Materialia 000 (2018) 1–7

Contents lists available at ScienceDirect

Materialia

journal homepage: www.elsevier.com/locate/mtla

Full Length Article

# Biodiesel production from shea butter: A suitable alternative fuel to premix fuel

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ARTICLE INFO

*Keywords:*
Shea butter
Premix fuel
Biodiesel
Transesterification
Methyl ester

ABSTRACT

This work focused on transesterification of shea butter into biodiesel via double stage acid-alkaline catalysis. It outlined the use of shea butter biodiesel as a substitute for premix fuel for fishing. Upon using 0.8–1.4 wt% KOH catalyst per shea butter weight, a methanol/oil ratio of 4:5 and 2 mL of 95% $H_2SO_4$ at 65 °C, the optimum biodiesel yield was 74% wt. FTIR was conducted to ascertain the biodiesel composition. Some of the important properties of the biodiesel (density, kinematic viscosity, flash point, cetane number, iodine value, methyl ester content and high heating value) at different temperatures were investigated and compared with the ASTM and EN standards for biodiesel, and No. 2 diesel. The compared values were similar, indicating that the shea butter biodiesel can be used as an alternative to premix fuel.

## 1. Introduction

Fossil fuels are the major sources of all energies that are globally utilized and consumed. Unfortunately, these sources are gradually diminishing, and will be finished in the near future. The continuous increasing consumption of fossil fuels such as petroleum tends to escalate air pollution and global warming related problems caused by carbon dioxide [1]. This has, therefore, prompted researchers to consider the utilization of alternative and renewable energy sources such as biofuels and biodegradable fuels which are more environmentally friendly [2,3]. Biodiesel had been mentioned as one of the alternative energy sources that can significantly solve most of these environmental and sustainability related problems [4].

Biodiesel is obtained from renewable, biodegradable and nontoxic biological sources which have low emission profiles, thereby, making it environmentally beneficial [4,5]. Biodiesel is generally preferred as an alternative fuel for diesel engines as a result of its technical, environmental and economic benefits. Biodiesel can prolong the life of an engine by 20% and reduce the rapid occurrence of heat-related damages [6,7]. In 1990, it was the first alternative fuel which met the US EPA Tier I and Tier II Health Effects testing requirements of the Clean Air Act Amendments [8]. Prior to World War II, biodiesel was first used in powering heavy-duty vehicles in South Africa [5].

As biodiesel is currently a major concern in many developing and advanced countries, the source is a significant factor that must be considered, especially, in our environment. One of the most promising sources of biodiesel is shea butter from shea nut tree (*Vitellaria paradoxa / Butyrospermum parkii*). It grows naturally and commonly in Africa, especially, Cameroon, Senegal, Nigeria, Ivory Coast and Ghana . It is less expensive in its raw state. The properties of biodiesel depend on the physicochemical properties of the feedstock. The shea butter comprises mainly of triglycerides, unsaponifiable materials and consists of 40–55% fat per kernel [6]. The triglyceride content is impregnated with oleic, stearic and linoleic acids [9,10]. Unlike many biodiesel fuels, one of the advantages of shea butter biodiesel is its resistant to oxidation due to the presence tocopherols and some phenolic compounds acting as natural antioxidants [11], hence, making it a better alternative fuel. However, the direct use of shea butter oil like other vegetable oils in fuel engines may be problematic due to their high density, high viscosity, low volatility, and its incomplete combustion in diesel engines. In view of these, different approaches have been reported to reduce the high viscosity and density of vegetable oils. Some of these are: superacid catalysis [12]; micro emulsions with short chain alcohols such as ethanol/methanol [13]; the use of zeolite [14]; and acid-alkaline catalyzed transesterification with either ethanol or methanol/methoxide, or both and enzyme catalyst [15].

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https://doi.org/10.1016/j.mtla.2018.08.038
Received 20 May 2018; Received in revised form 21 August 2018; Accepted 31 August 2018
Available online xxx

The transesterification of shea butter results in the production of a fuel comprised of mono-alkyl esters of long chain fatty acids called biodiesel [16]. In fact, the transesterification of vegetable oils such as shea butter oil is not a new process. For instance, in 1853, E. Duffy and J. Patrick were two of the renowned scientists who conducted such process. Thereafter, similar study was conducted on the application of biodiesel from pea nut for diesel engine between 1893 and 1911 [10]. It was further proposed that the use of biodiesel fuel will help develop the agriculture industry of the countries which use it significantly and consistently. Indeed, despite the benefits derived from shea butter biodiesel, few studies had been done on biodiesel production from shea butter and its use as fuel for diesel engines. For example, it was reported that the density, kinematic viscosity, cloud point and pour points of shea butter biodiesel blended with fossil diesel fuel yielded suitable results with ASTM 6751 and EN14214 standards [5,7,17]. It is very obvious that very few studies have been conducted on the production and characterization of biodiesel from shea butter, and its utilization as premix fuel.

This paper, therefore, focused on the production of biodiesel from shea butter via acid-alkaline transesterification process and its utilization as premix fuel. Some selected essential physical and chemical properties such as density, kinematic viscosity, flash point, cetane number, acid value, iodine value, methyl ester content and high heating value were measured to characterize the biodiesel produced, and compared with those of ASTM and EN standards for biodiesel and No. 2 diesel. Furthermore, FTIR measurements were conducted for the biodiesel produced to ascertain its composition. The produced biodiesel would be employed in fishing outboard motors as full or partial replacement for premix fuel to enhance preservation and sustainability of fishing environments. Premix fuels are petroleum fuels used in fishing outboard motors, mostly, in Africa.

## 2. Materials and methods

$CH_3OH$, $H_2SO_4$, and $H_3PO_4$ solvents from Sigma-Aldrich Chemical Co. with purities ≥ 99% were used. The Shea butter was obtained from Ho, Cape Coast and Kumasi markets, in Ghana. The KOH (≥ 98%) and $MgSO_4$ (≥ 99%), viscometer, and separation funnel used were obtained from the University of Cape Coast chemical store room. The different physical and chemical properties of the shea butter were determined as indicated in Table 1.

### 2.1. Production of biodiesel from shea butter

Shea butter was chosen because unlike many biodiesel fuels, the shea butter biodiesel is resistant to oxidation due to the presence of tocopherols and some phenolic compounds acting as natural antioxidants [11]. Tocopherol concentration of 0.986–1.302 mg/g has been detected in cotton seed and palm oil biodiesels produced at 60–65 °C for 2 h without deformation [18,19], therefore, it could also be present in the shea butter biodiesel without deformation. The method and approach of Kac [20], and Enweremadu and Alamu [5] were adopted for the production and characterization of the shea butter biodiesel. Also, this method involved pre-treating the shea butter by esterification to reduce the FFA% which could impede the biodiesel production to < 1, before transesterification (Scheme 1) to biodiesel (mono methyl ester)[21,22]. The shea butter was heated to 100 °C to eliminate the residual water, and to enhance the reaction.

**Table 1**
Properties of crude shea butter.

| Property | Mean values |
|---|---|
| Density ($kgm^{-3}$) at 25 °C | 914.0 |
| Viscosity ($mm^2s^{-1}$) at 40 °C | 39.98 |
| Acid value (mg KOH/g) | 3.62 |
| FFA% | 1.819 |
| Iodine value ($I_2$ g KOH/g) | 59.5 |
| Saponification value (mg KOH/g) | 190.0 |

In general, for each complete process, the total amount of methanol used was 80% of the shea butter volume, total amount of KOH used was 0.8–1.4% by weight of the oil, and the total amount of sulphuric acid used was 0.2% of the oil volume. The entire process was carried out at 65 °C and at atmospheric pressure. The process included three stages, namely, Stage A – acid catalyzed stage, Stage B – Neutralization stage, and Stage C – Alkaline catalyzed stage.

#### 2.1.1. Stage A – acid catalyzed stage

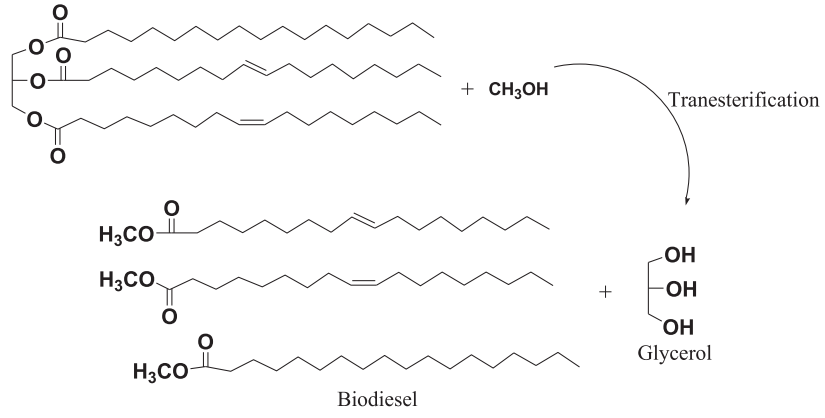
In a 1000 mL beaker, 500 mL of shea butter was heated to 65 °C. 340 mL of $CH_3OH$ was added whiles stirring at 500–600 rpm. The mixture was initially stirred continuously for 5 min during which 1 mL 95% $H_2SO_4$ was added whiles maintaining the temperature at 65 °C. The resulting mixture was further stirred continuously for 120 min at 500–600 rpm and the mixture transferred into a separating funnel. The separating funnel content was left for 120 min to settle before discarding the upper layer ($CH_3OH/H_2O$).

#### 2.1.2. Stage B – neutralization stage

4–7 g of KOH were measured and each dissolved in 60 mL of methanol to get potassium methoxide solution ($CH_3OK$). 30 mL of the $CH_3OK$ solution was added to the acid treated mixture at 65 °C and the resulting mixture was stirred gently for 5 min, transferred into a separating funnel, allowed to settle overnight and the glycerin drained off.

#### 2.1.3. Stage C – alkaline catalyzed stage

The remaining mixture was heated to 65 °C, and the rest of the $CH_3OK$ solution was slowly added while stirring. The stirring was done




**Scheme 1.** Production of biodiesel from shea butter.

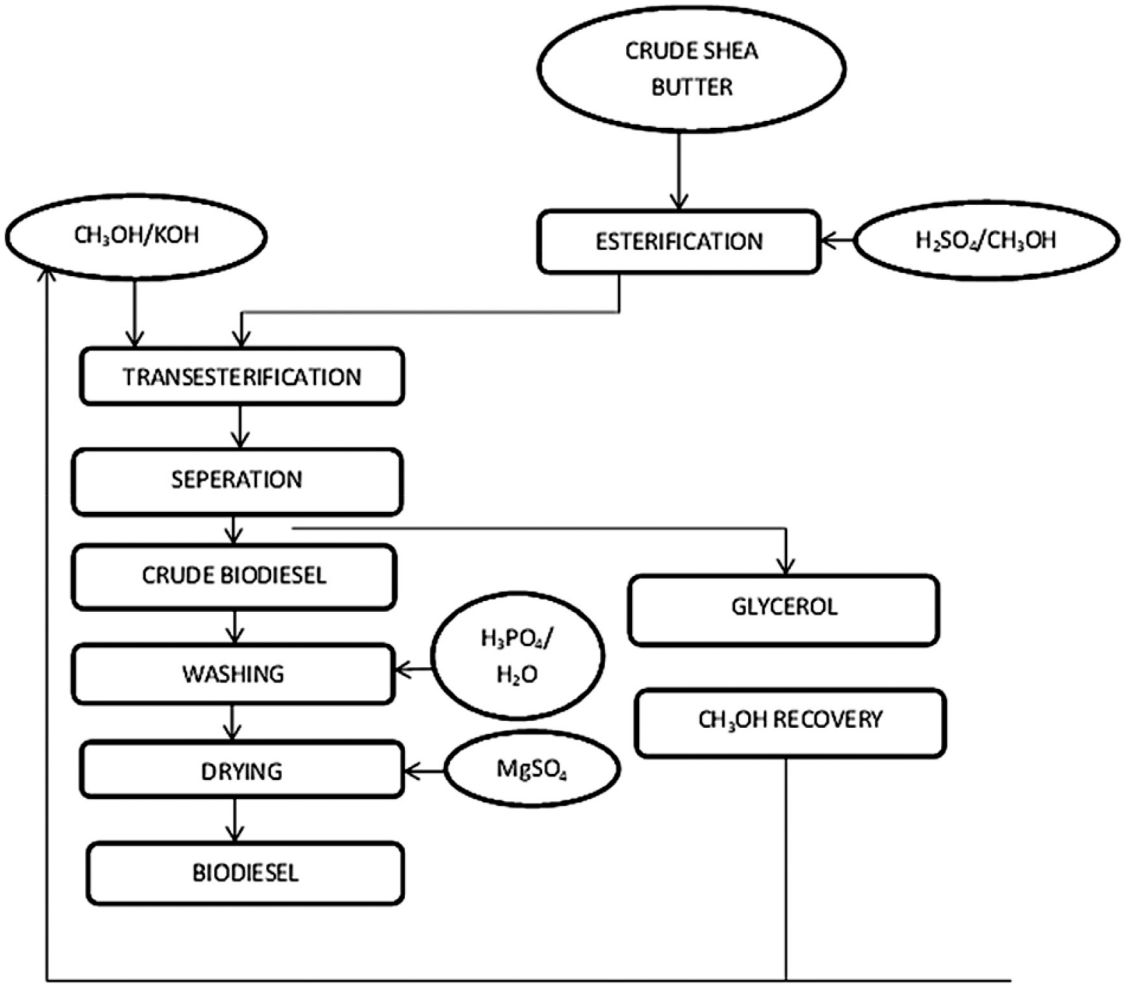



**Fig. 1.** Flow sheet of the transesterification process.

for 60 min and the entire mixture transferred into a separation funnel. The mixture was left for 24–48 h after which the crude biodiesel was drained off, washed with warm distilled water, acidified with 2 mL of 10% $H_3PO_4$ and dried with $MgSO_4$. The flow chart for the entire biodiesel production process is shown in Fig. 1.

The acid value, and the iodine value (IV) were respectively ascertained via color indicator titration and volumetric titration using Hanus reagent (ISO 3961, 1996). The kinematic viscosity, density at a temperature of 40 °C; and flash points of the biodiesel were also evaluated with ASTM standard methods.

The higher heating value (HHV) was calculated from the by Demirbas using iodine value (IV) and saponification value (SV) [9]:

$$HHV = 49.43 - (0.015IV) - (0.041SV) \quad (1)$$

From the iodine value and the saponification value of the biodiesel, the cetane index (CI) was calculated as per the correlation given by Krisnangkura 1986 [23]

$$CI = 46.30 + \frac{5458}{SV} - 0.225IV \quad (2)$$

Cetane number (CN) has been known not to differ much from cetane index. Therefore the correlation reported by Enweremadu 2010 [5] was used to calculate the cetane number

$$CN = CI - 1.50 \text{ to } 2.60 \quad (3)$$

The methyl ester content was determined using;

$$FAME(\%) = -45.055 * \ln\mu + 162.85 \quad (4)$$

Where: $\mu$ is kinematic viscosity at 40 °C [24].

The conversion of the shea butter to methyl ester was calculated using the approach of Yong 2007, and Marchetti 2008 [25,26]

$$\text{Conversion}(\%) = \left(1 - \frac{AV_b}{AV_o}\right) \quad (5)$$

Where: $AV_b$ is the acid value of biodiesel and $AV_o$ is the acid value of the shea nut butter.

### 2.2. Density measurement

Standard procedures were applied in evaluating the specific density of the biodiesel. A 50 mL specific glass bottle was employed in this measurement at different temperatures 25 °C, 30 °C, 40 °C, 50 °C, 55 °C, 60 °C and 70 °C. The subsequent density of the biodiesel was estimated by multiplying the specific density of the biodiesel with that of water.

### 2.3. Viscosity measurement

Kinematic viscosity values were determined with Fungilab Alpha L Rotational Viscometer from Brookfield Company, U.S.A. The cone spindle CPE 40 was used to measure the viscosity of the biodiesel at different temperatures 25 °C, 30 °C, 40 °C, 50 °C, 55 °C, 60 °C and 70 °C. In the measurement, the ASTM D445 standard method was applied [27].

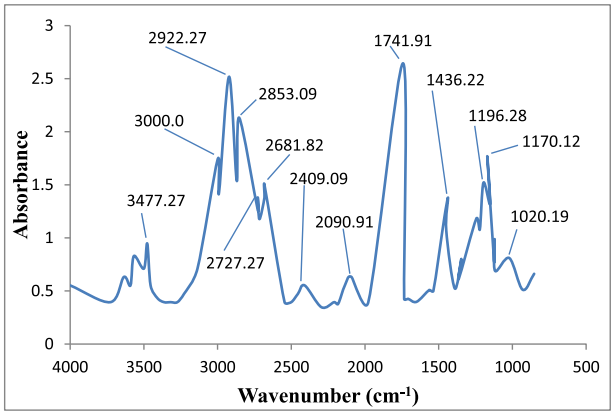



**Fig. 2.** FTIR peaks of the produced shea butter biodiesel.

### 2.4. FTIR measurement

Fourier transform infrared measurement was done at Kwame Nkrumah University of Science and Technology, Ghana, using Interspec 200-X FTIR spectrometer from Spectronic Camspec Ltd, UK. Biodiesel samples were prepared at the same concentrations and placed directly on the sample holder and spectra acquired, which was an average of 32 or 64 spectral acquisition and 4 $cm^{-1}$ resolution. The passing wave penetrated the sample to an average depth of about 20 μm. This measurement was used to ascertain the various functional groups which could be found in the biodiesel (mono methyl ester) produced.

The reproducibility of all the measurements was ascertained by repeating two experiments under the same conditions.

## 3. Results and discussion

### 3.1. Density, kinematic viscosity and FTIR analysis of the shea butter biodiesel

Table 2 shows the densities and viscosities of the shea butter biodiesel at different temperatures. Lower density values were interpreted to indicate a more complete ester conversion. Viscosity is the basic parameter reflecting the extent of the reaction, directly related to the methyl ester content.

It was observed that the densities and the kinematic viscosities decreased with increasing temperature implying that more of the heavy glycerin was removed, indicating a more complete reaction. This reflected in the FTIR spectrum (Fig. 2). Thus, the wavenumber 3000.00 $cm^{-1}$ is attributed to –CH bond. The -$CH_2$ stretching vibrations correspond to 2853.09 and 2922.27 $cm^{-1}$; –C = O bond stretch is assigned to 1741.91 [28].

In the 'finger print' region, the asymmetric stretching of –$CH_3$ which is absent in vegetable oil corresponds to 1436.22 $cm^{-1}$ [28]. The O–$CH_3$ stretch at 1196.28 $cm^{-1}$ is characteristic of biodiesel [29].

Conversely, Fig. 3(a) and (b) clearly show the effects of varying catalyst temperature on the kinematic viscosity and density of the biodiesel. The decrease in kinematic viscosity and density suggests that the flow properties were improved by the transesterification process as more of the heavy glycerin was removed whilst more of the required products (methyl esters) were formed.

The yield, density and kinematic viscosity of shea butter biodiesel at 40 °C are shown in Table 3. The values were obtained by varying the catalyst concentrations while keeping all other process parameters constant. It was observed that the density and kinematic viscosity decreased with increasing catalyst concentration.

Also, as depicted in Fig. 4, the yield of biodiesel generally increased with increasing catalyst concentration. The optimum yield (74% wt) was recorded at catalyst concentration of 1.24 wt% . This corresponded to the kinematic viscosity and density of 2.109 $mm^2/s$ and 0.84658 $g/cm^3$.

Beyond 1.24 wt% catalyst concentration, the yield of biodiesel decreased sharply due the appreciable formation of soap and gel. The results indicate that catalyst concentration at a given temperature

**Table 2**
Density and kinematic viscosity of the shea butter biodiesel at different temperatures.

| Temperature ( °C) | Density ($g/cm^3$) | Kinematic viscosity ($mm^2/s$) |
|---|---|---|
| 35 | 0.89638 | 2.474 |
| 40 | 0.84687 | 2.165 |
| 45 | 0.84578 | 1.924 |
| 50 | 0.81127 | 1.732 |
| 55 | 0.80604 | 1.575 |
| 60 | 0.80571 | 1.443 |
| 65 | 0.79219 | 1.332 |
| 70 | 0.78206 | 1.237 |

**Table 3**
Yield, density and kinematic viscosity of shea butter biodiesel with varying amount of catalyst.

| Amount of catalyst (wt%) | Yield (%wt) | Density ($g/cm^3$ at 40 °C) | Kinematic viscosity ($mm^2/s$) at 40 °C |
|---|---|---|---|
| 0.80 | 31.25 | 0.84687 | 2.165 |
| 1.00 | 41.00 | 0.84680 | 2.115 |
| 1.20 | 60.00 | 0.84678 | 2.112 |
| 1.24 | 74.00 | 0.84658 | 2.109 |
| 1.30 | 68.00 | 0.84640 | 2.104 |
| 1.40 | 15.00 | 0.84635 | 2.100 |

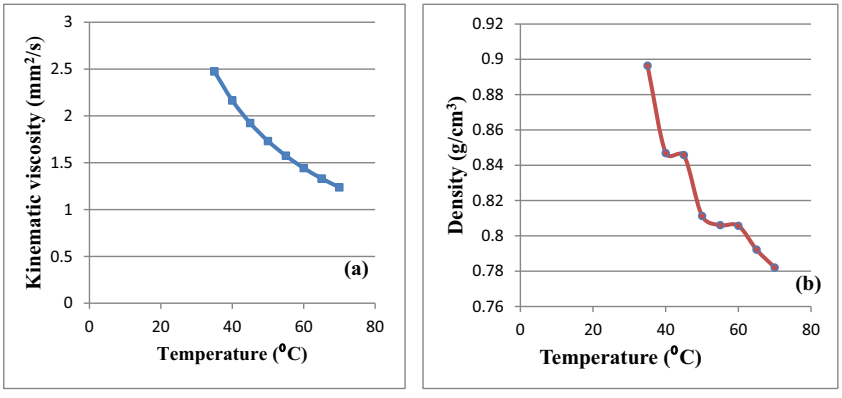



**Fig. 3.** (a): Effects of varying catalyst temperature on the biodiesel kinematic viscosity (b): Effects of varying catalyst temperature on the biodiesel density.

**Table 4**
Properties of shea butter biodiesel.

| Property | Mean value | Standards | | |
|---|---|---|---|---|
| | | ASTM | EN14214 | No. 2 diesel |
| Density ($kgm^{-3}$) at 25 °C | 865.18 | | 860 - 900 | 820 - 860 |
| Kinematic viscosity ($mm^2s^{-1}$) at 40 °C | 2.17 | 1.9 - 6.00 | 3.5 - 5.0 | 2.5 - 5.0 |
| Flash point ( °C) | 171 | 130 min | >100 | >55 |
| Heating value (MJ/kg) | 37.93 | | | 42 |
| Cetane number | 58 | 47 min | 51 min | 49 - 55 |
| Acid value (mg KOH/g) | 0.28 | 0.5 max | 0.5 max | |
| Iodine value ($I_2$g KOH/g) | 36.42 | | 120 max | |

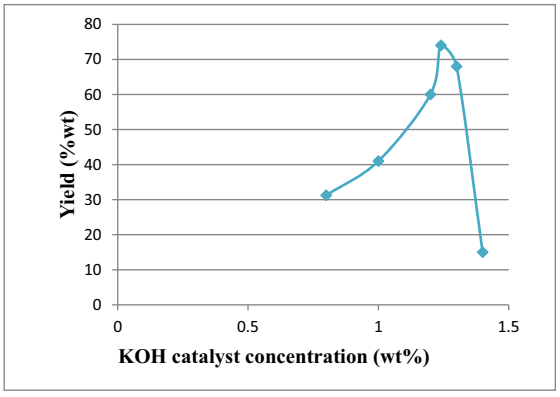



**Fig. 4.** Effects of catalyst concentration on the yield of shea butter biodiesel.

can significantly affect the shea butter biodiesel production and its properties. This result is consistent with other studies which reported optimum biodiesel at catalyst concentration of 1–1.4 wt% [15,30].

To ascertain the suitability of the biodiesel, most of its fuel properties were compared with ASTM D6751, EN14214 and No. 2 diesel standards, which was found to be suitable (Table 4). The results indicate that the biodiesel produced can also be suitable for diesel engines.

This is because kinematic viscosity, cetane number, density and acid value are known to be the key fuel properties for diesel engines. According to ASTM, for biodiesel to be used in diesel engines, the kinematic viscosity must be between 1.9 and 6.0 $mm^2\ s^{-1}$. Also, according to EN14214, the density must fall between 0.86 and 0.90 g $cm^{-3}$ (860–900 $kg/m^3$) [31]. The cetane number and acid value were within both the ASTM D6751 and EN-14214 standards. As observed, the methyl ester (biodiesel) of the shea butter had relatively closer fuel properties to the petroleum diesel than the crude shea nut butter in Table 1, and the effect was due to the transesterification of the crude shea butter.

### *3.2. Reaction mechanisms for acid and alkaline catalyzed shea butter biodiesel production*

#### *3.2.1. Reaction mechanisms for acid catalyzed shea butter biodiesel production*

Again, the mechanism for acid catalyzed reaction included three steps and as presented in Scheme 2. In the first step, protonation of the carbonyl oxygen atom of the triglyceride group in the presence of acid catalyst was attained. In the next step, formation of carbocation tetrahedral intermediate compound by nucleophilic attack of the alcohol occurred. In the final step, rearrangement of carbocation tetrahedral intermediate or migration of proton to form the biodiesel, diglyceride and proton was realized.

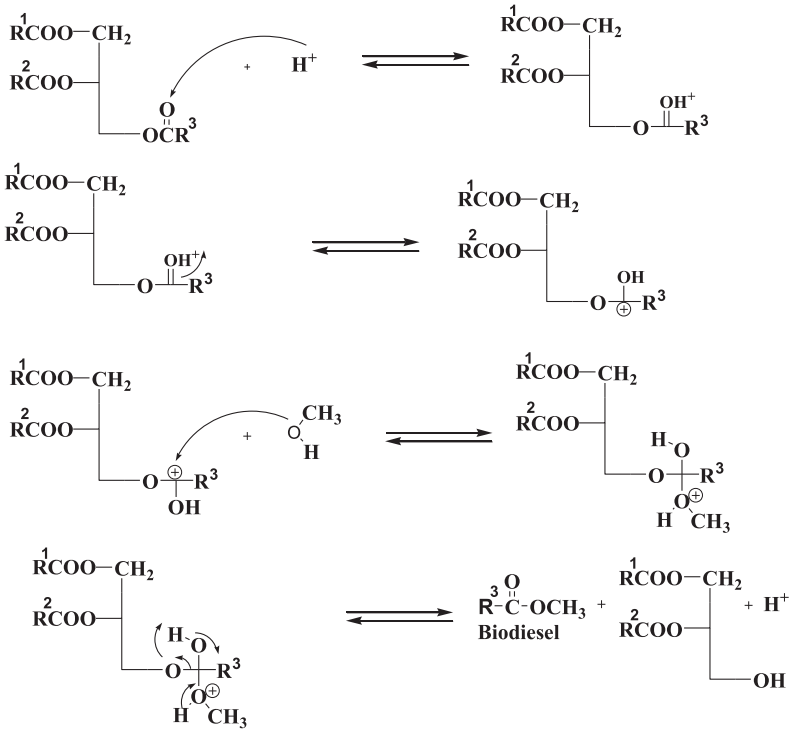
#### *3.2.2. Reaction mechanisms for alkaline catalyzed shea butter biodiesel production*

Few years back, Eckey et al. reported the three steps reaction mechanism for alkaline catalyzed transesterification reaction [32] as presented in Scheme 3. The first step involved attack of carbonyl carbon atom of the triglyceride group by anion (alkaline, methoxide) to form a tetrahedral intermediate compound. The second step applied a rearrangement of tetrahedral intermediate compound to form biodiesel and formation of diglyceride anion. In the third step, diglyceride anion reacts with alcohol (methanol) to form diglyceride and regeneration of methoxide was realized.

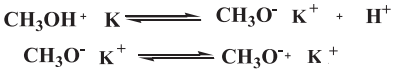
### *3.3. Shea butter biodiesel sustainability as a premix fuel for fishing*

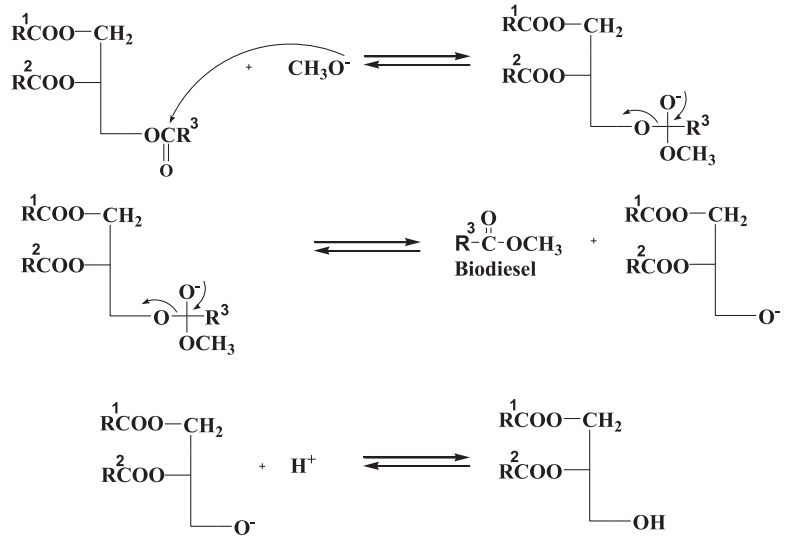
Shea butter is produced in nineteen African countries [33]. Most of these countries such as Cameroon, Senegal, Nigeria, Ivory Coast and Ghana significantly engage in fishing using premix fuel in their outboard motors. It is, therefore, very necessary to use shea butter biodiesel as premix fuel in outboard motor for the fishing. When there are leakages of the conventional fossil fuel from the parts of the outboard motor into the water bodies such as; sea, rivers or lakes, it may be toxic to the fish and other aquatic organisms present. Thus, spill of the fossil fuel on the water bodies would contaminate it, and eventually become harmful to the living organisms (fish, Shrimps etc) in the system. This has necessitated the application of biodiesel as fuel for fishing so as to replace the premix fuel (a fossil fuel) in the outboard motor. The application of the biodiesel from shea butter as fuel for fishing would promote environmentally friendly and sustainable fishing practices. Besides, shea butter biodiesel is resistant to oxidation since shea butter contains natural antioxidants such as tocopherols and some phenolic compounds [11].

There is also the need for sustaining the growth of the shea butter trees to be used on the large scale for the production of the biodiesel instead of the imported fuel. This can be realized by adopting sustainable principles; where the trees would be cultivated in its ecological zone in Africa, and other shea butter producing countries. Herein, the communities are poor and they are mostly composed of women who collect the shea butter from the wild trees [34]. Therefore, women in these deprived communities would be trained to cultivate on large scale the shea trees so that it can be used to produce large quantities of the biodiesel as premix fuel for fishing. The successful implementation of this planting project would make this idea sustainable. Furthermore, it would create jobs, equally improve the lives of the women and the vulnerable people in the communities, and also enhance the afforestation [34]. This will guarantee the principle of sustainable development, as the approach touches on social, economic and environmental issues. This also affirms the idea of green economy. For example, it is projected that about 9.4 million shea nut trees exist in Ghana, and these can potentially give 174,971 metric tonnes of shea butter annually [35]. This quantity would subsequently yield 129,715 metric tonnes of biodiesel per year. Conversely, the premix fuel consumptions in Ghana in 2012, 2013 and 2014, were 53,433.18 metric tonnes, 48, 443.67 metric tonnes and 50,983.78 metric tonnes, respectively [36]. So, considering the consumption of premix fuel over the years and the projected production of biodiesel yield per year in Ghana and the other African countries, we can conveniently say that it is very feasible economically to fully or partially replace premix fuel for fishing with the biodiesel or even to blend the premix fuel with the biodiesel.




**Scheme 2.** Plausible mechanism for acid catalyzed by biodiesel production.






**Scheme 3.** Plausible mechanism for alkaline catalyzed by biodiesel production.

## 4. Conclusions

A two-step acid-alkali catalyzed method was used in the synthesis of the shea butter biodiesel. The optimum biodiesel yield was 74% wt at 1.24 wt% catalyst concentration. Furthermore, the significant fuel properties such as density, kinematic viscosity, flash point, heating value, cetane number, acid value, and iodine value tested were within the ASTM and EN standards and were very close to No.2 petroleum diesel.

The results of the study suggest that, in the future, shea butter biodiesel could be considered a substitute for premix fuel since the investigated properties were closer to the compared standards and petroleum diesel.

## Acknowledgment

The authors are grateful to the University of Cape Coast-Ghana for the availability of their laboratories.

## Declaration of interest

The authors have no potential conflict of interest regarding the publication of this paper.

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