Determination of Optimum Conditions for the Production of Biodiesel from *Gmelina arborea* Seed Oil and Shea Butter Using Zeolite as Heterogeneous Catalyst

By

Alkairu Hassan*, Abdulrahman A. Audu*, Bishir Usman*, Ahmed A. Aliyu*, Dini Sabo*,
Said H. Musa***, Daniel Eric**, Adam S. Alhassan***

Abstract

Biodiesel quality and yield are affected by several processes. The study aimed to optimize the operating variables such as the effect of different zeolites, various concentrations of KOH support loaded during activation of zeolite, heterogeneous, and homogenous catalysts. Heterogeneous catalyst concentration (5wt % to oil mass), temperature (70°C), reaction time (7 h), and reaction speed (600rpm) were all fixed during the reaction while for homogenous catalyst NaOH concentration used was (1wt % to oil mass) and reaction time was one hour, mixing intensity and temperature condition were the same for zeolite. Biodiesel was synthesized by transesterification of Gmelina arborea seed oil and Sheabutter with methanol using KOH/Zeolite and NaOH. Biodiesel from both Shea butter and Gmelina arborea seed oil has high flash points, high cetane numbers,and low viscosity. Gmelina arborea seed oil as a non-food feedstock was transformed into biodiesel with a high yield of 89.4 % and the same conditions applied to Shea butter oil with a high yield of 95.8 %. Also, both biodiesels have good fuel parameters, and the biodiesel produced from fresh zeolite gave the highest flash point, lowest viscosity for Gmelina arborea oil methyl esters, and high cetane number and low viscosity for Shea butter methyl esters.

Keywords: Biodiesel, transesterification, *Gmelina arborea,* Cetane number, Zeolite,Catalyst, Fatty acid methyl esters

Introduction

The non-renewable nature of fossil fuel (petro-diesel) coupled with its environmental pollution, suggested the need to find alternative sources of energy such as biodiesel, especially from non-edible vegetable oils. Therefore the need to research the preparation and determination of optimum parameters for the production of biodiesel from non-edible seed.

Due to the increasing applications of vegetable oils, several seed oils have been characterized, but no research has been carried out on the production of biodiesel from *Gmelina arborea* oil using zeolite as a catalyst.

Gmelina arborea is a big forest tree popular for its use in making furniture and as building materials. *Gmelina arborea* is a fast-growing tree that grows to about 40 cm in height; the tree is commonly planted as a garden and an avenue tree. The fruit contains very little kernel, but the kernel is quite rich in oil.[1]

*****Department of Pure and Industrial Chemistry, Bayero University, Kano, Nigeria.
Corresponding author: Alkairu Hassan; Email: alkhairuh@gmail.com
******Richflood Laboratories Ltd, Abuja, Nigeria.
*******Centre for Renewable Energy Research, Bayero University, Kano, Nigeria.

Gmelina arborea grows in different localities and prefers the most fertile valleys with 750-5000 mm rainfall. It attains moderate to large heights up to 40 cm and 140 cm in diameter. It grows naturally in India, Thailand, Vietnam, Sierra Leone, and Nigeria.[1] Flowering takes place from February to April, whereas fruiting starts from May onwards up to June.[2] The fruit is up to 2.5cm long, smooth, dark green, turning yellow when ripe, and has a fruity smell.[1]*Gmelina arborea*seeds with woody shells contain little amount of kernel (about 7g kernel from 70g seeds, 10 %) but the kernel is quite rich in oil (53.38 % crude oil). Oleic acid is the major fatty acid followed by gondoic acid and palmitic acid in *Gmelina arborea* seed oil and this comprises about 60.83 % of unsaturated and 39.13 % of saturated fatty acids.[1]

Another promising feedstock for biodiesel production is Shea butter from the Shea nut tree (*Vitellaria paradoxa*/*Butyrospermum parkii*). It grows naturally and commonly in Africa, especially in Nigeria. It is less expensive in its raw state. The properties of biodiesel depend on the physicochemical properties of the feedstock. Shea butter comprises mainly triglyceridesand unsaponifiable materials that consist of 40-55 % fat per kernel.[3]

The study of biodiesel fuel is very timely due to the rising cost of fuel in the market, global warming, and health problems such as respiratory diseases caused by harmful byproducts of burning petroleum-based fuels. Another problem concerning the use of diesel is the deteriorating effects of the increased amount of greenhouse gases in the atmosphere. This is due to the high emission of carbon monoxide coming from incomplete combustion of diesel fuel in vehicles. In an attempt to minimize these problems, previous studies generated biofuel from a large number of fats and oils, however, there is a paucity of data on the generation of biodiesel from *Gmelina arborea* biodiesel using zeolite catalyst.

Biodiesel has received considerable attention because of its renewable and environmentally friendly properties.[4] Biodiesel chemically known as fatty acid methyl esters (FAMEs) is prepared by transesterification reaction in which a triglyceride or vegetable oil/fat reacts with an alcohol, usually methanol, in the presence of an alkaline catalyst.[5] [6] [7] [8]

---

[1] Basumatary, S., Barua, P., and Deka, D. C. (2014). *Gmelina arborea* and Tabernae Montana Varicate Seed Oils as non-edible feedstocks for biodiesel production. International Journal ChemTech Research, 6(2), 1440-1445.

[2] Ofomatah, A. C., Ezema, C. G., and Idoko, C. (2015). Production and analyses of Biodiesel from *Gmelina* Seed Oil. Nigerian Journal of Solar Energy, 26:135-139.

[3] Enweremadu, C. C., Rutto, H. L., and Peleowo, N. (2011). Performance evaluation of a diesel enginefueled with methyl ester of Shea butter. World Academy of Science, Engineering, and Technology, 79:142-6.

[4] Rashid, U., Anwar, F., and Knothe, G. (2009). Evaluation of biodiesel obtained from cottonseed oil. Fuel Processing Technology, 90(9):1157-63.

[5] Meher, L. C., Vidya, S. S., and Dharmagadda, S. N. N. (2006). Optimization of Alkali-catalyzed transesterification of Pongamia pinnata oil Production of Biodiesel. Bioresource Technology, 97(12):1392-7.

[6] Cerveró, J. M., Coca, J., and Luque, S. (2008). Production of biodiesel from vegetable oils. Grasas Y Aceites, 59(1): 76-83.

[7] Rashid, U., Anwar, F., and Knothe, G. (2011). Biodiesel Production from Milo (Thespesia populnea) seed oil. Biomass and Bioenergy, 35(9):4034-9.

[8] Li, Y., Zhu, H. G. Z., Feng, Y., and Li, S. (2012). Ethylene glycol monomethyl ether cotton seed oil monoester: Properties evaluation as biofuel. International Journal of Green Energy, 9(4):376-87.

The transesterification process under an alkali catalyst and short-chain alcohol gives a high level of conversion.[9] Different reaction parameters that mostly affect the yield and quality of biodiesel during transesterification are catalysts, concentration, oil/alcohol molar ratio, temperature, time, and stirring speed.[9,10] Rashid and Anwar (2008) reported that KOH is a more effective catalyst with 1 % wt. compared to NaOH, $NaOCH_3$, and $KOCH_3$, which showed almost 98 % of conversion yield of rapeseed oil at a 6:1 methanol/oil molar ratio, reaction temperature of 65^oC, mixing intensity of 600 rpm, and a reaction time of 2 h. Also, Rashid and Anwar (2008) reported a higher yield (98 %) of safflower oil methyl esters with methoxide catalysts at 60 ^oC a methanol/oil ratio of 6:1, and a catalyst concentration of 1 % wt.[9]In contrast,Anwar and coworkers observed that $NaOCH_3$ was a better catalyst in the transesterification of okra seed oil, which showed 96.8 % of biodiesel yield.[10]

Materials and Methods

Sample Sourcing and Preparation

Gmelina fruits were collected from Bayero University Kano, Gandu, and Aminu Kano Teaching Hospital all in Kano. It was soaked in water for eight days to easily separate the fruit pulp from the seed (de-pulp). The seeds were dried at room temperature and crushed using an aluminum mortar and pestle. The organic solvent used for the oil extraction was n-hexane.[11]

Physicochemical Analysis of *Gmelina* Oil and Shea Butter

Determination of Acid Value of the Oil

A 2.80 g of oil was weighed in a 250 mL conical flask and dissolved with 50 mL of ethanol, 5 drops of phenolphthalein indicator were added and the solution was titrated with 0.10 eq/dm^{3}solution, the endpoint was the pink color.[12] Using the equation below the acid value was calculated.

$$A\text{cid Value} = \frac{56.1 \times \mathrm{N} \times \mathrm{T}}{\mathrm{M}} - - - - - - \quad (1)$$

Where,

56.1= Molar Mass of KOH

N= Normality of KOH

T= Titre value

M= Mass of oil used (g)

---

[9] Rashid, U., and Anwar., F. (2008). Production of biodiesel through optimized alkaline-catalyzed transesterification of rapeseed oil. Fuel, 87(3):265-73.-

[10] Anwar, F., Rashid, U., Ashraf, M., and Nadeem, M. (2010). Okra (Hibiscuses culentus) seed oil for biodiesel production. Applied Energy, 87(3):779-85.

[11] Rashid, U., Ibrahim, M., Ali, S., Adil, M., Hina, S., and Yunus, R. (2012). Comparative Study of Methanolysis and Ethanolysisof Maize Oil Using Alkali Catalysts. Grasas Y Aceites, 63(1):35-43.

[12] Ahmad, M., Zafar, M., Sadia, H., Sultana, S., Arshad, M., and Irfan, M. (2013). Physico-chemical Characterization of Sunflower Biodiesel Oil by Using Base Catalyzed Transesterification. International Journal of Green Energy, 2(12):23-25.

Determination of Free Fatty Acid

The free fatty acid was determined from the results of the acid value using the FFA equation.[12]

$$FFA = \frac{AcidValue}{2} - - - - - - - - (2)$$

Determination of Specific Gravity

The determination was done using a specific gravity bottle. Specific gravity was calculated using the formula.[12]

$$Specificgravity = \frac{c-a}{b-a} - - - - (3)$$

Where,

Weight ofempty density bottle = a

Weight of density bottle + distilled water= b

Weight of density bottle + oil = c

Determination of Moisture Content

The *Gmelina* oil sample (5g) was weighed in a crucible and placed in an oven at 110^{o}C, and set for an hour, removed, and re-weighed until a constant weight is obtained.[13]

$$\%\,Moisture\ content = \frac{W2\ - W3}{W2\ - W1} \times 100 - -(4)$$

Where,

W1 = mass of empty crucible

W2 = mass of crucible with sample before heating

W3 = mass of crucible with sample after heating

Determination of Saponification Value

2 g of oil sample was taken into a 250 cm^3 conical flask. About 25 cm^3 of alcoholic KOH was added and heated for 30 minutes with occasional shaking until the solution became homogenous and cooled under tap water. The sample was titrated against 0.50 N HCl with 0.5 cm^3 phenolphthalein indicator. A blank titration was carried out without oil.[14] The following formula was used to determine the saponification value.

---

[13] Alptekin, E., Canakci, M. (2008). Determination of the density and the viscosities of biodiesel-diesel fuel blends. Renewable Energy, 33(12):2623-30.

$$SaponificationValue = \frac{(B - S) \times N \times 56.1}{W} - -(5)$$

Where,

B= volume(cm^3) of HCl required by blank

S= volume(cm^3) of HCl required by the oil sample

N= Normality of HCl, W= Weight of oil

Determination of Iodine Value

A 0.30 g of oil was weighed in a 500 cm^3 conical flask and 10 cm^3 of chloroform was added. 25 cm^3 Hanus solution was added, mixed vigorously, and kept in a dark place for 30 minutes. After that 10.00 cm^3, 15% KI solution,and 100 cm^3 distilled water was added and mixed thoroughly. The solution was titrated against standard 0.1N $Na_2S_2O_3$solution with continuous shaking until the dark color almost disappeared. Titration was continued by adding 1 cm^3 starch solution till the color had fully vanished. The blank determination was also conducted in the same way without a sample.[14]

$$\text{Iodine value} = \frac{(B - S) \times N \times 12.69}{W} - - - - - - - - (6)$$

Where,

B= volume(cm^3) of $Na_2S_2O_3$ required by blank
S= volume(cm^3) required by the oil sample
N= Normality of $Na_2S_2O_3$, W= Weight of oil

Deacidification of Crude Gmelina arborea Seed Oil and Shea Butter

80 g of fat /oil was mixed intensively with 91 g of methanol and 10 g of ethanolamine. The extraction was carried out while stirring vigorously for 15 minutes at 60 ^oC. The resultant mixture was allowed to separate overnight and the phases so obtained were subsequently withdrawn and analyzed.[15]

Activation of Zeolite Catalmyst

The zeolite was calcined at 550^{o}C for 3 h. To modify zeolite with different amounts of KOH solution loaded from (15-50 %), zeolite was impregnated with an aqueous solution of KOH. It was dried at 110^{o}C for 24 h, before use as a catalyst in the reaction.[16]

---

[14] Quader, F., Shaokat, M A., Ahmed, S., Irfan, A. F. M., Roy, S., and Ahmed, S. (2018). Qualitative assessment of common edible oils available in Bangladesh. Asian-Australas Journal Biotechnology, 3(2):156-161.

[15] Hassan, A., Audu, A. A., Usman, B., Aliyu, A. A., Sabo, D., Musa, S.H., Eric, D., and Alhassan, A.S. Determination of Optimum Conditions for the Production of Biodiesel From Gmelina arborea Seed Oil and Shea Butter Using Zeolite as Heterogenous Catalyst, Msc Thesis Submitted to the Department of pure and Industrial Chemistry, Bayero University, Kano.

[16] Pisitpong, I., Sotsanan, I., Pitchaya, P., Apanee, L., and Samai, J. (2013). The activity of basic properties of KOH/Mordenite for transesterification of Palm oil, Journal of Energy Chemistry, 22:690-700.

Production of Fatty Acid Methyl Ester (Biodiesel)

Before the initiation of the reaction, a weighed mass (30 g) of *Gmelina arborea* seed oil and Shea butter was preheated at 100^0C using a hot plate. The methanol/oil molar ratio was calculated as *Gmelina arborea* seed oil and Shea butter (30g, 9:1 molar ratio) and methanol (9:1 molar ratio), which were then added to the reaction flask. Followed by a calculated amount of freshly synthesized catalyst, zeolite/KOH. Each experiment was continued for 7h to ensure the complete conversion of the *Gmelina arborea* oil into biodiesel (fatty acid methyl ester, FAME). After the reaction was complete, the product mixture was brought to room temperature by cooling and allowed to equilibrate for separation of the two layers/phases. The upper phase comprised of FAMEs with minor contents of some impurities such as glycerol, partial glycerides, and residual alcohol. The lower phase mainly consisted of a valuable byproduct (glycerol/glycerin) along with traces of remaining components such as excess methanol, soaps formed during the reaction, catalyst, some methyl esters, and partial glycerides. Both phases were recovered. The upper biodiesel phase was purified by washing with distilled water until the clear phase of biodiesel was obtained. Pure biodiesel was obtained after drying it.

Results

Table 1 shows some physicochemical parameters of *Gmelina* Seed Oil (GSO) and Shea Butter (SB). The levels of the acid value, iodine number, saponification value, and moisture content in GSO were comparatively higher than those for SB.

Table 1: Some Physiochemical parameters of GSO and SB compared to ASTM

Properties	***Gmelina* seed oil**	**Shea butter**	**ASTM**
Moisture Content (%)	2.26 ± 0.03	0.56 ± 0.01	<0.5
Saponification value (mg KOH/g)	196.1 ± 0.21	172.60 ± 0.56	No specification
Density (g/cm^3)	0.93 ± 0.01	0.93 ± 0.03	0.87-0.90
Acid Value(mg KOH/g)	23.00 ± 0.23	13.00 ± 0.28	<1.00
Free fatty acid (FFA)	11.5 ± 0.12	6.50 ± 0.07	<0.50
Iodine value(mg I_2/100 g fats)	32.70 ± 0.98	7.91 ± 0.01	No specification

Values are mean ± Standard deviation (n=2)

Table 2: Some Physiochemical Analysis of De-Acidified GSO and SB

Properties	**First Esterified Oil**	**Second Esterified Oil**
Acid Value of GSO (Mg KOH/g)	2.45 ± 0.015	0.92 ± 0.01
Free fatty Acid Value of GSO (Mg KOH/g)	1.22 ± 0.021	0.46 ± 0.01
Acid Value of SB (mg KOH/g)	1.98 ± 0.028	1.00 ± 0.021
Free Fatty Acid Value of SB (Mg KOH/g)	0.99 ± 0.01	0.50 ± 0.02

Values are mean ± Standard deviation (n=2)

The value obtained in this study was compared with the American Society for Testing and Materials (ASTM) values before transesterification. Both the two oils had high FFA and also GSO and SB have moisture above the ranges of ASTM. Therefore, the oil had to be treated before the transesterification reaction. High FFA value and high moisture content cause a lot of challenges during transesterification. Some of the problems are: complicating the mixture of biodiesel and glycerin, and also more soap will be formed which can cause difficulty in separation and washing.

From Table 2, it was observed that the acid values obtained from both crude *Gmelina* oil and Shea butter were reduced by the esterification reaction twice.

It was observed from Figures 1, 2, 4 and 5 that fresh zeolite catalysts are more effective in biodiesel yield than used zeolite. There is a significant increase in yield for FAME produced from fresh zeolite and used zeolite for GOME while for SBME. The increase in yield is not significant as a p-value greater than 0.05. It was observed from Figures 2 and 4 that fresh zeolite catalysts are more effective in the yield of GOME and SBME than sodium hydroxide and used zeolite. There is no significant increase in yield for FAME produced from fresh zeolite and sodium hydroxide for both GOME and SBME as a p-value greater than 0.05.

It was observed from Figures 3 and 6 that an increase in the concentration of KOH during the activation of the zeolite catalyst increased the yield of GOME and SBME. Increased KOH concentration above 25 % during activation of zeolite, decreased GOME and SBME yield. There is a significant increase in FAME produced from 15 % to 25 % KOH/zeolite for both GOME and SBMEas$P<0.05$.

.

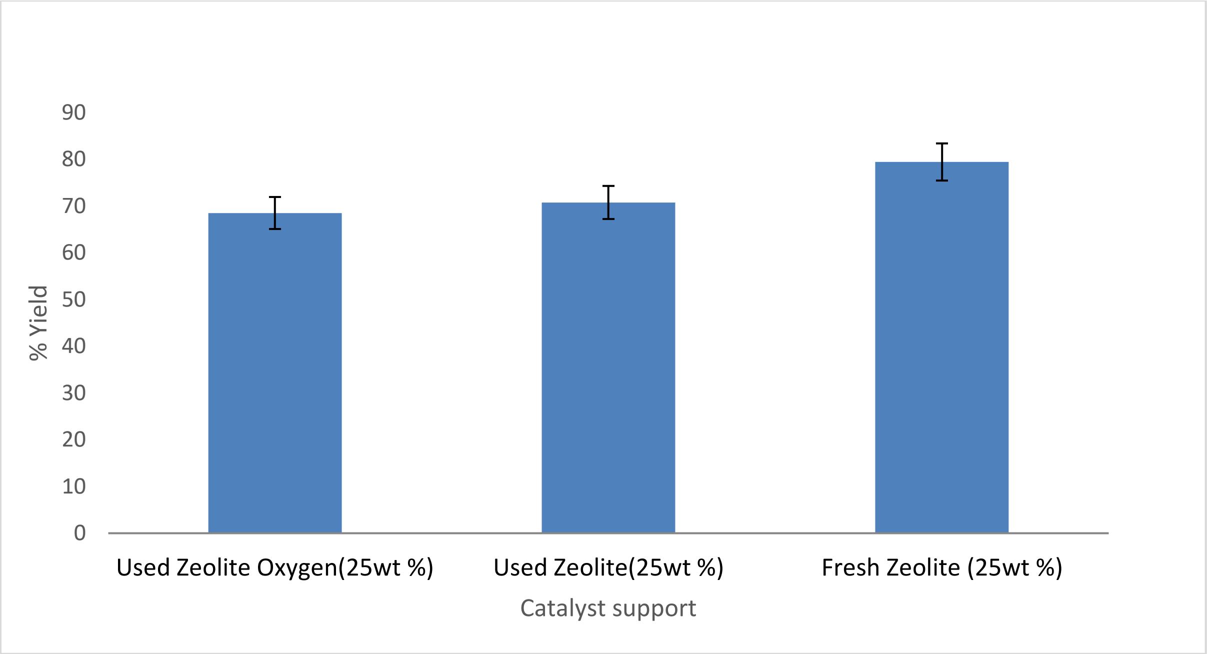

Figure 1: Effect of different zeolite support on GOME yield, temperature:70^{0}C, zeolite %: 5wt %, time: 7hours

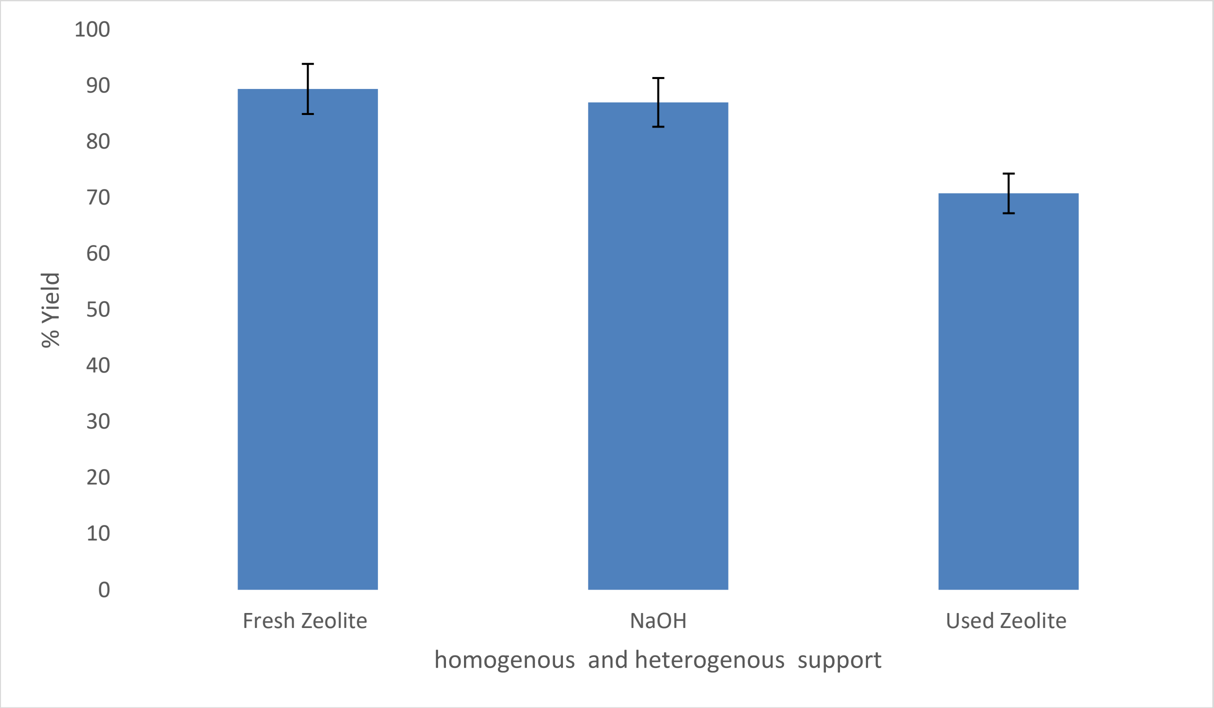

Figure 2: Effect of homogenous and heterogeneous support on GOME yield temperature:70^{o}C, zeolite %: 5wt %, NaOH%: 1wt %, time: 7 hours and 1 hour

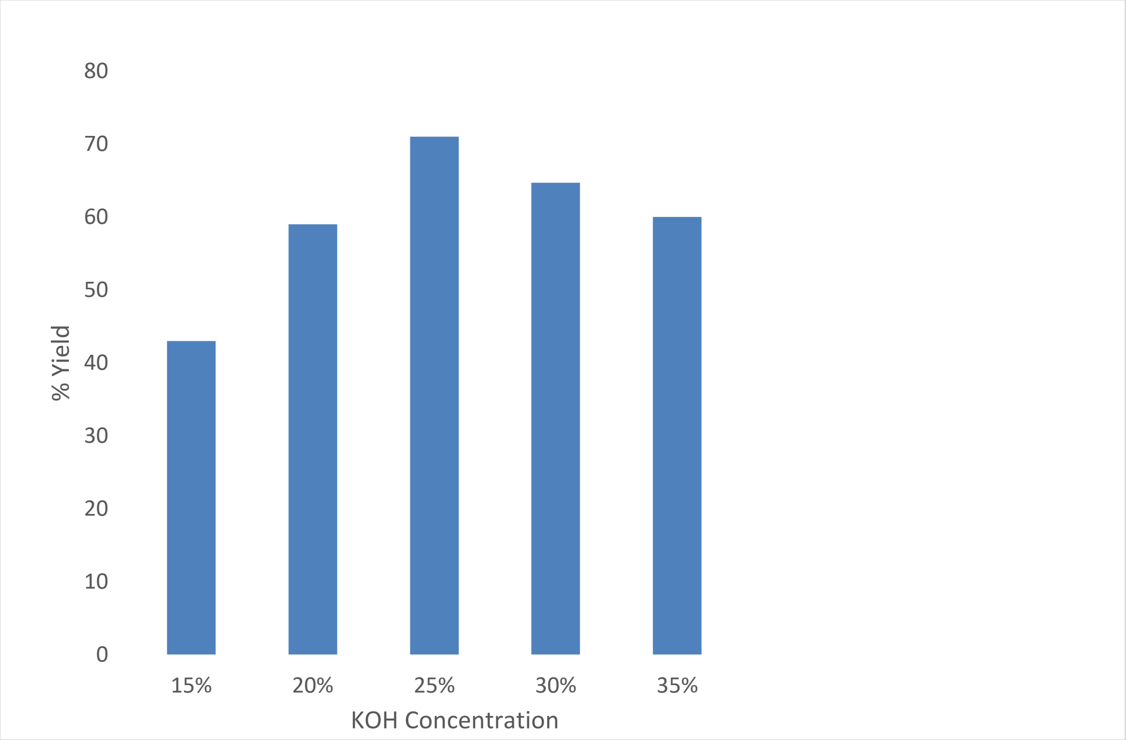

Figure 3: Effects of varied concentrations of KOH solution loaded in used zeolite on GOME yield, temperature:70^{0}C, zeolite: 5wt %, time: 7hours

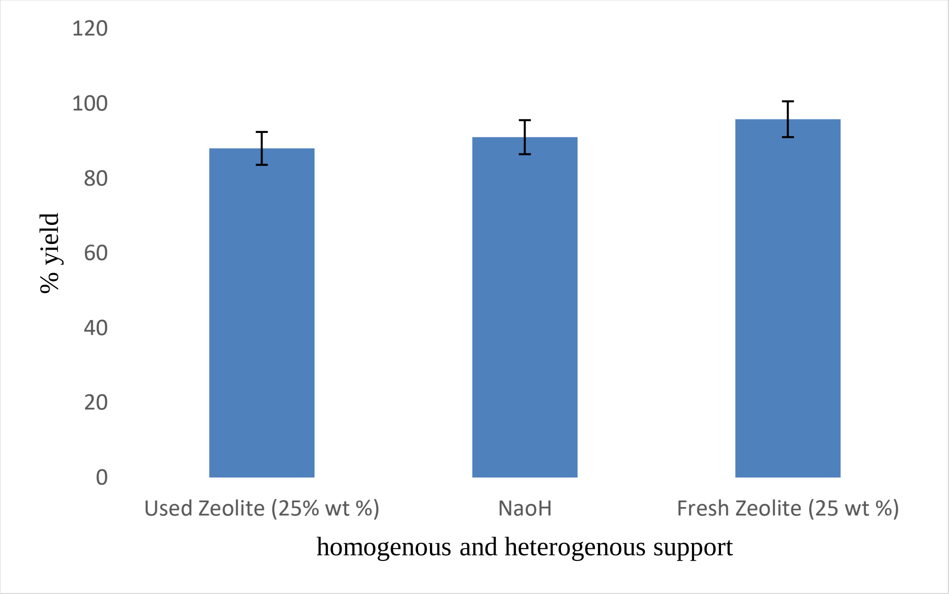

Figure 4: Effect of homogenous and Heterogenous support on SBME yield

Temperature:70^{0}C, zeolite : 5wt %, NaOH :1 %: 1wt %, time: 7 hours and 1 hour

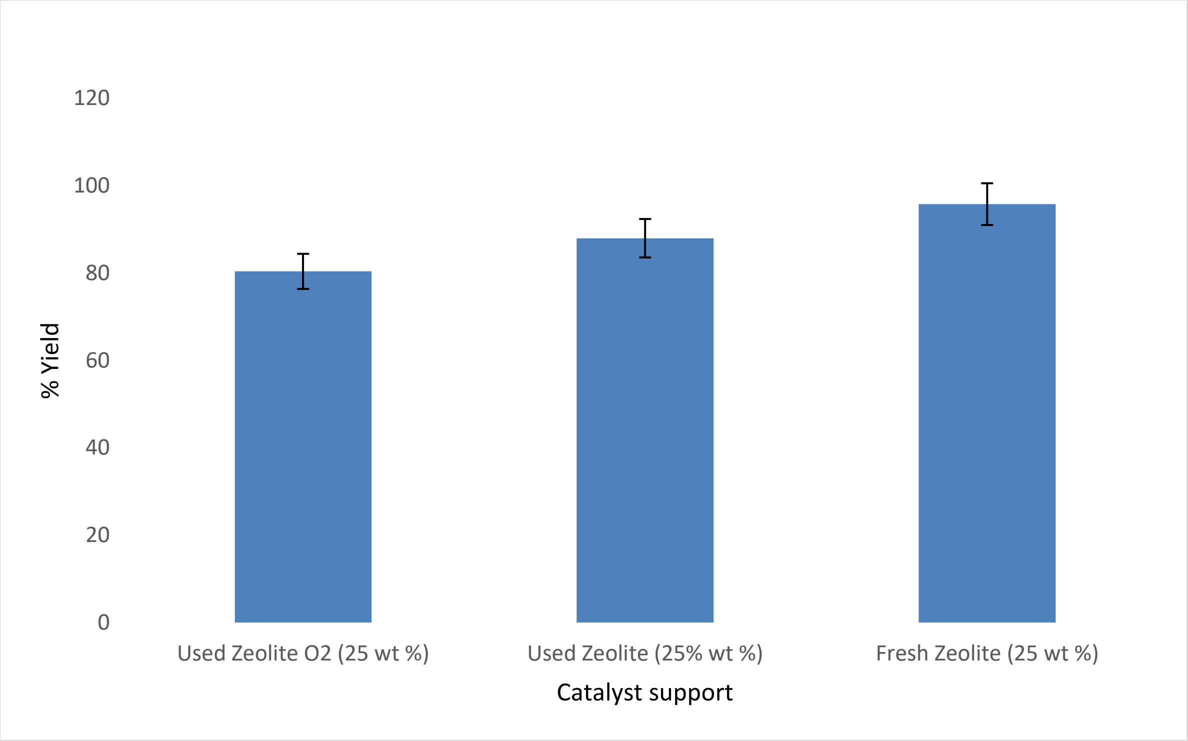

Figure 5: Effect of Zeolite catalyst on SBME yield, temperature:70^0C, zeolite: 5wt %, time: 7hours

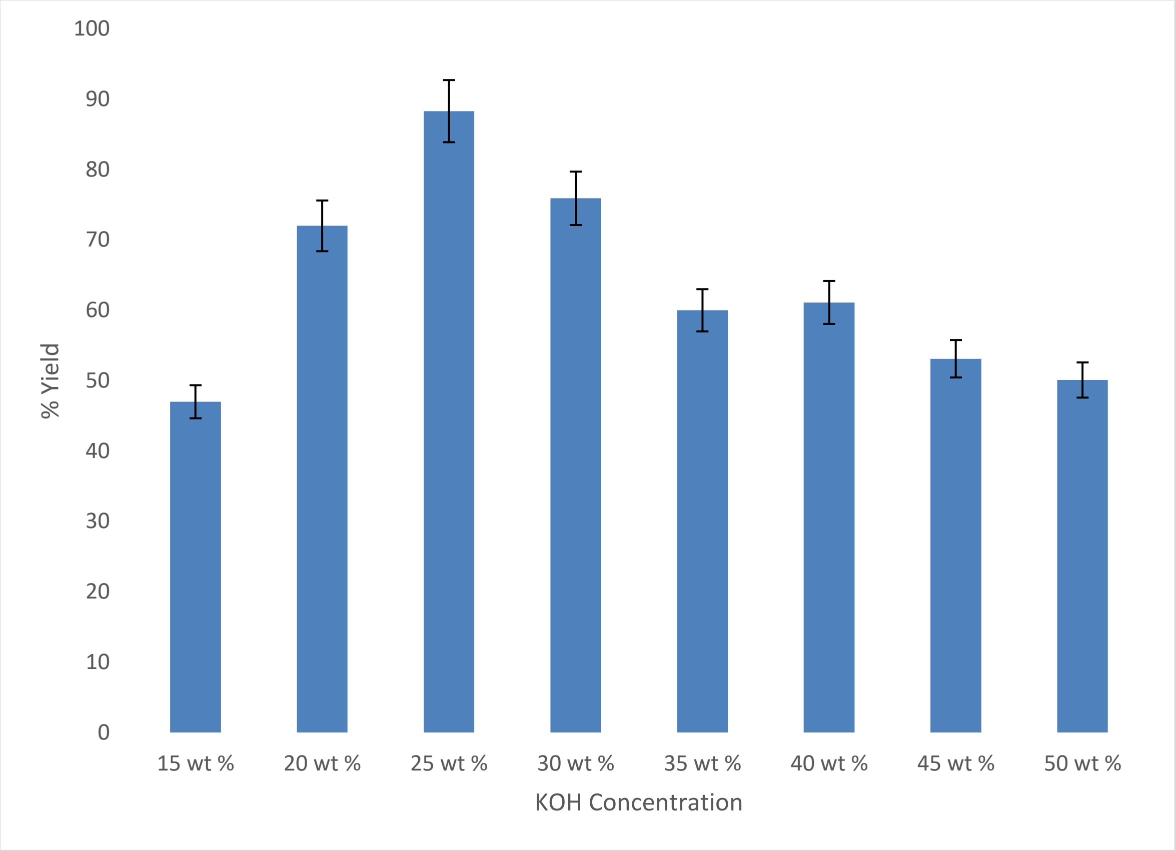

Figure 6: Effects of varied concentrations of KOH solution loaded in used catalyst on SBME yield, temperature:70^{0}C, zeolite: 5wt %, time: 7hours

From Tables 3 and 4, biodiesel from both *Gmelina arborea* seed oil and Shea butter has good quality fuel parameters such as high flash point, good cetane number, and low viscosity. However, biodiesel from both Shea butter and *Gmelina* seed oil hasa high cloud point and pour point.

Table 3: Biodiesel Fuel Parameters (GOME)

Parameters	**Fresh Zeolite**	**NaOH**	**Used Zeolite**	**ASTM**
Viscosity(mm^{2}S^{-1})	2.09 ± 0.01	2.46 ± 0.03	3.01 ± 0.01	1.9-6.00
Cetane number	50.00 ± 0.71	54.00 ± 0.71	53.00 ± 1.41	48-65
Flash point(^{0}C)	155.00 ± 0.70	147.00 ± 0.71	139.0 ± 0.71	100-170
Pour point(^{0}C)	4.10 ± 0.14	3.50 ± 0.14	2.00 ± 0.01	-15-10
Cloud point(^{0}C)	5.00 ± 0.02	3.50 ± 0.01	4.60 ± 0.10	-6-12

Table 4: Biodiesel fuel parameters of SBME

Parameters	**Fresh Zeolite**	**NaOH**	**Used Zeolite**	**ASTM**
Viscosity(mm^2/S)	2.67 ± 0.21	2.97 ± 0.071	3.00 ± 0.16	1.9-6
Cetane number	60.00 ± 0.71	57.00 ± 0.03	59.00 ±2.82	48-65
Flash point(^{0}C)	147.00 ± 0.01	151.00 ± 0.70	158.0 ± 0.71	100-170
Pour point(^{0}C)	4.12 ± 0.16	3.57 ± 0.35	3.25 ± 0.15	-15-10
Cloud point(^{0}C)	5.00 ± 0.04	3.50 ± 0.14	6.75 ± 0.11	-6-12

Values are mean ± standard deviation (n=2)

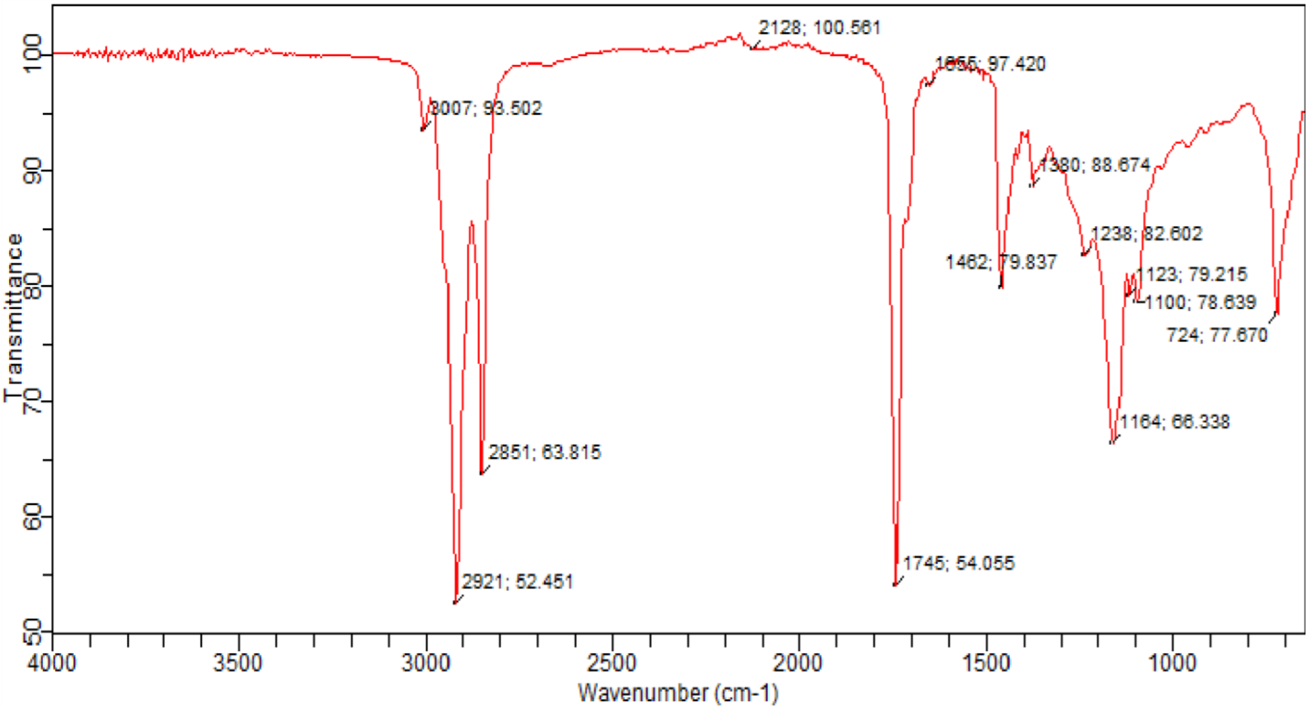

Figure 7: FTIR of crude *Gmelina arborea* seed oil

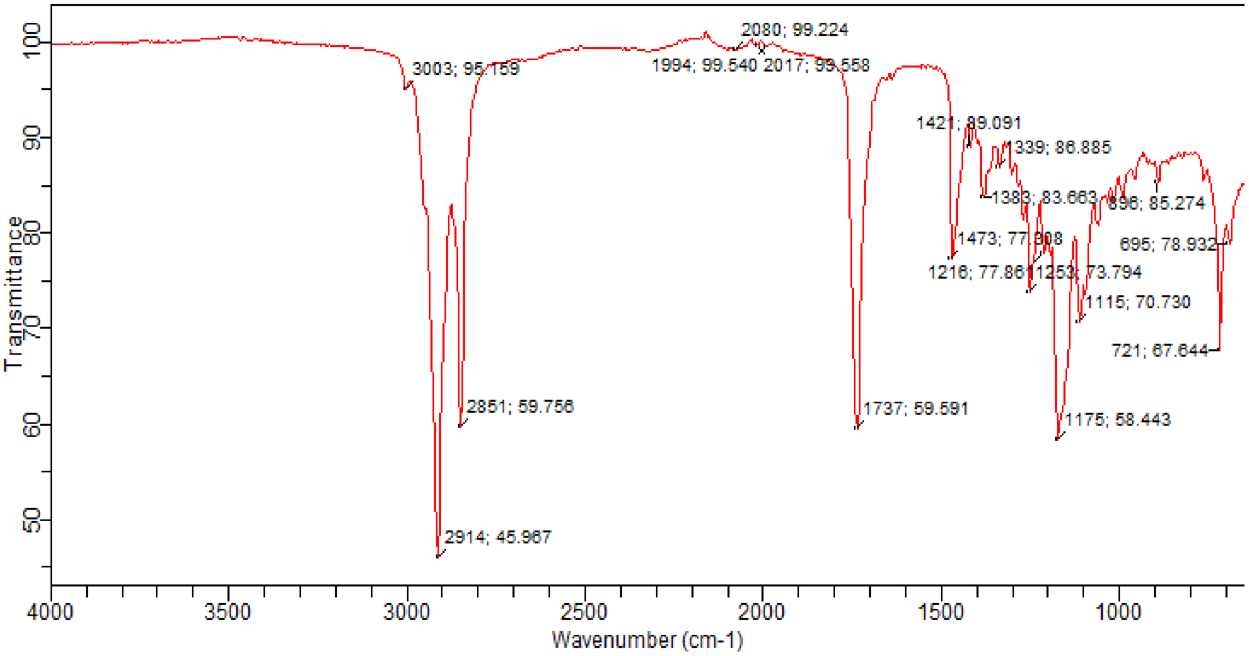

Figure 8: FTIR of crude *Shea butter*

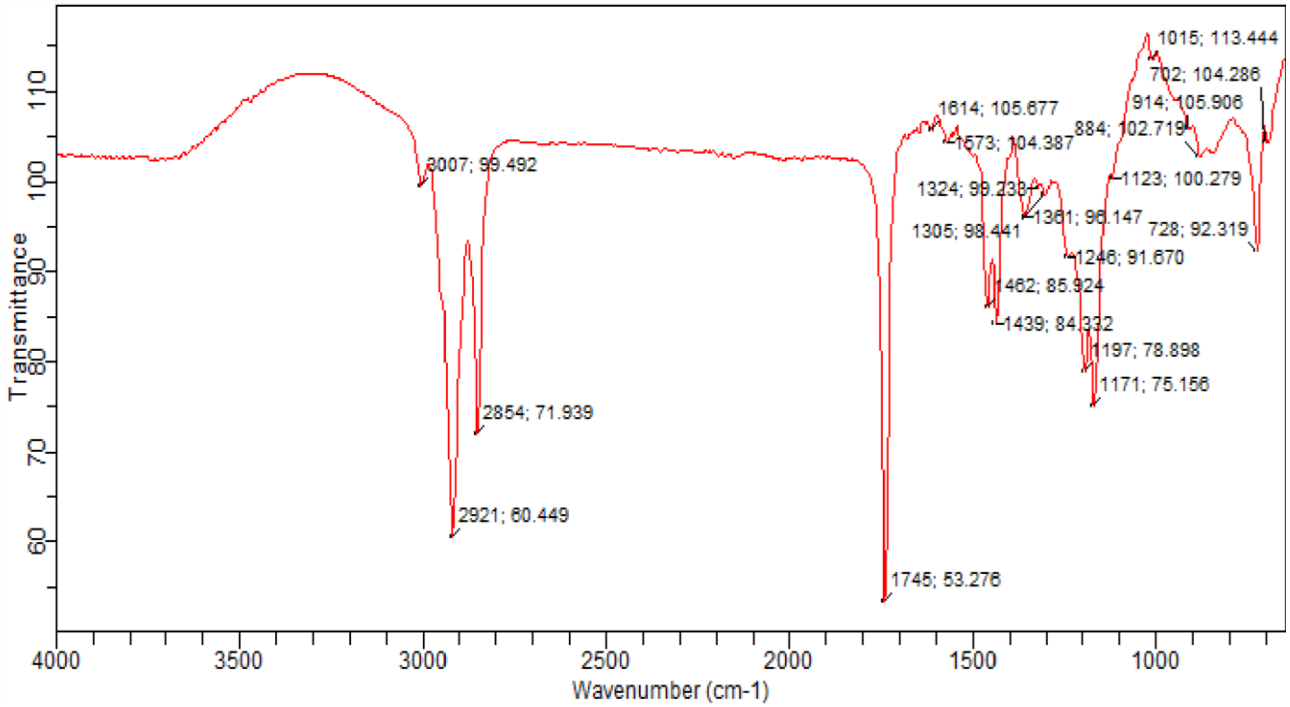

Figure 9: FTIR of *Gmelina arborea* biodiesel(used zeolite support)

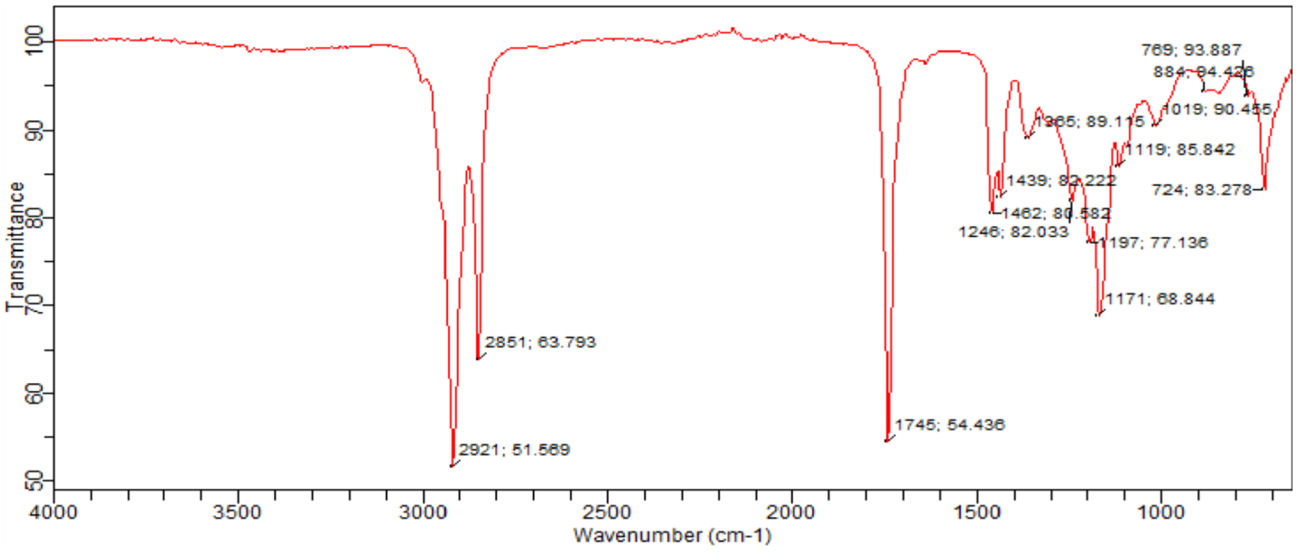

Figure 10: FTIR of Shea butter biodiesel(used zeolite support)

Discussion

Table 1 depicts some physicochemical parameters obtained from crude GSO and SB respectively. The percentage moisture content of GSO and SB are respectively 2.26 and 0.56 %.

Physicochemical analysis of crude *Gmelinaarborea* and Shea butter oil was compared as both of them, have high acid value, and high moisture content, the crude Shea butter hasa low iodine number compared to crude oil. Shea butter is more stable than *Gmelinaarborea* oil. And also both the two raw materials need to be treated before transesterification. The higher acid values in GSO and SB might be responsible for the low yield. This is supported by the fact that high acid content damages fuel pumps and fuel filters leading to low yield of biodiesel.[17]

The physicochemical analysis of crude Shea butter and *Gmelina* seed oil obtained has a saponification value of 172.59 ± 0.56 and 196.10 ± 0.21mgKOH/g respectively. The saponification value of the Shea butter oil was lower than that of *Gmelinaarborea* oil but higher than that of the Shea oil reported in the previous study.[18] This property can be considered to be very useful in soap making. This indicates that both the two oils have the potential to be used in soap making. Hence, high saponification values justify the usage of oil in soap making. The reason for different values might be due to different geographic areas, different species, the nature of extraction, and storage.[19] The iodine value obtained for Shea butter was 7.91 ± 0.01 and 33 ± 0.98 I2/100g for *Gmelina arborea* oil . The iodine values obtained are less than 100, which show that both two oils could be classified as a non-drying oil. It was reported that non-drying oils have iodine values less than 100, which are useful in the production of soaps.[20]

The acid value of *Gmelina arborea* seed oil was greater than that of Shea butter and also the acid value was greater than 11.64mgKOH/g oil.[21] It was also higher than that of avocado seed oil 5.00mg/g.[22] The density of oil was found to be 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.[23]

Physicochemical parameters obtained were compared to ASTM, moisture content, density, and acid value does not fall within the ASTM ranges, therefore the crude oil had to be treated before transesterification. High acid content will damage fuel pumps and fuel filters; as a result, will lead to a low yield of biodiesel.[17]

The values obtained after the first esterification were 2.45 ± 0.015 for GSO and 1.98 ± 0.028 for SB, based on the acid value obtained both GSO and SB had to be treated by esterification again before biodiesel production. The values obtained after the second esterification were 0.92 ± 0.01 for GSO and 0.99 ± 0.021 for SB respectively.Therefore the acid value was within the ranges of ASTM. Hence the oilsare suitable for biodiesel production.

---

[17] Canakci, M and Gerpen, V. J. (2001).Biodiesel Production from Oils and Fats with High-Free Fatty Acids. Transaction of the American Society of Agricultural Engineers, 44(6): 1429-1436.

[18] Warra, A. A., Wawata, I. G., Gunu, S. Y. and Aujaka, K. M. (2011). Extraction and physicochemical Analysis of some northern Nigeria industrial oils. Archives of Applied Science Research, 3:536-541.

[19] Abali,Y., Colak, S., and Yapici, S. (1997).The optimization of the dissolution of phosphate rock with Cl2 SO2 gas mixtures in aqueous medium. Hydrometallurgy, 46(1): 27-35.

[20] Asuquo, J.E. (2008) . Studies on the adsorption of some selected metallic soaps ono hematite. Ph.D. Dissertation, University of Port Harcourt, Nigeria.

[21] Sani, H., Gaya, U. (2019). Methanolysis of *Gmelina* seed oil to biodiesel with KNO3 activated Mgo-ZnO composite catalyst. Journal of the Turkish Chemical Society, 6(3):335-348.

[22] Ikhuria, E.U. and Maliki, M. (2007). Characterization of avocado pea and African pea extracts. African Journal Biotechnology, 6(7):950-952

[23] Afolabi, I. S. (2008). Chemical qualities of oils from fresh and market vegetable crops within Kwara State Nigeria. Nigerian Society for Experimental Biology, 20:71-75

Based on the result obtained, the reaction time (7 h), the speed of reaction 600 rpm, and the volume of methanol to oil (9:1) were fixed throughout the reaction. KOH/fresh zeolite catalyst is more effective in the yield of GOME and SBME, followed by the used zeolite catalyst.

Biodiesel yield from freshly modified zeolite is more effective in the yield of both GOME and SBME followed by homogenous catalyst (NaOH) and modified KOH used zeolite, hence, for GOME the yield varied from 89.4, 87 and 72 % respectively, whereas for SBME the yield varied from 95.8, 90.6 and 88 % respectively. As reported in the literature, the heterogeneous transesterification method proved to be superior as compared to the homogenous transesterification method, especially in the separation and purification of the FAME product.[24]

It can be suggested that zeolite cannot be used directly for the transesterification reaction; it has to be modified for the transesterification reaction to take place. Different concentrations of KOH solution were used during the activation of the spent zeolite catalyst. Both GOME and SBME yield increased from 15-25 % KOH zeolite, the yield increased from 47-88.3 % for SBME yield and 43.7 to 72 % in GOME. The concentration of KOH above 25 % during activation of used zeolite did not increase the yield. It was documented that palm oil biodiesel increased with the concentration of KOH during the activation of modified zeolite.[16]

The most characteristic absorption peaks of the waste vegetable oil were indicated in Figure 7-10 The absorption peak appearing at 721 cm^{-1}, in crude Shea butter and 724 cm^{-1} in crude *Gmelina* seed oil, is representative of $-CH_2$ rocking, and the other one at 1737 cm^{-1} in crude Shea butter and 1745 cm^{-1} in crude *Gmelina* seed oil, are representative to C═O ester stretch. From the spectra, it showed the produced biodiesel absorption peaks appeared at 1439 cm^{-1} which is the methyl ester group ($CO-O-CH_3$), and the characterization peak at 1197 cm^{-1} corresponds to the (C-O) ester peak. It was obvious the reduction of CH_2-O- groups in oil and the appearance of CH_3-O- vibrations in biodiesel. Also, the split of 1164 cm^{-1} in the oil sample into 1197 cm^{-1} and 1171 cm^{-1} in the biodiesel sample indicates the conversion of oil into biodiesel. The main difference between the two FTIR spectra is related to the transformation of ester groups in the waste oil sample into methyl esters in the produced biodiesel.

Based on the result biodiesel obtained from GSO and SB have very good fuel quality, biodiesel from GSO and SB have-low viscosity but high cetane number, high flash point, high pour point, and high cloud point. The biodiesel obtained from fresh zeolite catalyst has the optimum yield, lowest viscosity, and high flash point but has high pour and cloud points. It was observed that biodiesel from GSO and SB had similar fuel parameters and also both the values obtained are within the range of ASTM. In general, the optimum flash point and cetane number were obtained from biodieselfrom SB, while the optimum viscosity, cloud point, and pour point were obtained from biodiesel from GSO.

As reported in the literature, the heterogeneous transesterification method proved to be superior compared to the homogenous transesterification method, especially in the separation and purification of the FAME product.[24] Currently, there are many feasible heterogeneous catalysts used in the transesterification process to provide lower cost and produce biodiesel in a more

---

[24] Zabeti, M., Daud,W. M. A. W., andAroua, M. K. (2009). Optimization of the activity of CaO/Al2O3 catalyst for biodiesel production using response surface methodology.Applied. Catalysis, 366:154-159.

environmentally friendly manner such as metal oxides.[25] Metal complexes.[26] active metals loaded on supports.[27] resins.[28] and lipases.[29]

Conclusion

Biodiesel was successfully prepared from high-free fatty acid GSO and SB via transesterification using a homogenous catalyst (sodium hydroxide) and heterogeneous catalyst (zeolite). The effect of homogenous catalyst, freshly modified zeolite, and modified used zeolite on the yield of biodiesel was evaluated. Fuel parameters were determined to study the effect of 25 % KOH/fresh zeolite, 25 % KOH/ used zeolite, and homogenous catalyst(NaOH) 1wt % onthe quality of both GOME and SBME. GOME obtained from 25 % KOH/fresh zeolite gave the highest flash point and also SBME obtained from 25 % KOH/fresh zeolite gave the highest cetane number for biodiesel from Shea butter.Sodium methoxide can be used in the preparation of GOME and SBME due to its effectiveness concerning yield and quality of biodiesel, but this needs to be done with caution (due to its environmental effect) or when heterogeneous catalysts like zeolite are not available.

---

[25] Taufiq, Y. H., Lee, H. V., Hussein, M. Z. and Yunus, R. (2011). Calcium-based mixed oxide catalysts for methanolysis of *Jatrophacurcas* oil to biodiesel. Biomass and Bioenergy 35:827-834.

[26] Ferreira, D. A. C., Meneghetti, M. R., Meneghetti, S. M. .P., and Wolf, S. R. (2007). Methanolysis General, 317:58-61.

[27] Xie, W., Huang, X., and Li, H. (2007). Soybean oil methyl esters preparation using NaX zeolites loaded with KOH as a heterogeneous catalyst. Bioresource Technology:936-939.

[28] Feng, Y., Zhang, A., Li, J., and He, B. A. (2011). Continuous process for biodiesel production in a fixed bed reactor packed with cation exchange resin as heterogeneous catalyst. Bioresource Technology, 102:3607-3609.

[29] Liu, C., Zhang, S., Su, Z., and Wang, P. (2012).LiCl-induced improvement of multilayer nanofibrous lipase for biodiesel synthesis. Bioresource Technology, 103:266-272.