changes and is considered to be sulphur-free, less toxic, biodegradable, oxygenated and environmentally friendly.[8] Biodiesel has no aromatic compounds, about 10-11% of biodiesel is oxygen and it has lower emissions of hydrocarbons, carbon monoxide and carbon dioxide.[9] Biodiesel can be used in diesel engines without modification and conventional diesel is miscible with biodiesel. Usually, BX is used to represent biodiesel blended with diesel where X is the percentage of biodiesel. For example, B20 contains 20% biodiesel and 80% diesel.[10]

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In transesterification, the molecules of the triglycerides are broken down by alcohol in the presence of a catalyst to yield biodiesel (alkyl esters) and glycerol which is the by-product.[11] The transesterification reaction, the chemical structure of the triglycerides in the oil is broken down by exchanging alkyl groups between the alcohol and the triglyceride.[12] In situ transesterification involves the extraction of the oil from the seed and its conversion to biodiesel in a single step.[13] Almost every oil-bearing material can be subjected to in situ transesterification.[14] Extraction and transesterification are done simultaneously in situ transesterification.[15] In situ transesterification reduces cost and it is time effective.[16] Heterogeneous catalysts are more advantageous than homogeneous catalysts due to their simple separation process, great catalytic activity, environmentally benign, reusability of catalyst, fewer washing steps, less toxicity and less cost.[17] Calcium oxide is an encouraging heterogeneous basic catalyst that is renewable, cheap, and easily available.[18]

Cotton belongs to the *Malvaceae* family and it is used to weave garments.[19] Cotton seed oil has 55.2-55.5% of linoleic acid, 19.2-23.6% of oleic acid and 11.67-20.1% of palmitic acid.[20] Calabash is amongst the crops cultivated in north-western Nigeria with no large-scale application and it has a short maturity time of 3-4 months that produces about 50% inedible oil.[21] According to Ibrahim *et al.*[22] calabash seeds have 59.98% of saturated ester and 40.02% of unsaturated ester. This work presents a study on the optimal production of biodiesel from a mixture of underutilized seeds. The idea is to check for the potential of producing biodiesel that will replace conventional diesel. Cost-effective methods and processes were employed in this study to complement the seventh Sustainable Development Goal "clean and affordable energy".

Materials and Methods

Sampling

Cotton and calabash seeds were purchased in March 2019 from markets in Kaduna State, Nigeria. The cotton seeds were identified by U.S. Gallah of the Kaduna State University as *Gossypium hirsutum* and assigned the voucher 1492. The calabash seeds were identified as *Lagenaria siceraria* and assigned the voucher number 3792. The seeds were washed with water to remove impurities. The seeds were ground to powder separately and stored separately in a polythene bag for further use. Egg shells were sourced from food vendors in Kaduna, Nigeria. The impurities in the shells were removed and then reduced to fine powder. The ground egg shell was stored in a polythene bag for future use.

Catalyst preparation

The stored egg shell was calcinated for two hours at 800°C. The thermal decomposition of the $CaCO_3$ in the egg shells produced the catalyst, CaO, as shown in equation 1.

$$CaCO_3 \xrightarrow{800^\circ C} CaO + CO_2 \quad 1$$

Production of Biodiesel via In situ Transesterification

The standard method of Muhammad *et al.*[23] was used to produce biodiesel via in situ transesterification. In this method, the oil is extracted and converted to biodiesel in a single step. The seeds of calabash and cotton were mixed at different ratios (1:2, 1:3, 1:4, and 1:5) to produce eight biodiesel samples. Exactly 30 g of the powdered seed was measured and transferred to a thimble. Exactly 300 mL of methanol was added to 150 mL of n-hexane to make the solvent system. The solvent system was transferred to a round bottom flask. The catalyst at 1% of the seed sample weight (30 g) was added to the round bottom flask.

The round bottom flask was placed on a heating mantle and joined to the soxhlet extractor. The heating mantle temperature was set at 60°C and the reaction took place for two hours. After two hours, the set-up was allowed to cool before disconnecting the round bottom flask from the soxhlet extractor. The extract in the round bottom flask was siphoned in to a separatory funnel and allowed to settle till the next day. After settling, the extract formed two phases. The upper phase was biodiesel while the lower phase was glycerol. The glycerol was drained out of the separating funnel. The biodiesel in the separating funnel was washed with water to remove impurities. The biodiesel was drained and heated at 110°C. The purified biodiesel was stored in a sample bottle.

Physicochemical analysis

Determination of High Heating Value (HHV)

High heating value is the energy that is released when biodiesel is completely burnt.[24] According to Muhammad *et al*.[25] high heating value was calculated by substituting the values for iodine value and saponification value of the biodiesel in equation 2:

$$HHV = 49.43 - 0.041SV - 0.015IV \quad 2$$

Where HHV = high heating value; IV = iodine value; and SV = saponification value

Determination of iodine value

Iodine value is the degree of unsaturation of biodiesel and with high iodine value, the biodiesel will easily undergo oxidative rancidity.[26] The iodine value was obtained according to the standard procedure reported by Junior *et al.*[27]. The biodiesel (1 g) was measured into a conical flask and carbon tetrachloride (20 mL) was added then Wijs solution (25 mL). The mouth of the conical flask was covered and the flask was swirled until there was a homogeneous mixture. The conical flask was stored for an hour and 20 mL of 10% potassium iodide solution was added, then 125 mL of distilled water was added. The mixture in the flask was titrated against 0.1 M sodium thiosulphate until the yellow color almost disappeared; then 2 mL of starch indicator was added turning the solution bluish. Titration was stopped when the blue colour disappeared. A blank titration was carried out under the same conditions without the sample. The iodine value of the biodiesel was determined using equation 3:

$$Iodine\ value = \frac{(A - X) x\ M\ x\ 12.69}{mass\ (g) of\ biodiesel} \quad 3$$

A = volume of sodium thiosulphate used in blank titration
X = volume of sodium thiosulphate used in test titration
M = molarity of sodium thiosulphate solution.

Determination of saponification value

The amount of potassium hydroxide that will saponify the oil is known as saponification value.[28] The saponification value of the biodiesel was determined according to the standard method of Muhammad *et al.*[29] A sample of the biodiesel was weighed (2.0 g) into a conical flask and ethanolic potassium hydroxide (0.5 M, 20 mL) was added. The flask was heated for exactly 30 minutes and cooled to room temperature. The excess potassium hydroxide was back titrated with hydrochloric acid (0.5 M) and the indicator used was phenolphthalein. Blank titration was conducted under the same exact conditions without the biodiesel. The saponification value was calculated using the equation 4:

$$Saponification\ value = \frac{56.1\ x\ M\ (Va - Vb)}{W} \quad 4$$

Where V_a is the volume of hydrochloric acid used in blank titration, V_b is the volume of hydrochloric acid in test titration; M is the molarity of the HCl used and W is the weight of the biodiesel.

Determination of ester value

According to Onukwuli *et al.*[30] ester value of the biodiesel sample was determined by the difference between the acid value and the saponification value as shown in equation 5. Thus:

$$Ester\ value = Saponification\ value - Acid\ value \quad 5$$

Determination of acid value and free fatty acid

The acid value is the amount of unreacted fatty acids in the oil.[31] The acid value and free fatty acid values were determined according to a standard procedure as reported by Muhammad *et al*.[32] Isopropyl alcohol (75 mL) was measured and transferred into a beaker; then toluene (75 mL) was measured and transferred into the same beaker. The biodiesel (2 g) was added followed by 2 drops of phenolphthalein indicator into the beaker. The mixture was titrated against 0.1 M potassium hydroxide until pink color appeared. The acid value of the biodiesel was calculated using equation 6:

$$Acid\ value = \frac{56.1\ x\ M\ x\ Titre\ value}{W} \quad 6$$

Where M = Molarity of potassium hydroxide, 56.1 = Molecular weight of potassium hydroxide, W = Weight of the sample.
The Free Fatty Acid (FFA) was determined using equation 7

$$FFA = 0.5\ x\ Acid\ value \quad 7$$

Determination of specific gravity

The Specific gravity measures the density of the biodiesel to that of water.[33] The specific gravity was determined according to Onukwuli *et al.*[34] An empty beaker was weighed and 20 mL of water was measured and transferred into the empty beaker. The new weight of the beaker containing the water was recorded. Furthermore, 20 mL of the biodiesel sample was transferred into the same empty beaker and the measured weight was recorded. Equation 8 was used to calculate the specific gravity of the biodiesel:

$$Specific\ gravity = \frac{Weight\ of\ sample}{Weight\ of\ equal\ volume\ of\ water} \quad 8$$

Determination of cetane number

Cetane number is an important quality indicator of biodiesel and it measures the easiness for biodiesel to undergo combustion in the engine.[35] According to Muhammad *et al.*[36] the cetane number of the

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biodiesel was determined by substituting the saponification and iodine values of the biodiesel in equation 9.

$$Cetane\ number: 46.3 + \frac{5458}{SV} - 0.225IV \quad 9$$

Where SV = saponification value; IV = iodine value

Results and Discussion

The catalyst was characterized using XRF analysis and it revealed that it contained 91.322% CaO (Figure 1). Figure 1 presents the yield by the percentage of biodiesel produced from the blend. The results of the physicochemical analysis of the biodiesel are shown in Table 1.

The blend was produced in varied ratios of calabash and cotton seeds. The yield continuously increased with more ratios of calabash seeds. So, the highest yield was obtained at a 5:1 ratio of calabash to cotton seeds while the least yield was produced at 1:2 ratio of calabash to cotton seeds. It can, therefore, be deduced that the more the calabash seeds the more the biodiesel yield. Saponification value can be used to check the quality of biodiesel. The adulteration of the biodiesel can be checked with saponification value.[37] A high amount of saponification can reduce biodiesel yield and lead to challenges during purification.[38] A high saponification value indicates that the oil is rich in triglycerides.[39] Triglycerides in oil transform completely to biodiesel when there is a surplus amount of methanol.[40] If the methanol in this study had been supplied in a surplus amount, the yield of the biodiesel would have increased. This is because the triglycerides would have been transformed in to biodiesel.

Iodine value determines the chemical stability of biodiesel.[41] The iodine value of the biodiesel is within the standards of EN14214 and ASTM D6571. This indicates that the biodiesel will not be easily susceptible to oxidative rancidity. High acid value corrodes combustion engines and fuel supply systems.[42] The acid value of the biodiesel produced is beyond ASTM D6571 and EN14214 standards. To reduce the adverse effect of high acid value, the biodiesel should be blended before use or should be used in highly corrosion-resistant engines. Engine performance and emission of gaseous and particulate matter can be influenced by cetane number.[43] The produced biodiesel in this study has a cetane number within permissible limits. This shows that the fuel will be of good quality. Specific gravity is used to assess the quality of biodiesel.[44]

The efficiency of biodiesel and its energy content can be determined through the high heating value of the fuel.[45] Both specific gravity and high heating value of the biodiesel are within standard limits of ASTM D6571 and EN14214. This shows that the biodiesel possesses excellent properties. The feedstock is both rich in unsaturated fatty acids. The GCMS result shows that the biodiesel contains octadecenoic acid (Figure 3) as the dominant fatty acid methyl esters (FAME). Other FAMEs present are hexadecanoic acid (Figure 4) and methyl stearate (Figure 5). This is why some of the physicochemical parameters like cetane number and high heating value are not very high because of the dominant effect of the unsaturated fatty acid.

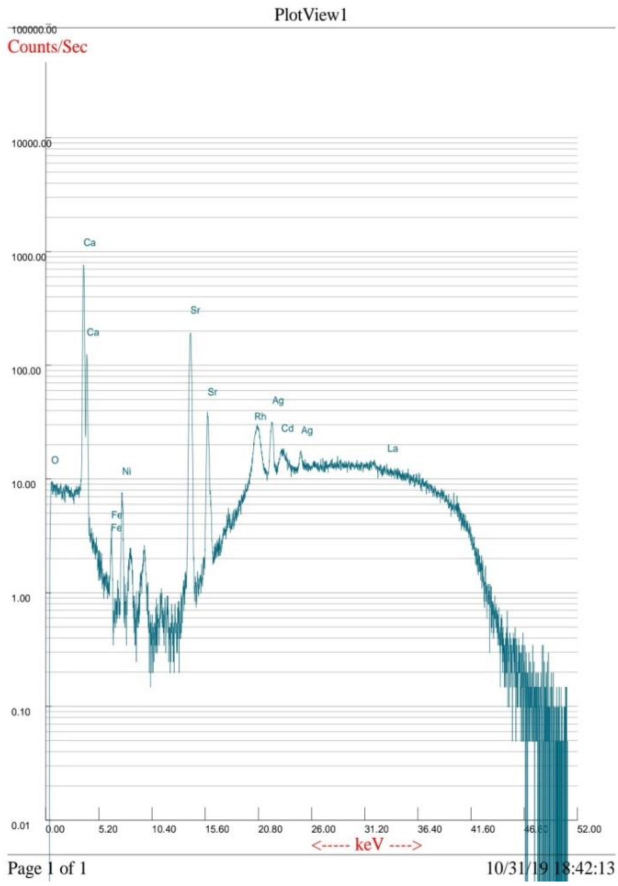

Figure 1: The XRF spectra of the Egg Shell after calcination

Table 1: Physicochemical test results of produced biodiesel from blend of cotton and calabash seeds

Test	Biodiesel	ASTM D6571	EN 14214
Saponification Value (mg KOH/g)	210.38	-	-
Iodine Value (mgI_2/100g)	59.14	115 max	120 max
Acid Value (mg/g)	1.68	0.8 max	0.5 max
Free Fatty Acid	0.84	0.4	-
Cetane Number	58.93	47 min	51 min
Ester Value (mg/g)	208.7	-	-
High Heating Value (MJ/kJ)	39.91	-	35 min
Specific Gravity	0.87	0.88 max	0.9 max

Conclusion

The depletion of supplies and environmental impacts of fossil fuels has led researchers to search for better substitutes like biodiesel. In this study, the biodiesel was produced from easily available raw materials via in situ transesterification in order to lower cost. This is to conform to the United Nations seventh Sustainable Development Goal “clean and affordable energy”. The biodiesel produced from a mixture of cotton and calabash seeds in this study have promising characteristics to serve as a substitute for conventional diesel.

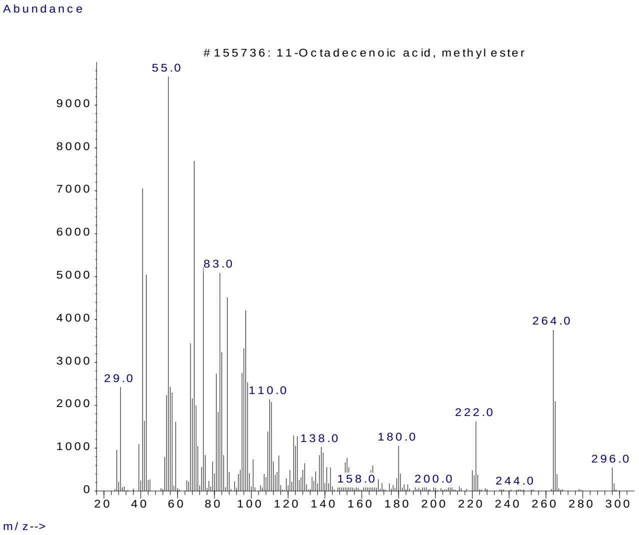

Figure 3: GC-MS spectra of octadecenoic acid

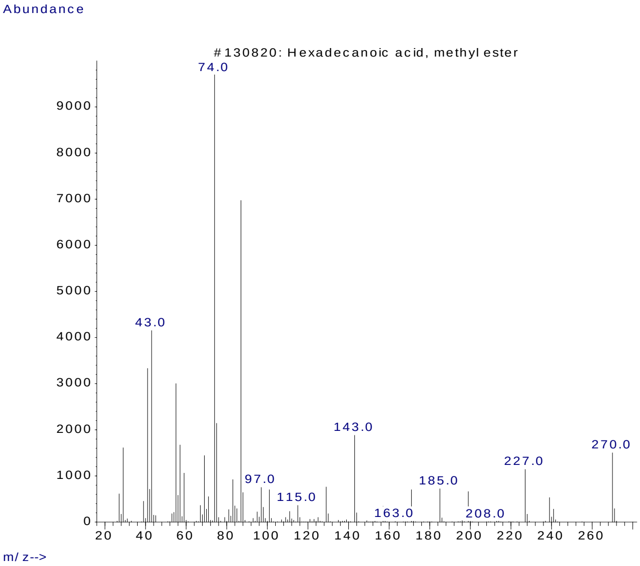

Figure 4: GC-MS spectra of hexadecanoic acid

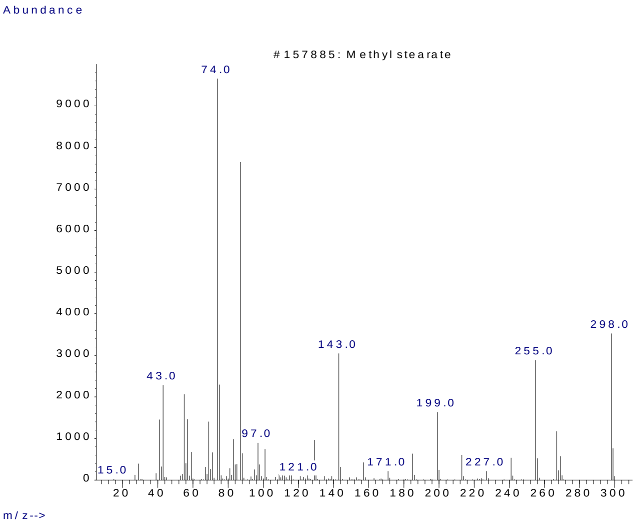

Figure 5: GC-MS spectra of methyl sterate

Conflict of Interest

The authors declare no conflict of interest.

Authors' Declaration

The authors hereby declare that the work presented in this article is original and that any liability for claims relating to the content of this article will be borne by them.

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