

Research Article

Ghanaian Clay as a Catalyst for Transesterificating Shea Butter Oil as Alternative Feedstock for Green Energy Production

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In this study, catalytic transesterification of *Shea butter* oil with methanol to biodiesel was carried out. The clay particle was first sieved with a 250 ml sieve and then calcined at $800\mu\text{C}$ for 8 hours in a furnace. The calcined clay was then modified with 5% each of $\text{Zn}(\text{NO}_3)_2$ and SnCl_2 separately for 3 h at 60°C using incipient wetness impregnation method. The slurry compositions were then dried at 120°C overnight in an oven. The modified clay catalysts were subsequently calcined for 3 hours at 600°C . The clay that had been modified with SnO_2 and ZnO was assigned SD and ZD, respectively, whereas the clay that had not been doped (control) was assigned as AC. The catalysts were characterized using X-ray Diffractometer (XRD), Brunauer Emmett Teller (BET) surface area, and Scanning Electron Microscopy (SEM). Biodiesel was then produced from each of the catalysts under varying conditions of methanol to oil molar ratio, catalyst concentration, reaction temperature, and reaction time. When compared the performances of the catalysts (activated clay modified with SnO_2 , ZnO , and the control), activated clay modified with SnO_2 resulted in an optimal conversion yield of 99.8% under 8 : 1 methanol to oil molar ratio, 1% catalyst concentration, 60°C of reaction time in 60 minutes. The catalytic performance was affected by the basicity and surface area of the catalysts. The biodiesel produced was comparable to American and European Union standards.

1. Introduction

Conventional fuel is commonly deployed for power production in a variety of industries including transport, agriculture, commercial, household, and manufacturing. The rising consumption of conventional diesel contributes to the reduction in fossil fuel supplies [1, 2]. The negative impact of increasing reliance on fossil fuels has sparked interest in alternative energy sources.

Biodiesel, a mono-alkyl ester of long chain fatty acids derived mostly from vegetable oils, is projected to substitute fossil diesel [3, 4]. When compared to mineral diesel, biodiesel is non-toxic, renewable, biodegradable, and emits less CO , SO_2 , volatile organic compounds, and unburned hydrocarbons. Because its properties are remarkably similar to those of petroleum diesel, biodiesel may be applied in any diesel engine without modification [5, 6].

Blending with diesel, micro-emulsions, thermal cracking, and transesterification are some of the processes used to

produce biodiesel [7–10]. Transesterification is, however, the most prevalent and well-established of the numerous processes [11]. Transesterification reaction basically involves reacting alcohol with fatty acid triglycerides (vegetable oil) in the presence of a catalyst to produce glycerol and esters [12]. Stoichiometrically, a 3 : 1 molar ratio of alcohol to oil is appropriate. Because the reaction is reversible, an excess of alcohol (>3 : 1) is necessary to shift the equilibrium to the products and allow phase separation from glycerol to occur [10]. Most transesterification reactions employ methanol and ethanol particularly, methanol due to its low cost and benefits (polar and shortest chain alcohol).

Vegetable oil, non-edible oil, used cooking oil, used frying oil, and beef fat are the most common feedstocks for biodiesel manufacturing [11, 13, 14]. The utilization of the second-generation feedstock in BD production should be exploited [15–17]. The costs of oil-containing materials constitute substantially, 70 to 80% of the cost of biodiesel resulting in a 1.5 times higher production cost of biodiesel

than diesel [7]. Several initiatives are underway to lower feedstock costs [18, 19]. Non-edible waste oil is about 2 to 3 times less expensive than edible oils on average. Biodiesel made from waste oils and greases is expected to cost \$1 to \$1.50 a gallon in the future (<https://www.nwbiodiesel.org>). At optimal operating conditions, the quality of biodiesel from waste non-edible oil and vegetable oil may be achieved [20]. Furthermore, rising food consumption and industrial use have resulted in a huge increase in the creation of waste oils and fats. The undesirable environmental effects of waste oil disposal are also eliminated by converting it to fuel [21, 22]. Aside from choosing low-cost raw ingredients, boosting reaction conversion is critical for lowering biodiesel costs. As a result, catalysts play a key part in the reaction process and choosing the proper catalyst and concentration is critical for achieving the best reaction conditions. Catalyst speeds up the process of correcting the equilibrium.

Clay is a non-toxic base catalyst that could be found all over the planet. Clay is made of magnesium carbonate (MgCO_3), calcium carbonate (CaCO_3), and other chemicals in various amounts. Clay is quite inexpensive in Ghana (in certain situations, it is almost free) and it is mostly utilized in building and ceramics. The active phases of clay minerals are produced by thermal breakdown of the clay into CaO , CaMgO , CaCO_2 , and MgO comparable with other natural sources such as CaCO_3 [11, 12]. In this study, the feedstock used for the biodiesel processes was *Shea butter* oil.

2. Experimental

2.1. Materials and Methods. The raw *Shea butter* oil was sourced from a local company in Ho, Ghana's Volta Region capital. The Department of Soil Science, University of Cape Coast provided powdered clay for this project. SnCl_2 (98%), $\text{Zn}(\text{NO}_3)_2$ (98%), and MeOH (99.7%) were acquired from RYTE Aid Chemicals in Ho, Ghana.

2.2. Refining of the Crude Shea Butter Oil. The process of refining the crude *Shea butter* oil comprised three stages: acid conditioning, deacidification, and post treatment.

2.3. Step 1: Acid Conditioning. In this regard, 300 g of raw *Shea butter* oil was heat to 60°C for 1 h initially. The oil was then treated with phosphoric acid (molar ratio of phosphoric acid to oil was 0.05). Because phosphoric acid dissolves metal/phospholipid complexes, this method was employed to convert non-hydrated phospholipids to hydrated phospholipids. The oil was subsequently, treated with distilled water (6:1 water to oil molar ratio). The oil-water-phosphoric acid combination was then agitated for 1 hour at 60°C . After that, the mixture was allowed to set foot for 12 hours in a separating funnel by gravity. Having drawn out the water and other impurities at the bottom layer, the top and middle layers were centrifuged with Anke TDL-5-A Centrifugal separator (10 min, 20°C and 5000 rpm) to obtain the oil.

2.4. Step 2: Deacidification. The deacidification process was followed by neutralizing the acid in the oil with sodium hydroxide. Thus, 250 g of the crude oil was mixed with 7.5 ml of 18% (NaOH) solution. The mixture was then heat at 60°C for 1 h with a magnetic stirrer at 4000 rpm. Addition of NaCl then followed as an electrolyte after the temperature was adjusted to 80°C (after the 1 h) while mixing by magnetic stirrer at 2000 rpm for 20 min. An emulsion was obtained in this case. After centrifugation with Anke TDL-5-A Centrifugal Separator (10 min, 20°C and 5000 rpm) soap was separated from the oil phase.

2.5. Step 3: Post Treatment. The *Shea butter* oil obtained in Section 2.4 was washed by adding some amount of distilled water at 60°C for 1 h (4000 rpm magnetic stirring rate). The product of the mixture of water and oil was then transferred to a separating funnel and allowed to settle under gravity for 12 h. The oil occupied the top layer and the bottom layer made of water and other impurities were drawn out. The remaining water in the oil was removed by evaporation under vacuum.

2.6. Determination of the Physical Properties of the Crude and Refined Shea Butter Oil. Standard procedures were used to determine the principal physical characteristics of the crude and refined *Shea butter* oil. Using ASTM D6304, the quantity of water was determined by Coulometric Karl Fischer Titration equipment. The acid value (in mg KOH/g) was calculated using the ASTM D664-01 technique, whereas the kinematic viscosity at 20°C was determined using ASTM D445. The amount of sulfur was determined using ASTM D4294 standards. The other properties including high heating value (mj/g oil), iodine value (centigram l/g oil), carbon residue (wt%), ash content, and density at 20°C were determined using PRC standards GB/T3535, GB/T510, GB/T 2540, GB/T508, and SH/T0248, respectively.

2.7. Preparation of Catalyst. The clay particle was first sieved with a 250 ml sieve and then calcined for 8 hours at 800°C in a furnace [16]. The activated clay catalyst was then doped with 5% of two distinct metal salts thus; $\text{Zn}(\text{NO}_3)_2$ and SnCl_2 separately for 3 h at 60°C to obtain the modified clay catalysts. This process was by incipient wetness impregnation process [23]. The slurry compositions were then dried in a 120°C oven overnight. The catalyst was prepared by calcining ground and sieved doped clay for 3 hours at 600°C . The clay that had been doped with SnO_2 and ZnO was assigned as SD and ZD, respectively, whereas the clay that had not been doped (control) was assigned AC.

2.8. Characterization of the Catalyst. The Shimadzu XRD-6000 Diffractometer was used for the XRD analysis with a scanning rate of $2^\circ/\text{min}$ ($20\text{--}80^\circ$). The $\text{Cu K}\alpha$ radiation produced by Philip glass diffraction X-ray tube (broad focus 2.7 kW type) was applied on a plate-mounted catalyst. On the basis of the locations and intensities of the peaks, the

resultant patterns were compared to Joint Committee on Powder Diffraction Standards (JCPDS) files.

Brunauer Emmet Teller (BET) surface area instrument, Thermo Finnigan Scorpomatic 1900 type was used to evaluate the surface area of the catalysts. To eliminate moisture and foreign gases deposited on the catalyst surfaces, the catalysts were dried overnight at 423 K in a vacuum chamber set to 77 K. The adsorption and desorption of N₂ on catalyst surfaces was studied.

A JEOL scanning electron microscope, model JSM-6400, was used to investigate the surface morphologies of the catalysts. With double-sided tape, a thin coating of catalyst was spread over the sample holder, then coated with a thin film of gold using BIO-RAS Sputter. Images were captured at various magnifications. Leaching tests using an Atomic Absorption Spectrometer were used to assess the stability of each catalyst. Before being sucked up with a nebulizer, the methyl esters were first diluted in chloroform (99.99%). The sample was combined with acetylene (C₂H₂) as the fuel and an oxidant with nitrogen oxides (NO₂) in a nebulizer to create a heterogeneous mixture of fine aerosols. To evaporate the organic materials from the solvent, the fuel flow was adjusted to 4.2 l/min and a high temperature flame with a height of 11.0 nm applied.

The light emitted by the hollow cathode lamp was of the same wavelength as the light absorbed by the targeted element (calcium Ca). The light intensity was proportional to the amount of calcium in the sample. The resulting Ca concentration was then compared to the concentrations of 1 ml, 3 ml, 5 ml, and 10 ml standard solutions of Ca diluted in 100 ml distilled water.

2.9. Transesterification of the Shea Butter Oil. Methyl esters of the *Shea butter* oil were prepared by refluxing the oil in a 250 mL three-neck reaction flask fitted with a condenser for 1 hour at a predetermined temperature with a volume of methanol containing modified clay as catalyst. In a separating funnel, two layers were formed as a result of the reaction. Methyl esters, residual catalyst, residues of methanol, and other contaminants constituted the top layer. After drawing out the bottom layer of glycerol, the product was refined by neutralizing it with 10% sulfuric acid. Having thoroughly washed the product, the methyl ester layer was dried with a rotary evaporator under decreased pressure at 70°C.

2.10. Methyl Esters Yield Determination. The yields of methyl esters were measured using an Agilent 7890A gas chromatograph with a flame-ionization detector (FID) and HP-5 capillary column (30 m 0.32 mm 0.25 μm). The carrier gas was helium. GC-MS was used to perform the oven temperature ramp procedure. Tetradecane was used as an internal standard for the calculation of the yields. The biodiesel was analyzed by dissolving 1 ml of the sample in 5 mL of n-hexane and injecting 0.5 L of the product into GC for each sample. Using the equation below, the yields were computed from the methyl esters' content:

$$\text{Biodiesel yield} = \frac{W_{\text{tetradecane}} \times A_B \times f_{\text{tetradecane}}}{A_{\text{tetradecane}} \times W_s} \times 100\%, \quad (1)$$

where $W_{\text{tetradecane}}$ is the weight of tetradecane, $A_{\text{tetradecane}}$ is the peak area of tetradecane, A_B is the peak area of methyl esters, $f_{\text{tetradecane}}$ is the response factor, and W_s is the weight of the sample.

2.11. Methyl Esters Composition Determination. GC-MS was used to analyze the makeup of the obtained methyl esters. The analysis was carried out on an Agilent HP-6890 gas chromatograph (Agilent Technologies, Palo Alto, CA, USA) using an HP-5MS 5% phenylmethylsiloxane capillary column (30 m 0.25 mm i.d., film thickness 0.25 μm; Restek, Bellefonte, PA). The carrier gas was helium at a flow rate of 1.0 ml/min. Using split ratio of 15:1, each sample (1 L) was injected into the column. The recommended procedure was used to carry out the oven temperature ramp program [24]. By matching the observed mass spectra with NIST05.LIB and NIST05s.LIB (National Institute of Standards and Technology) library data given by the software (AMDIS-Chromatogram) of the GC-MS system, the fatty acids were identified.

2.12. Characterization of the Methyl Esters. Standard procedures were used to analyze the characteristics of the methyl esters. The cetane number was calculated using the ASTM D6890 standard while the flash point was determined using ASTM D93. The water content of biodiesel was determined by coulometric Karl Fisher titration equipment and the ASTM D6304 technique. ASTM D664-01 was used to calculate the acid value (given as mg KOH/g). The Rancimat technique was used to test oxidative stability at 110°C using EN 14112. Copper corrosion was determined using ASTM D130 while kinematic viscosity was determined using ASTM D445 at 40°C. ASTM D4294 was used to determine the sulfur concentration. PRC standards GB/T3535, GB/T510, GB/T 2540, SH/T0248, GB/T508, and SH/T0796 were used to determine other parameters such as pour point, cloud point, cold filter plugging point, density at 20°C, ash content, and glycerin content, respectively.

2.13. Improvement of Properties of Shea Butter Methyl Esters

2.13.1. Winterization Experiment. 100 g of *Shea butter* methyl esters (in a flask) was kept in cold water bath at constant temperature of 0°C for 5 h. Crystal precipitation was observed in the bottom. The winterized methyl esters were vacuum filtered, and the pour point (PP), cloud point CP, and the cold filter plugging point (CFPP) were determined.

2.13.2. The Improvement of Oxidation Stability. To assess the function of oxidative stability in biodiesel, ascorbic acid was used as an antioxidant. Before and after the experiment, the peroxide value (POV) was calculated using the GB/T5538 technique.

3. Results and Discussion

3.1. Properties of Shea Butter Oil. Tables 1 and 2 show key quality parameters and the GC-MS results of fatty acid contents of crude and refined *Shea butter* oil, respectively. The physical properties and content of the oil guaranteed it as standard for biodiesel production [25–31].

3.2. Characterization of the Catalyst. Figures 1(C), 1(F), and 1(A) show the XRD patterns of undoped clay, SnO₂-clay, and ZnO-clay catalysts, respectively. Undoped clay (AC) catalyst displayed accentuated peaks at $\Theta^\circ = 29.1^\circ, 30.2^\circ, 36.7^\circ, 55.4^\circ, 67.8^\circ,$ and 68.1° (C) corresponding to CaO and Θ° values of $21.5^\circ, 29.2^\circ, 34.5^\circ, 37.2^\circ, 43.2^\circ, 45.1^\circ, 62.5^\circ$ (F) related to MgO. Molaei Dehkordi and Ghasemi [11] and Hassan and Fadhil [15] found a similar tendency. The high CaO and MgO peak intensities were observed in AD, suggesting that CaO-MgO complexes with high crystallinity were present. Previous studies on the transesterification process via CaO and MgO indicated that basic active sites influence biodiesel conversion [20]. Other minor peaks in the SD pattern have been detected in SnO₂ modified catalysts. The peaks at $\Theta^\circ = 15.6^\circ, 18.9^\circ, 47.5^\circ,$ and 53.2° corresponds to ZnO in the catalyst.

CaO and MgO reflection peaks existed in SD and ZD catalyst patterns. However, their strengths were relatively weak indicating that CaO and MgO concentrations reduced with the introduction of other metal oxide. Metal salts added to the undoped clay may obscure the parent material thereby reducing the intensity of the clay peaks [13, 23].

Figure 2 shows the surface area of undoped clay and modified catalysts. At 900°C the fresh clay was calcined to produce a mesoporous structure of AD with a specific surface area of 21 m²/g, which is greater than the specific surface area (8 m²/g) reported by Cetinkaya and Karaosmanoglu [20]. Cetinkaya and Karaosmanoglu [20] found that the surface area and shape of calcined clay differed dramatically from that of fresh clay due to thermal disintegration which allows for chemical and physical changes. The specific surface area of the SD catalyst increased to 80 m²/g after SnO₂ was added to the undoped calcined clay (AC) catalyst. This was presumably, owing to SnO₂'s higher surface area than the parent material. As ZnO was doped on AD, the specific surface area of the catalyst increased to 55 m²/g, suggesting that the molecules of ZnO had entered the pores of the AD catalysts resulting in a decrease in surface area when compared with SD. The inclusion of high surface area metal oxides SnO₂ and ZnO greatly enhanced the specific surface area of the parent material, providing additional active sites to aid in the transesterification process. Through impregnation, calcination and catalyst composition, clay was changed to improve the surface area of the modified clay catalyst [20].

The variations in surface morphologies of the catalyst and modified catalysts owing to dopant addition were seen in the SEM images (Figure 2). The flattened flakes or saddle-like form of the AD catalyst in Figure 2(a) reduced when SnO₂ was doped onto it. When compared with the shape of

TABLE 1: Comparison of crude shea butter oil properties to literature standards.

Property	Crude shea butter	[29, 30] Recommendation
Kinematic viscosity (mm ² /s)	40.3	40.2
Cetane number	18.3	—
Higher heating value (mj/g)	25.01	24.67
Ash content (w %)	1.40	—
Sulfur content (wt %)	7.0	5
Acid value (mg KOH/g oil)	6.92	5.96
Density at 15°C (kg/m ³)	304 (20°C)	210
Water (mg/kg)	580	—
Iodine value (centigram I/goil)	18.4	13.2
Carbon residue (wt %)	0.23	0.18

TABLE 2: Properties of refined shea butter oil in comparison to literature standards.

Property	Refined shea butter oil	[31]
Kinematic viscosity (mm ² /s)	34.4	35.4
Cetane number	24	23
Higher heating value (mj/g)	33	33
Ash content (w %)	0.68	0.7
Sulfur content (wt %)	0.012	0.02
Acid value (mg KOH/g oil)	4.5	4.0
Density at 15°C (kg/m ³)	857 (20°C)	918
Water (mg/kg)	430	—
Iodine value (centigram IN/foil)	100.9	101
Carbon residue (wt %)	0.22	0.3

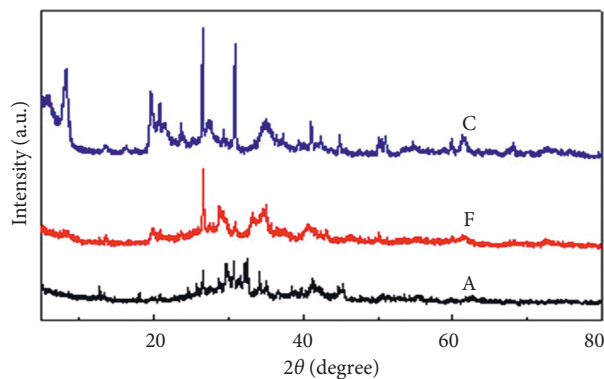


FIGURE 1: (A) XRD pattern of ZnO doped on activated clay (ZD). (C) XRD pattern of activated dolomite (AC). (F) XRD pattern of SnO₂ doped on activated clay (SD).

AD particles, the shape of SD particles was indistinct (Figure 2(c)). The catalyst agglomerated when ZnO was added to clay (Figure 2(b)). This was attributed to tiny ZnO particles dispersing over the surface of the parent material and covering up the CaO-MgO active site.

3.3. Influence of Transesterification Reaction Conditions

3.3.1. Effect of Methanol to Oil Molar Ratio on Yield of Methyl Esters. Because transesterification is a reversible process,

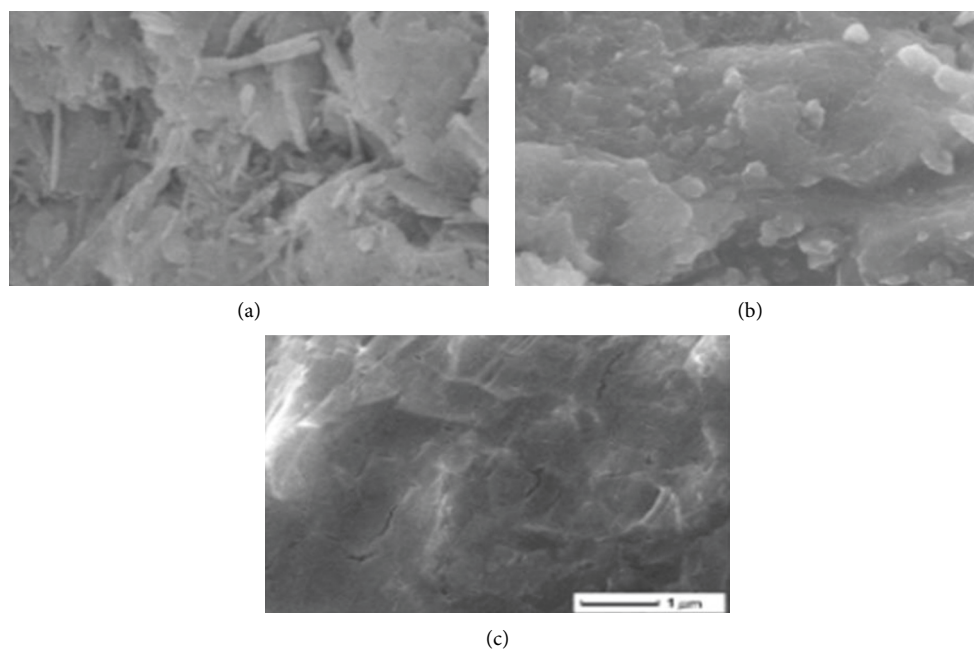


FIGURE 2: (a) SEM image of activated dolomite (AC), (b) SEM image of ZnO doped on activated clay (ZD), and (c) SEM image of SnO₂ doped on activated clay (SD).

additional methanol promotes the reaction in the forward direction; hence, the influence of molar ratio was investigated by altering the methanol to oil molar ratio at different levels (Figure 3). When the methanol to oil molar ratio was adjusted from 2:1 to 8:1, the biodiesel produced increase from 78.4% to 97.6% and then declined to 92.3% at 10:1. The excess methanol had no significant influence on the yield above a molar ratio of 10:1. When the molar ratio of methanol to oil surpassed 9:1, glycerol separation became more difficult, leading to lowering of methyl esters yield from 97.6% to 94.5% which agrees with Amish et al. [32] and Encinar et al. [33]. According to Takase et al. [34–37], excess amounts of methanol in a transesterification reaction leads to dilution of the oil. This phenomenon might have occurred in this study. Methanol to oil ratio of 8:1 was chosen as the optimum.

3.3.2. Effect of Catalyst Concentration. The effect of catalyst concentration on the yield of methyl esters is indicated in Figure 4. From Figure 4, the yield first increased from 71.8% to 97.6% with an increased in catalyst concentration from 0.5% to 1%. After 1% concentration was exceeded, the yield began decreasing. This might be explained by the development of soap which could impede the separation of the methyl ester phase during the washing process [38]. The soap particles produce emulsions with water which may enhance viscosity. The presence of a large amount of catalyst in the same amount of oil and methanol concentrates the solution, resulting in poor mixing at the same agitation speed. And this leads to increased mass transfer resistance since some catalysts might stay unused, resulting in a drop in conversion from 97.6% to 92.1% when the catalyst was further increased from 1% to 1.5%, respectively [38]. Studies

by Xie et al. [39] agrees with the outcome of the current study. Since the optimum yield (97.6%) occurred at 1%, catalyst concentration of 1% was selected as optimum.

3.3.3. Effect of Reaction Temperature. In this study, the preparation of methyl esters from *Shea butter* oil was carried out at different temperatures (30°C, 45°C, 60°C, 65°C, and 70°C). At low temperatures, the reaction rate was sluggish (as seen in Figure 5). The yield was optimum (97.6%) at 60°C and gradually declined as the reaction temperature was further increased. Because of the endothermic nature of the transesterification process, a faster reaction rate may often be attained at higher temperatures [40–44]. However, at higher temperatures, methanol may have evaporated and generated a significant number of bubbles, which impeded the process. According to the preceding findings, the reaction does not need a high temperature to get the desired outcome [40–44]. And hence reaction temperature of 60°C was picked as the optimum.

3.3.4. Effect of Reaction Time. For transesterification reactions, reaction time is critical. Figure 6 depicts the link between reaction time and biodiesel yield. According to Figure 6, as the reaction time grows, the biodiesel yield increases as well and beyond a given period, it remains constant owing to the reversible nature of the process. The yield was 82.3% during the first 30 minutes of batch time, demonstrating that the transesterification process is highly quick during the first 30 minutes. After 60 minutes of the optimum reaction time with the optimum yield of 97.6%, the yield fell (94.1%) as the reaction time was increased further to 90 minutes. Comparing the results of Figure 6, it can be

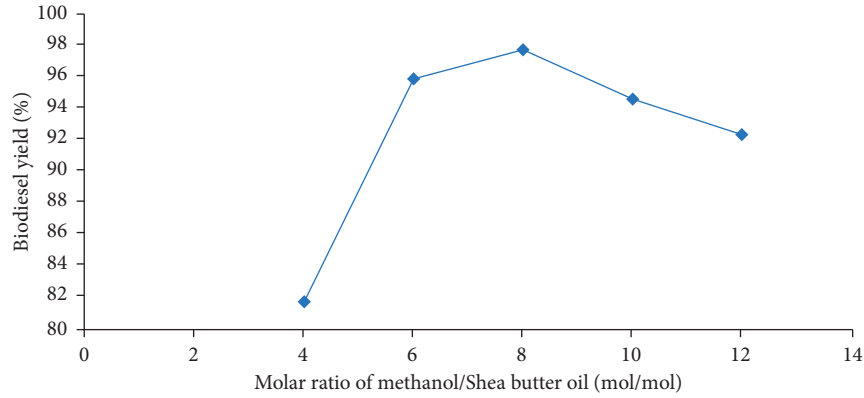


FIGURE 3: Effect of methanol to oil molar ratio on the FAME yield (reaction time 1.5 hrs; catalyst amount 6 wt%; reaction temperature 60°C).

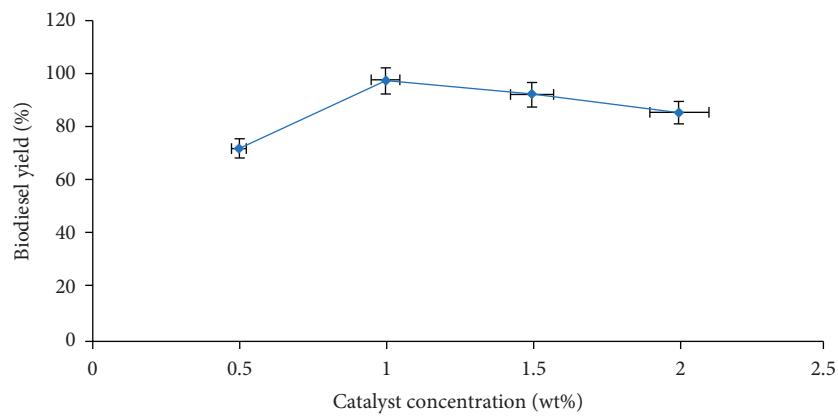


FIGURE 4: Effect of catalyst concentration on methyl esters yield (temperature 60°C; molar ratio 8:1; reaction time 1 h).

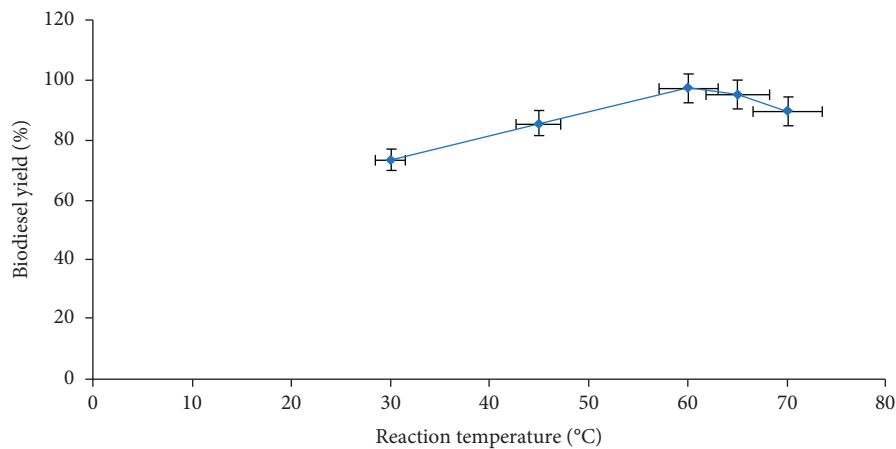


FIGURE 5: Effect of reaction temperature on the FAME yield (reaction temperature 60°C; catalyst amount 6 wt.%; reaction time, 1 hrs).

observed that the general yields of ethyl esters were relatively higher than the corresponding yields of methyl esters of Takase et al. [37] in their study but lower than those of Yee et al. [45]. On the basis of the optimum yield (97.6%), 90 minutes was chosen as the optimum reaction time (Sample chromatograms of the oil and optimum reaction conditions of the maximum methyl esters yield of

the Shea butter oil are shown in Figures 7 and 8, respectively).

3.3.5. Improvement of Low Temperature Performance. To improve the fatty acid methyl esters' low temperature qualities, a winterization experiment was conducted to

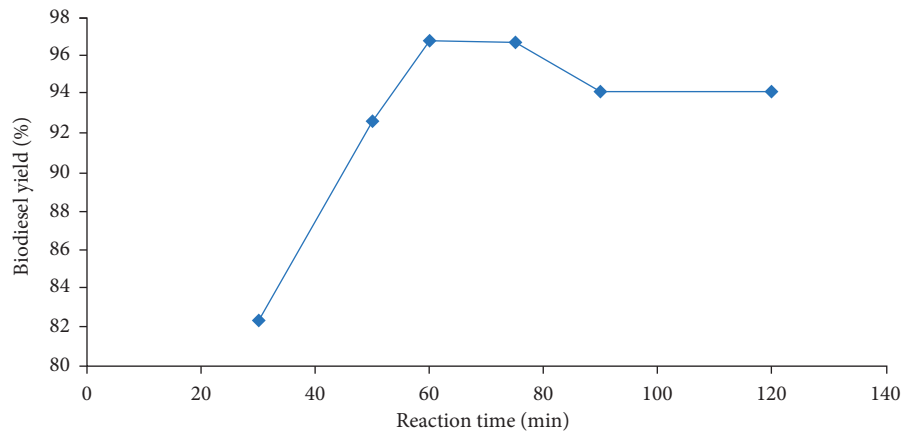


FIGURE 6: Effect of reaction time on the FAME yield (methanol/oil molar ratio 8:1; catalyst amount 6 wt.%; reaction temperature 60°C).

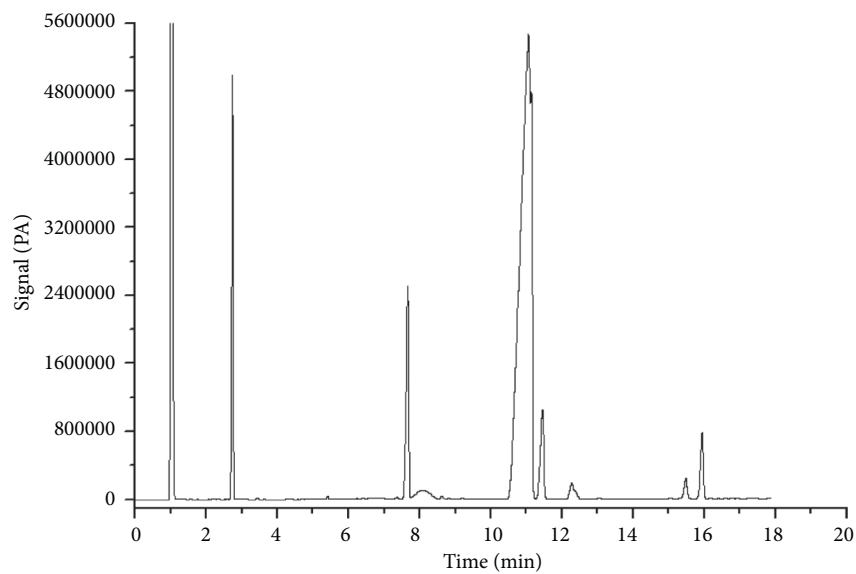


FIGURE 7: GC analysis of *shea butter* oil. 1. Octanoic acid, 2. Tridecenal, 3,2,4-Decadienal, 4,2,4-Decadienal, 5. Nonanoic acid, 6. Hexadecanoic acid (palmitic acid), 7. Octadecanoic acid. (stearic acid), 8. Hexadecanoic acid (palmitic acid), 9,2,6,10,14,18,22-Tetracosahexaene, 10. Octadecenoic acid (oleic acid), 11. Octadecadienoic acid (linoleic acid).

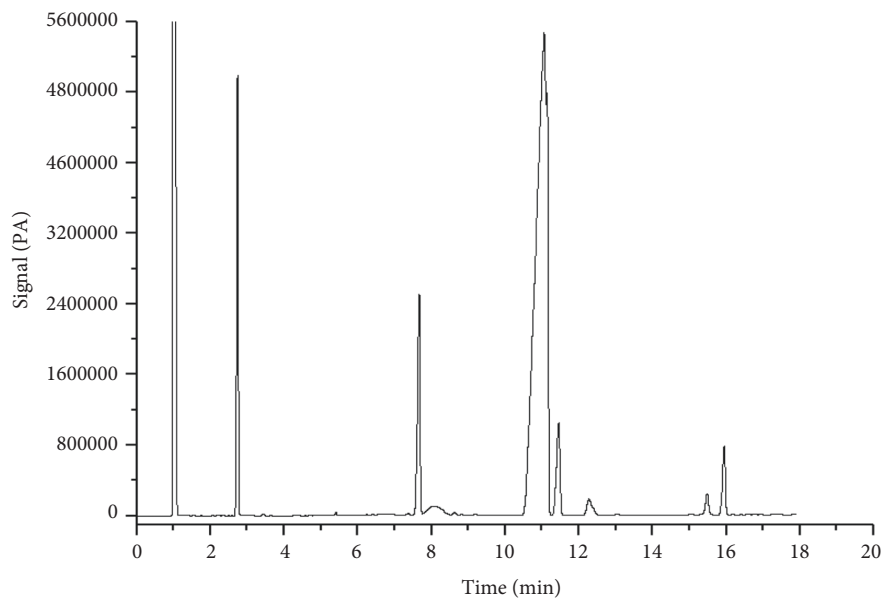


FIGURE 8: Chromatogram of *shea butter* methyl esters: (1) petroleum ether (solvent); (2) tetradecane (used as internal standard); (3) methyl palmitate (C16:0); (4) methyl linoleate (C18:2(cis-9,12)); (5) methyl oleate (C18:1(trans-9)); (6) methyl stearate (C18:0) and (7) methyl arachidate (C20:0).

TABLE 3: Cold flow properties of *shea butter* biodiesel.

Designation of substance	Freezing point (°C)	Pour point (°C)	Cold filter plugging point (°C)	Viscosity (°C)
Before winterization biodiesel	2	6	13	4.26
After winterization biodiesel	-3	-3	0	4.25

TABLE 4: Comparison of properties of the *shea butter* biodiesel and the standards of biodiesel United States and Europe and improvement of key cold flow Properties.

Fuel property	<i>Shea butter</i> <i>Shea butter</i>	ASTM D6751	EN 14214
Cetane number	51	≥47	≥51
Oxidative stability (h)	2.87	≥3	≥6
Kinematic viscosity (mm ² /s; 40°C)	4.298	1.9–6.0	3.5–5.0
Pour point (°C)	6	Report ^d	Country specific ^d
Cold filter plugging point (°C)	13	—	—
Cloud point (°C)	2	Report ^d	Country specific ^d
Flash point (°C)	152	≥93	≥120
Ash content (% w/w)	0.018	≤0.02	≤0.02
Sulfur content (% w/w)	NG	≤0.05	0.020
Acid value (KOH mg/kg)	0.454	≤0.5	≤0.5
Water (mg/kg)	0.4	≤0.05	≤0.05
Density (20°C)	865	—	860–900
Free glycerin (% w/w)	0.01	≤0.02	≤0.020
Total glycerin (% w/w)	0.14	0.24	≤0.25

NG = negligible, CFP = cold flow properties^d Low temperature properties are not strictly specified, but agreed upon by the fuel supplier or purchaser.

minimize the concentration of the long carbon chain saturated methyl ester. Table 3 compares the low-temperature characteristics and viscosity of biodiesel before and after the winterization. The cloud point, pour point, and cold filter clouding point were lowered from 2, 6, and 13 to -3, -3, and 0 following winterization, respectively. At low temperatures, these values are comparable to diesel.

Table 4 compares the biodiesel qualities obtained from *Shea butter* oil to those found in the United States (ASTM D6751) and Europe (EN 14214). The parameters of the produced biodiesel showed numerous similarities, notably after winterization which enhanced the essential cold flow properties (cloud point (CP), pour point (PP), and cold-filter plugging point (CFPP) (Table 3). As a result, biodiesel derived from *Shea butter* oil is recognized as a viable biofuel alternative to diesel.

4. Conclusions

In this study, catalytic transesterification of *shea butter* oil with methanol to biodiesel was carried out. The clay particle was sieved with a 250 ml sieve and then calcined at 800°C for 8 hours in a furnace. The activated clay was then modified with 5% each of Zn(NO₃)₂ and SnCl₂ separately for 3 h at 60°C using incipient wetness impregnation method. The modified clay catalysts were subsequently calcined for 3 hours at 600°C. The clay that had been modified with SnO₂ and ZnO was assigned SD and ZD, respectively, whereas the clay that had not been doped (control) was assigned as AC. The catalysts were characterized by means of X-ray Diffractometer (XRD), Brunauer Emmett Teller (BET) surface area, and Scanning Electron Microscopy (SEM). The transesterification process was then performed with all

catalysts under varying conditions (amount of catalyst, methanol to oil molar ratio, and time). SnO₂ doped on activated clay (GC) achieves an optimal conversion (97.6%) in 90 minutes under 8 : 1 methanol to oil molar ratio, catalyst concentration of 1% at reaction temperature of 60°C. The properties of the obtained biodiesel in general indicated many similarities especially after the winterization which improved the key cold flow properties (cloud point (CP), pour point (PP), and cold-filter plugging point (CFPP)). The biodiesel from *Shea butter* oil is rated as a realistic biofuel as an alternative to diesel.

Data Availability

All the data associated with this study are embedded in the manuscript.

Conflicts of Interest

The authors declare that there are no conflicts of interest.

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