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Preparation and Characterization of Biodiesel from Gmelina *arborea* Seed using Waste Material Derived Catalyst

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ABSTRACT

The emission of greenhouse gases, produced by burning of fossil fuel, is reduced by the use of biodiesel. The adoption of biodiesel helps to produce a cleaner environment and renewable fuel, fossil fuels are depleted by continuous usage. The aim of this study is to prepare and characterize biodiesel using Gmelina *arborea* in the presence of solid waste-derived heterogeneous catalysts. The first step was the synthesis of catalyst using banana peels at different calcination temperatures leading to the production of 4 catalytic samples .Then, the catalysts were impregnated with KOH and utilized for biodiesel preparation. The catalysts were characterized using FTIR spectroscopy. The ASTM standard method was used to analyze the physicochemical properties of the biodiesel. The efficiency of the catalyst at different calcination temperatures (700, 600, 500 and 400 ^{0}C) were tested for the trans-esterification of purified oil to biodiesel at the catalyst weight (1-5% wt), temperature (60-120 °C), and oil to methanol ratios ranging from 1:6 to 1:15.The highest biodiesel yield (95.4 %) was obtained when 3% wt of catalyst A_1 (700), with oil to methanol ratio of 1:9 at 60 °C and a reaction time of 2 hours were used. This study showed that Gmelina *arboea* oil is a good source of biodiesel with physicochemical properties that suit the present day diesel engine and could replace nonrenewable energy in the future. The preparation of catalyst from waste materials can reduce the expenses incurred in the acquisition of chemical catalyst and expensive waste disposal systems. Additionally, biodiesel produced from Gmelina *arborea* could be an alternative source for many industrial chemicals and fuels that are currently sourced from petroleum which is non-renewable, and very harmful to the ecosystem.

Key words: Biodiesel, physicochemical properties, Gmelina *arborea*, trans-esterification, renewable energy.

INTRODUCTION

Trans-esterification is a procedure used to produce biodiesel. This is accomplished by forming methyl, ethyl, or propyl ester by combining lipids or some vegetable oil with an alcohol [1]. Because biodiesel is compatible with existing diesel engines, there is no need to modify them to run on the fuel engine; nonetheless, most engines require certain changes in order to run on pure biodiesel [2]. For more than 200 years, the world has relied on fossil fuels such as; coal, oil, and natural gas. The rate at which the source of fossil fuels disappears is directly correlated with the rate at which those fuels are consumed. Due to the significant production of greenhouse gases, the widespread use of fossil fuels is the primary cause of the destruction of our ecosystem. Nations requires biodiesel as a renewable energy source to replace gasoline and diesel [3]. The search for a clean, renewable energy source that is also economically viable to replace fossil fuels is what led to the development of biodiesel. The fact that biodiesel emits fewer greenhouse gases than regular diesel is one of its advantages over regular diesel. With low cost, wide availability, ease of transport and storage, fossil fuels have a significant impact on the advancement of modern society, but the environment is severely harmed when fossil fuels are burned. The use of biodiesel as an additional fuel for diesel engines is promoted [3].

According to Umeuzuegbu et al [4], mono-alkyl esters of long-chain fatty acids generated from vegetable oil are the building blocks of biodiesel. The commercialization of biodiesel is hampered by the price of raw materials which makes producing it more expensive than producing regular diesel. To make the production of biodiesel more affordable and to prevent food shortages that could result from using edible vegetable oils for biodiesel, it is therefore necessary to investigate alternative feed stocks such as the non-edible vegetable oils from seed and leaves [6].

Gmelina *arbohea* is a large tree which provides wood used in construction and furniture. The tree is widespread and has a rapid rate of growth. At about 40 meters tall and 0.14 meters in diameter, it naturally exists in Asian nations like Cambodia, Thailand, Vietnam, Myanmar, and

India. It has also been planted as a fast-growing wood tree in Brazil, the Philippines, Malaysia, Malawi, the Ivory Coast, and Nigeria. Basumatary [5] stated that it is employed for gardens and avenues. The tree is an important medicinal plant. It contains digestive, cardiotonic, laxative, astringent, bitter, diuretic, nervine, and laxative effects [7].

The two types of heterogeneous catalysts used in trans-esterification are alkaline and acidic. Heterogeneous catalysts can be easily taken out of the finished product and recycled for future use. Cost-effective production of biodiesel is made possible by the catalysts repeatability [4]. Various types of homogeneous and heterogeneous catalysts are used in the trans-esterification reaction to produce biodiesel, with homogeneous catalysts being the most common. Homogeneously catalyzed reactions occur faster compared to heterogeneously catalyzed reactions [1]. Any fuel made from biomass, such as plant, algae, or animal waste, falls under this category.

Biofuel is regarded as a renewable form of energy because its source (feedstock) can be simply and quickly renewed [7]. Biofuel is seen as a secure and suitable substitute for petroleum and other fossil fuels, particularly given its affordability, which can help slow the rise in petroleum prices. When compared to the extremely slow natural process involved in the development of fossil fuel, the manufacturing of biofuel takes place in a very short amount of time [8]. The United States Energy Information Administration (EIA) noted that biofuels can be utilized for heating and electricity in addition to transportation. When compared to fossil fuels, biofuel has a significantly higher capacity to contribute to the reduction of greenhouse gas emissions [9].

Some biofuels, such as wood, have been utilized as a raw material for heating systems for a long time. Wood can be burned to produce heat, which can be used to run a generator in a power plant to produce electricity. Due to the requirement for them to be used as a source of fuel for transportation, liquid biofuel is of great importance. Bioethanol is one of the types of biofuels and is created from ethanol by fermenting starch or sugar. According to Rodionova et al [10], the second type of biofuel is biodiesel, which is made from oily feedstock like groundnut or used cooking oil. Due to the similarities in qualities between biodiesel and fossil fuel, diesel engines may use biodiesel with little to no changes [7].

Modern technology is compatible with biodiesel. Unlike engines that are modified to run on vegetable oil or waste oil, diesel engines. However, as most engines cannot run on a pure diesel engine without modifications, it is frequently blended with petrol diesel to a normal level of less than 10% [11].

The aim of this study is to produce and characterize biodiesel using Gmelina *arborea* in the presence of solid waste-derived heterogeneous catalysts. Thus biodiesel produced from Gmelina *arborea* could be a good alternative source for many industrial chemicals, fuels that are currently sourced from petroleum which is non-renewable and affects the ecosystem.

MATERIALS AND METHODS

Sample Collection and Preparation

The fruits of Gmelina *arbohea* was collected from two different locations; University of Abuja Mini campus FCT and Ibadan, Nigeria. The fruit was soaked for eight days to ease the de-husking process of the seed. Then, the fleshy part of the fruit seed was removed and the woody part was dried in the sun for three days. Then, the part of the seed which contains the oil was removed from the woody kennel. It was sun-dried and ground into coarse powder. The sample was further separated according to their sizes after grinding. The sample was stored in a dried container [12].

Catalyst Preparation

Pieces of banana peel were washed with distilled water to remove unwanted impurities. About 200 g of the banana peel was introduced into an oven and dried at 373 k for 12 hours. It was ground with mortar and pestle to powder. The powder was divided into two equal portions. The first portion was calcined at 700 ^{o}C for 2 hours at a heating rate of 10 ^{o}C min^{-1} and was allowed to stay in the furnace at a constant temperature of 700 ^{o}C for 2 hours. The second portion was calcined at 600 ^{o}C for 2 hours at a heating rate of 10 ^{o}C min^{-1} and was allowed to stay in the furnace at a constant temperature of 600 ^{o}C for 2 hours. Each of the samples was allowed to cool at the same rate it was heated, after which the powder was collected and stored in an airtight container and were labeled sample A and sample B respectively [13].

Impregnation of Catalyst

About 20% wt of KOH was dissolved in a 50 ml of distilled water in a beaker. The beaker was heated at a temperature of 80 ^{o}C and was stirred continuously. While stirring, 100 g of sample A was added slowly and the stirring continued until a uniform mixture was obtained and dried. The sample was divided into half. The procedure was repeated for sample B.-About 50 g of Sample A was calcined at 600 °C for 2 hours at a heating rate of 10 °C $min.^{-1.}$ The remaining 50g of Sample A was calcined at 500 °C for 2 hours at a heating rate of 10 °C min^{-1}. The resulting sample was labeled A_1 and A_2 respectively. About 50 g of Sample B was calcined at a temperature of 700 °C for 2 hours at a heating rate of 10 °C min^{-1} and the remaining 50 g of Sample B was calcined at a temperature of 600 °C for 2 hours at a heating rate of 10 °C $min.^{-1}$ The resulting samples was labeled B_1 and B_2 respectively. Samples A_1, A_2, B_1, B_2 remained at their calcined temperature for 2 hours and allowed to cool at same heating rate.. Samples A_1, A_2, B_1, B_2 were separately ground into fine powder using mortar and pestle and the powder was stored in an airtight container was labeled accordingly before the trans-esterification process [14].

Soxhlet Extraction

The ground seed particls were charged into the thimble of the soxhlet apparatus. About 300 ml of the solvent was transferred into the round-bottom flask of the soxhlet apparatus. Heat was applied to the solvent in the flask and was kept at isothermal condition. The flask was heated until the solvent boiled, and vaporized on top of the apparatus. The vaporized solvent was allowed to condense and then flowed to the thimble containing the ground seed, hence, extraction occurs. This process continued for about 4 hours to ensure maximum extraction. After the extraction, the solvent and oil was heated and the solvent vaporized because it has lower boiling point leaving the oil behind. The separated oil was kept in a dark container for further physiochemical tests and trans-esterification [14].

Trans-esterification of Oil

A 250 ml 3-necked round bottom flask was placed on a hot plate with stirrer. About 6.6 ml of the oil was introduced into the flask and catalyst (A_1) which was about 5% wt, was added to the oil, The condenser and a thermometer were attached while the third opening was closed. The mixture was heated to a temperature of about 60 °C and was continuously stirred. Methanol was added immediately the temperature reached 60 °C and the heating continued for 1 hour. Then, the condenser was removed and the round bottom flask was opened while the heating continued to ensure the methanol evaporates. The mixture was allowed to cool for about 10 min and was left for 24 hours in the flask .A thick brown layer settled at the bottom which was the biodiesel and the upper light layer was the glycerin. The layers were separated using separating funnel and the volume of biodiesel obtained was recorded. The procedure was repeated for catalyst $A_{2℃}$, B_1, and B_2 [5].

Determination of Flash Point

About 3 ml of the oil was introduced into a cup and closed. A thermometer was attached. The cup was heated. Flame was applied to the surface of the oil and the temperature at which there was an ignition sound was recorded, which was the flash point [15].

Determination of Pour Point

About 5 g of oil was introduced into the conical flask with a thermometer attached and it was heated to about 45 ^{0}C. It was removed from the water bath and allowed to cool down to a decrease of about 3 ^{0}C. The container was tilted to observe the movement of the oil. The temperature at which the oil stopped moving was noted .The pour point was 2.94 ^{0}C higher than the temperature at which the oil stopped moving (ASTM method). [17]

Determination of Acid Value

About 10 g of the oil was measured and dispensed in a conical flask. About 50 ml of ethyl alcohol and 1 ml of phenolphthalein indicator were added. The mixture was heated in a water bath to temperature of about 75 ^{0}C. The burette was filled with alkali solution. The hot solution was titrated against the alkali solution. The end point of the reaction was taken. The burette reading was noted and recorded when the colour changed to dark pink [12].

Mathematically:

$$\text{Acid value} = \frac{56.1 \times V \times N}{W}$$

V= Volume of KOH used in titration
N= normality of KOH
W= weight of the sample in gram

Determination of Free Fatty Acid Content [7]

The FFA content is the acid value divided by 2, gives the free fatty acid content

Hence

$$FFA = \frac{56.1xVxN}{2W}$$

Determination of Water Content

About 3 g of the oil was weighed in to a beaker of known mass. The weight of beaker and oil was measured and recorded W_1. The beaker was heated to a temperature of about 120 ^{0}C. The content was stirred and then heated to aid circulation of the heat. The beaker was then weighed again with the oil and recorded as W_2. The difference between W_1 and W_2 was determined and it was recorded as the weight of water in the sample W_f [14].

$$\text{Water content} = \frac{\text{Oil weight}}{W_f}$$

Determination of Viscosity

The viscosity of the oil was determined manually in the lab by the use of pipette. Some quantity of the oil was pipetted and allowed to flow out freely and the time of flow was recorded per seconds [16].

Determination of Iodine Value

About 0.31 g of the oil was measured and transferred into a flask. About 11 ml of chloroform was measured and transferred into the flask in order for the oil to dissolve. 25 ml of hanus solution was pipetted and transferred to the flask. A stopper was moistened with potassium iodide solution and was inserted into the flask. The mixture was transferred to a dark cupboard for about 30 min. 11 ml of 15 % Potassium iodide was introduced into the mixture and was shaken with high intensity. 100 ml of distilled water was added. The mixture was titrated against 0.1 M of thiosulphate solution until yellow solution was about to turn color less. Starch solution indicator was then added in drops until a colorless solution was obtained, the volume of thiosulphate used was measured and recorded as X. The blank titration was carried out and volume of thiosulphate used was recorded as Y [14].

$$\text{Iodine value} = \frac{(Y\text{-}X)\text{-}N\text{-}12.69}{\text{Weight of oil}}$$

Determination of Density

An empty cylinder was weighed**.** The cylinder was filled with the oil and was measured as M_1. The mass of oil was determined as M_2**.** The volume of the oil was V [14].

$P = M_2/V$

RESULTS AND DISCUSSION

The results of the analysis of the pure oil and biodiesel extracted from Gmelina *arborea* seed are presented in Table 1. The result showed the quantity of biodiesel yield at different conditions and parameters. Gmelina *arborea* seed is a good source of biodiesel which contains about 44.4 % of oil ad shown in the table 1:

Table 1.0: Oil Yield from Gmelina Seed

Mass of seed(g)	50.0	48.0	53.0	60.0
Mass of oil (g)	22.2	21.2	23.43	26.64
Percentage yield (%)	44.4	44.0	44.2	44.4

$$\text{Average \% yield is} = \frac{44.4+44+44.2+44.4}{4} = 44.25\%$$

Trans-esterification of Gmelina *arborea* Seed Oil

The trans-esterification of the oil was carried out under varying conditions and parameters which include oil to methanol ratio 1:6 ,1:9,1:12 and 1: 15,catalyst load (1-5% wt.), temperature of 60 °C – 120 °C, reaction time from 1-4 hrs and also varying different catalyst carbonized at different temperature (A_1,A_2,B_1,B_2). The highest biodiesel yield of 95.41 % was gotten at oil to methanol ratio of 1:9, catalyst load of 3 % wt. when A_1 catalyst was used at a temperature of 60 °C for 2 hours of reaction time. At 5 % catalyst load, using catalyst A_1 with oil to methanol ratio of 1:9 at 60 ° C for 1 hour reaction time , 92.42 % of the biodiesel was yielded but when A_2 which was impregnated and calcined at a lower temperature than A_1 produced a lower yield of 88.27 % which showed that catalyst impregnated at higher temperature leads to higher yield. The result obtained was in agreement with that of Oyedoh et al [5].

Effect of Oil to Methanol Ratio

According to Table. 2. The highest biodiesel yield of 95.41 % was obtained at oil to methanol ratio of 1:9 using A_1 catalyst at a reaction time of 2 hrs. The lowest yield was observed when oil to methanol ratio of 1:15 was used. It was observed that when a lower oil to methanol ratio of 1:6 was used, a lower yield of 75.93 % was observed which was better than when 1;15 was used. There was gradual increase in biodiesel yield from 1:6 to 1:9 but a decrease was observed when the oil to methanol ratio was increased to 1:12 to 1:15.The result showed that the optimum oil to methanol ratio was 1:9.

Table 2: Effect of oil to methanol ratio on biodiesel yield.

Oil : Methanol	1:6	1:9	1:12	1:15
Percentage yield (%)	75.93	92.42	76.63	74.3

Effect of Temperature

As presented in Table 3, the effect of temperature on biodiesel yield was analyzed using oil to methanol ratio of 1:9 for 1 hour with 5 % catalyst load of A_1 catalyst. It was observed that the best yield of 92.42 % occurred at a temperature of 60 ^oC which was lower than the boiling point of alcohol. As the temperature increased to 80 ^oC which was above the boiling point of alcohol, there was a steady decrease in the biodiesel yield. The temperature was further increased to 100 and 120 ^{o} C and this resulted in low biodiesel yield. This steady decrease maybe due to methanol in its vapour form which reduces the interaction between methanol and the oil. This result was in accordance with the result of the study carried out by Muhammad et al [14], which postulated that the reaction temperature must be lower than the boiling point of alcohol to avoid loss of methanol through vaporization from the reaction set up.

Table 3: Effect of temperature on biodiesel yield

Temperature	60 ^oC	80 ^oC	100 ^oC	120 ^oC
% Yield	92.42	80.52	63.30	50.10

Effect of Reaction Time

As shown in Table 4, the effect of reaction time on the yield of biodiesel was analyzed using oil to methanol ratio of 1:9 at a temperature of 60 ^oC. The highest conversion of the oil to biodiesel was observed at reaction time of 2 hours. As the time increases to 3 hours, a decrease in the biodiesel yield was noticed, this might be due to some of the methanol escaping as vapor due to prolonged heating.

Table 4: Effect of reaction time on the biodiesel yield

Time(s)	60	120	180	240
Percentage yield (%)	92.42	92.77	91.36	88.27

Effect of Catalyst Load

The impact of catalyst load on the biodiesel yield was analyzed using oil to methanol ratio of 1:9 and using A_1 catalyst at 60 °C for 2 hours by varying the catalyst amount from 1 % to 5 %. It was observed that 95.41 % of the oil was converted to biodiesel when 3 % of the catalyst was used. There was a gradual decrease in the biodiesel as the catalyst load was increased to 4 %. The optimum catalyst load was 3 % to obtain a maximum yield as presented in Table 5.

Table 5: Effect of catalyst load on biodiesel yield .

Catalyst load	1 %	2 %	3 %	4 %	5 %
Percentage yield (%)	63.49	78.40	95.41	94.17	92.77

Physiochemical Properties of the Biodiesel and Pure Oil

The physiochemical properties of the biodiesel and pure oil was illustrated in Table 6.

Table 6: Physiochemical Properties of the Oil

Physicochemical properties	Pure oil	Biodiesel	Pure diesel
Pour point (°C)	4.94	3.94	-13.1
Flash point (°C)	346	146	60.0
Acid value(mg/KOH/g)	6.34	0.68	0.50
Free fatty acid (mg/KOH/g)	3.17	0.34	
Viscosity (mm^2/s)	15.40	4.80	6.0
Density (kg/m^3)	893.8	1045.9	840.0
Water content (%)	21.6	3.6	0.10

The acid value indicates the free fatty acid present in the oil. The Gmelina seed oil contains about 6.34 (mg/KOH/g) of free fatty acid while the Gmelina seed biodiesel contains 0.68 (mg/KOH/g) of biodiesel which was lower than that of the regular biodiesel which was 0.5 (mg/KOH/g). Low acid value indicates the state where the oil hasn't been degraded and the biodiesel acid value was 0.1 greater than that of pure diesel, it also indicated that the quantity of the triglyceride had been decomposed by the activities of the lipase.

Viscosity is the measure of the resistance of flow of a fluid. The viscosity of the oil was lower than the viscosity of the regular biodiesel and this signifies that the biodiesel was suitable for industrial uses.

The flash point of the biodiesel obtained was 146 ^{o} C which was in accordance with the ASTM D6751 [13].

The density of biodiesel obtained from Gmelina *arborea* seed was greater than that of the pure diesel which was in the range of ASTM specification.

The pour point of biodiesel from Gmelina *arbohea* seed was relatively higher than the pour point of pure diesel; this implies that the biodiesel from Gmelina seed could lose its ability to flow before pure diesel. The pour point of the biodiesel obtained was 3.94 ^{o}C and this simply showed that if the environmental condition was below this temperature, the biodiesel may not be suitable for application.

Gmelina *arborea* seed oil, as a non-food feedstock, was transformed into biodiesel with a high yield of 89.4%. Additionally, the biodiesel has good fuel parameters, and the biodiesel produced from fresh zeolite gave the higher flash point, lower viscosity and high cetane number than for Gmelina *arborea* oil methyl esters [7]. The acid value of Gmelina *arborea* seed oil was greater than that of Shea butter oil, whereas the acid value was greater than 11.64 mgKOH/g. It was also higher than that of avocado seed oil at 5.00 mg/g. The density of oil was 0.91 for GSO and 0.93 for SB, Oil with low density is an indication that it contains low molecular weight fatty acid. Likewise, it will have a high saponification value which makes it suitable for soap production [7].

Biodiesel, fatty acid, benzene derivatives, furfural and its derivatives were produced from calcium oxide catalyzed hydrolysis of Gmelina *arborea* dead leaves. Approximately 77.84 g of biodiesel was produced from 50 g of Gmelina *arborea* dead leaves which yielded 155.68%, the highest selectivity ever in biodiesel process technology. This process technology converts wastes to fuel and industrial chemicals which is capable of generating energy, employment and empower rural dwellers, regenerates our forests and cleanse our environment [5]. High-quality biodiesel with 93% yield was obtained under optimal reaction conditions with the ratio of oil to methanol (1:9) mol, amount of catalyst (1.5% by weight of oil), at temperature (60 °C), and reaction time of 2 h [11].

FTIR Characterization of Catalyst A_1

As presented in Figure 1, the FTIR spectra of catalyst A_1 indicated an intense peak around 1992.48 cm^{-1} which indicates the presence of C=C stretching of allenes. There was a medium peak at 3095.54 cm^{-1} which indicates the stretching of OH of carboxylic acid.

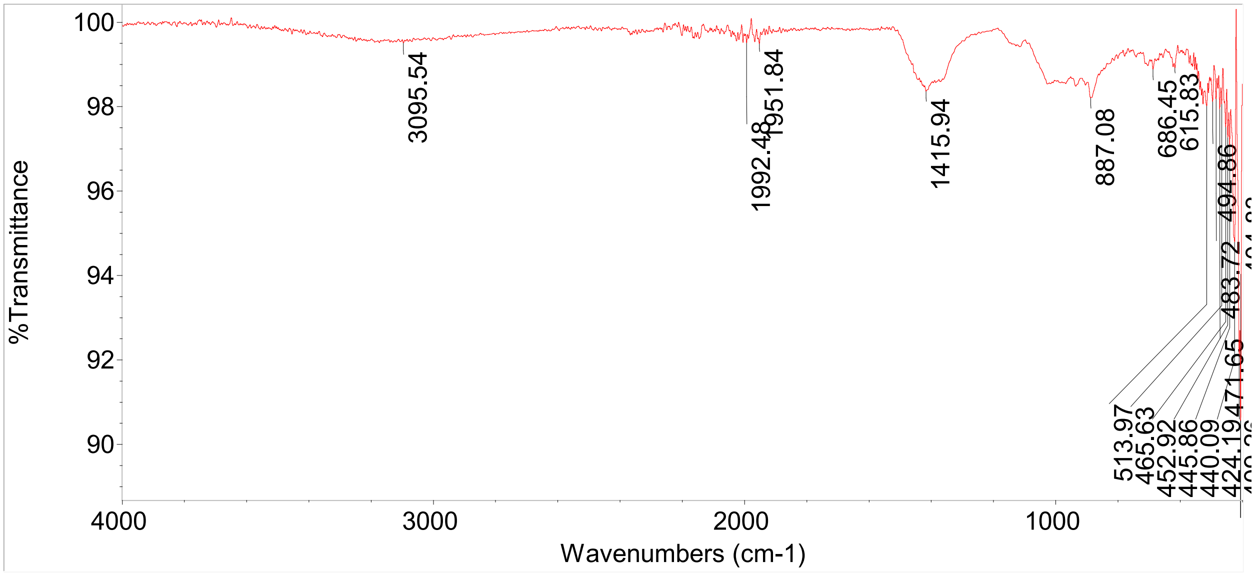

Figure 1. FTIR Spectrum of Catalyst A_1

FTIR Characterization of Catalyst A_2

According to Figure 2, the FTIR spectra of catalyst A_2 showed a strong peak at 1738.10 cm^{-1} which indicated C=O stretching of an aldehyde. The spectra showed a medium peak at 2969.83 cm^{-1} which indicated the presence of C-H stretching of methyl group.

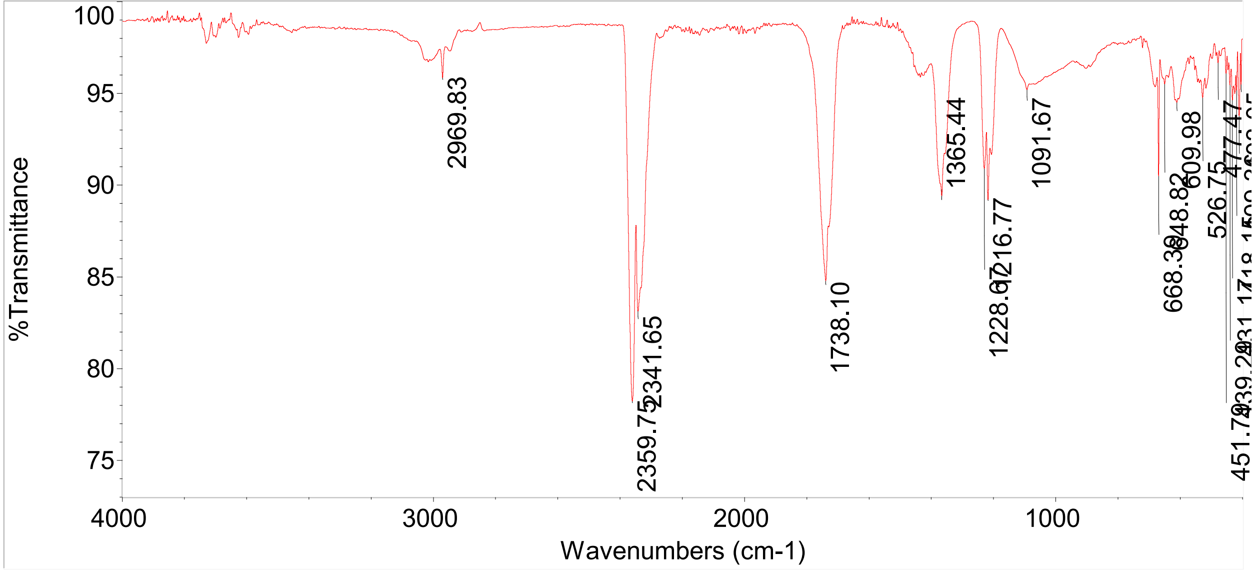

Figure 2. FTIR Spectrum of Catalyst A2

FTIR Characterization of Catalyst B_1

The FTIR spectra of catalyst B_1 showed that there was a slightly intense peak at 1738.20 cm^{-1} which indicated the stretching C=O of an aldehyde according to Figure 3.

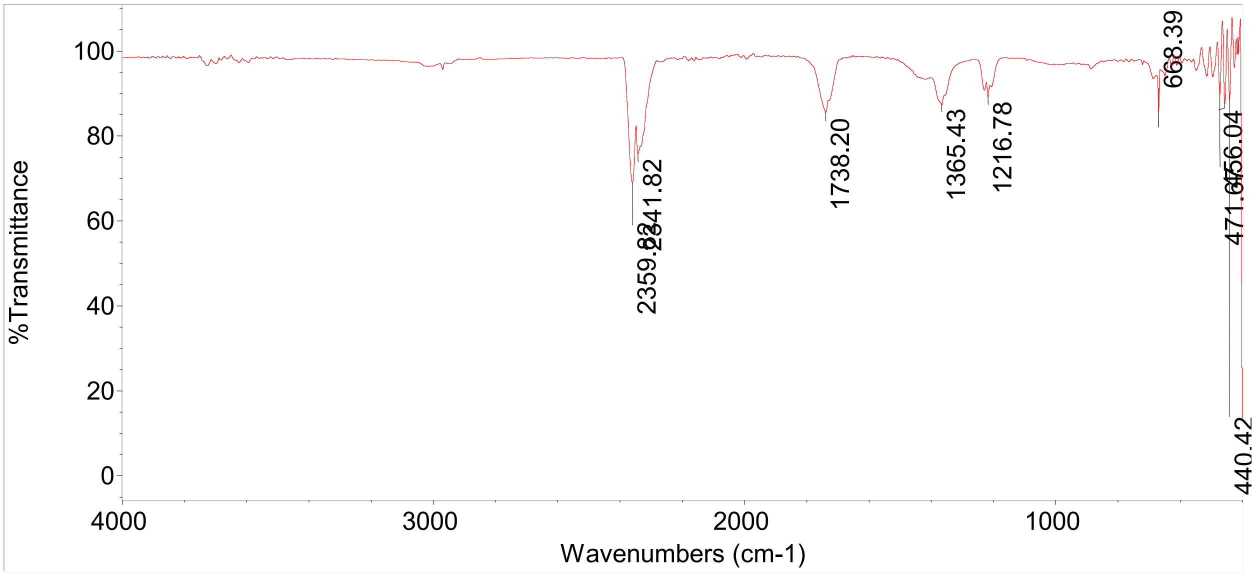

Figure 3. FTIR spectrum of Catalyst B_1

FTIR Characterization of Catalyst B_2

FTIR spectra of catalyst B_2 showed sharp peak at 2173 cm^{-1} which indicated C≡C stretching of alkyne. There was a sharp peak at 1951 cm^{-1} which indicates C=C stretching of ketones and also a peak at 3008 cm^{-1} indicating C-H stretching of aromatics as presented in Figure 4.

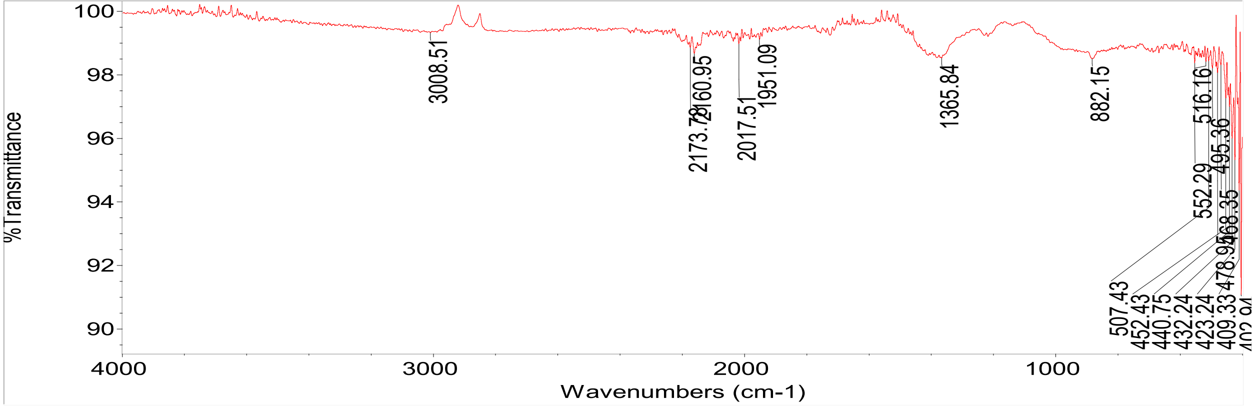

Figure 4. FTIR spectrum of Catalyst B_2

Comparison of the Catalyst Samples

The highest biodiesel yield of 92.42 % was produced when catalyst A_1 was used at oil to methanol ratio of 1:9 and a reaction time of 1 hour at a temperature of 60 ^{o} C. This might be due to the present of the carboxylic acid as indicated by the IR peak at 3059 cm^{-1} and made it a strong acid catalyst. Catalyst A_2 had the lowest yield of biodiesel 88.27 %. Catalyst B_1 produced a slightly higher yield of 90.91 % and catalyst B_2 yielded 91.09 % of biodiesel. The difference in the effectiveness of these catalysts might be due to the fact that each of these catalysts was carbonized and impregnated at different temperatures. Catalyst A_1 was carbonized at 700 ^{o}C and impregnated at 600 ^{o}C while A_2 was carbonized at 700 ^{o}C and impregnated at 500 ^{o}C. The lower impregnation temperature of A_2 might be the reason for the lower yield of biodiesel as brought about by A_2. Catalyst B_1 and B_2 were carbonized at 600 ^{o}C but B_1 was impregnated at a temperature of 700 ^{o}C and B_2 was impregnated at a temperature of 500 ^{o}C. Catalyst B_1 was resulted in a higher yield and this might be due to the higher impregnation temperature of B_1.

CONCLUSIONS

This study focused on the production of biodiesel from Gmelina *arborea* seed using trans-esterification method. Banana peel was used to prepare the catalyst. It was deduced from this study that the Oil to methanol ratio of 1:9, reaction time of 2 hours, catalyst load of 3% and the temperature of 60 ^{0}C are the optimum conditions for the extraction of biodiesel from Gmelina *arbohea* seed. Thus biodiesel produced from Gmelina *arborea* could be a good alternative source for many industrial chemicals, fuels that are currently being sourced from petroleum which is non-renewable and negatively affects the environment and ecosystem during the process of exploration and application.

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