


New non-edible *Allanblackia parviflora* seed oil as an alternative feedstock for biodiesel production and characterization of the fuel

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Abstract

This study is aimed at producing biodiesel from *Allanblackia parviflora* seed oil as a new non-edible alternative resource. The amount of oil extracted from the seeds was approximately 49% and was mainly composed of linoleic acids (66.68). The *Allanblackia parviflora* methyl esters (APME) were prepared using NaOH as a homogeneous base-catalyst for the transesterification. The rate of conversion of the triglycerides to methyl esters was determined using Gas Chromatograph with an optimum yield of 97.1%. The conditions under which this maximum yield (97.1%) was obtained include methanol to *Allanblackia parviflora* oil ratio of 6:1, reaction temperature of 60 °C, NaOH concentration of 1% and reaction time of 2 h. The properties of the methyl esters were comparable to fuel properties of conventional diesel using ASTM and European Union standards. The seeds of *Allanblackia parviflora* can be described as promising non-edible resource.

Keywords Biodiesel · Heterogeneous catalyst · Transesterification · Magnesium oxide · Non-edible oil · *Allanblackia parviflora* · NaOH

1 Introduction

Conventional oil from petroleum is commonly used for transportation, agriculture, trade and for power generation. The increase in the use of conventional fuel has resulted in exhaustion of the fossil resources that are already limited in supply [1]. The negative effect associated with the rising consumption of energy from petroleum reserve has generated interest for alternative resources. Biodiesel is therefore said to be the most alternative substitute to petroleum diesel [2]. It is mono-alkyl esters of long chain fatty acids which is derived from vegetable oils or animal fats. Biodiesel is renewable, non-toxic, environmentally friendly biodegradable and emit low amounts of CO, SO₂, volatile organic compounds and hydrocarbon that are fully burned [3]. Biodiesel is applicable to all engines that uses diesel since the characteristics of biodiesel is similar to mineral diesel [4].

Production technologies for biodiesel include blending, transesterification, micro-emulsions and thermal cracking [5–7]. Transesterification is however; the popular and widely used [8]. The transesterification process is catalyzed by alkalis, acids or enzymes. Alkali catalysts have proven to be more practical and industrially, applied because of their short reaction times and less corrosive nature compared to acid catalysts. They are also less expensive when compared with enzymes [4, 9, 10].

Short chain alcohols such as methanol, ethanol and butanol are normally, used in biodiesel production. Among the mostly used alcohols, methanol is much preferred since it is less expensive and has lot of physical and chemical

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advantages [5–7]. Alkali catalysts such as sodium and potassium hydroxide are commonly used since they produce high amount of biodiesel [11, 12].

Edible and non-edible oils are both used for biodiesel production. But presently, majority used are edible [13]. Biodiesel from edible oil is presently, affected by the issue of cost due to the competition between oil for food and fuel from edible oils; a major reason generating interest for more non-edible low-cost alternative resources [14]. Growing non-edible oil plants on commercial scale can relatively be cheaper than edible ones [15–17]. Non-edibles can be grown in large scale on non-cropped marginal and wastelands and this will definitely increase the desire and appreciation for “marginal lands” [10, 18].

In Ghana, the main non-edible resource for biodiesel include jatropha. However, jatropha alone cannot be sufficient for biodiesel production. Studies however, indicate that there are alternative non-edible oil-yielding crops which can be used as feedstocks [19]. An emerging and promising of them is *Allanblackia parviflora* oil, which has not yet been studied for biodiesel, but gaining popularity in medicinal and soap industry [20].

Allanblackia parviflora is a woody dicotyledonous plant which belongs to the Guttiferae family. The plant is ever-green and flourishes well in areas with a lot of rainfall. *Allanblackia parviflora* is grown in many areas of Africa including Ghana, Sierra Leone, Congo Brazzaville, Cameroon and Uganda. Studies by Ofori et al. [20] on the oil yield of *Allanblackia parviflora* indicate a very good oil (49%) yield. The oil has qualities suitable for producing soaps and confectionaries but not yet been exploited for biodiesel [20], thus providing a basis for this research. In this study, oil was extracted from the *Allanblackia parviflora* seed using Takase et al. approach [21, 22] and the amount determined [23]. The rate of conversion of the triglycerides to methyl esters was determined using Gas Chromatograph with an optimum yield of 97.1% at methanol to *Allanblackia parviflora* oil ratio of 6:1, reaction temperature of 60 °C, NaOH concentration of 1% and reaction time of 2 h.

2 Experimental

2.1 Materials and method

The *Allanblackia parviflora* seed was obtained from Ryte Aid Pharmaceutical Co., Ltd (Ho, Ghana). Methanol, Sodium hydroxides (NaOH), Hydrochloric acid (HCl) and anhydrous sodium sulfate were procured from School of Biological Sciences Chemical Store (University of Cape Coast, Ghana). Entire solvents had AR grade and were used without purification.

2.2 Equipment used

The instrument used in this study include oven (DGH-9140A, YIHENG), soxhlet extractor, rotatory evaporator, 250 ml three-neck reaction flask, condenser, magnetic stirrer, gas chromatograph (GC) (Agilent Technology Inc. USA), gas chromatograph mass spectra (GCMS) Agilent HP-6890 Technologies, Palo Alto, CA, USA) [21, 22].

The experimentations were carried out using 250 ml conical flask. The conical flask was heated using heating plated that has magnetic stirrer with highest heating capacity of 400 °C while the speed of stirring was 600 rpm. A thermometer was fixed using a retort stand that was immersed inside the beaker.

2.3 Transesterification and optimization experiments

30 g of the extracted *Allanblackia parviflora* oil at varied molar ratio of methanol to oil (2:1 to 8:1), varied amount of NaOH (from 0.25 to 2.0%) (reference to the weight of *Allanblackia parviflora* oil) at different reaction time (0.5 to 2.5 h) and reaction temperature (30 to 70 °C), were reacted together in the 250 ml conical flask equipped with a condenser and magnetic stirrer (600 rpm). After each reaction, methanol was removed by means of evaporator that was rotating. Each batch of mixture was then kept in a separating funnel and allowed for 24 h for phase separation to occur. The layers in the upper portion consisted of methyl esters whilst the lower layer comprised slight glycerol. After the 24 h phase separation, the lower layer of glycerol was drawn out and the upper layer made up of biodiesel was then washed with deionized water for three times. Drying and analyses was then done [24].

2.4 Determination of biodiesel yield

Determination of the yield of the biodiesel was carried out using Takase et al. [21, 22] method with little modification. Specifically, the ramp of the oven temperature programming was kept at 175 °C for 2 min, increased to 220 °C at 25 °C/min for 3 min, then increased from 215 to 225 °C at 0.4 °C/min for 4 min and finally increased from 225 to 255 °C at 45 °C/min for 2 min. The rate of flow of hydrogen was 45 mL/min and the one for air was 410 mL/min. The injector and detector temperatures were both kept at 255 °C with pressure of injection at 100 kPa. The injection was carried out in split mode with a split ratio of 55:1. Analysis of each sample of biodiesel was done through dissolving 1 mL of the sample in 5 mL of petroleum ether (60–90 °C) mixed with tetradecane and 0.6 µL of the solution injected into the Gas Chromatography. The yields of the samples of the biodiesel were subsequently, calculated using the equation indicated below:

$$\text{Biodiesel yeild} = \frac{w_{\text{tetradecane}} \times A_B \times f_{\text{tetradecane}}}{A_{\text{tetradecane}} \times w_s} \times 100\%$$

Note: $w_{\text{tetradecane}}$ = weight of the tetradecane; A_B = peak area of the biodiesel; $f_{\text{tetradecane}}$ = response factor; $A_{\text{tetradecane}}$ = peak area of the tetradecane; w_s = weight of the biodiesel used.

All the data points in the study are mean values of triplicate of experimental and GC values.

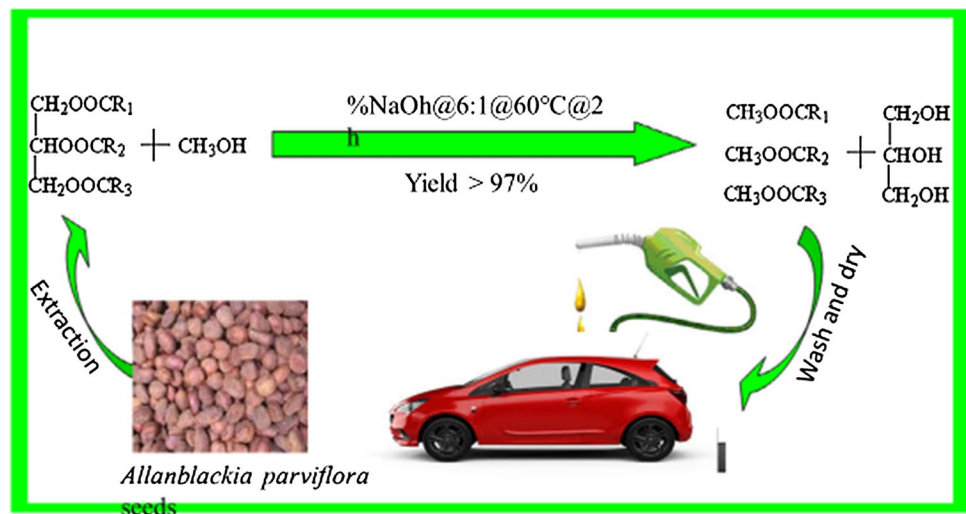
2.5 Identifying the composition of the biodiesel

The compositions of the biodiesel were identified using Gas Chromatography –Mass Spectra as reported by Takase et al. [21, 22] with little modification. The carrier gas used include Helium at the rate of flow being 2.0 mL/min. The samples of biodiesel (1 µL) was injected into the column at a split ratio of 45:1. The oven temperature ramp program was done by Gas Chromatography. The composition of the biodiesel was identified by comparing the mass spectra that was obtained with NIST05.LIB and NIST05s.LIB programming software.

2.6 Determination of the properties of the biodiesel

The determination of properties of the biodiesel were done using ASTM methods. The properties of the biodiesel and the respective methods used in determining it include kinematic viscosity (mm^2/s @40 °C) using ASTM D 445, flash point using D 93, sulfur content using ASTM D 4294, ash content using ASTM D 874, density (20 °C) using ASTM D 5002 and water content using ASTM D 2709. The value for the saponification number was determined by means ISO 3657. The cetane number was determined using ASTM D6890 method. The Thermogravimetric (TGA) curve was determined using TGA-50 (Shimadzu, Kyoto, Japan) thermogravimetric equipment in range of 25 °C and 400 °C (Fig. 1).

Fig. 1 Flow chart of *Allanblackia parviflora* seed oil for the production of biodiesel



3 Discussion of the results

3.1 Characterizing *Allanblackia parviflora* oil

Content of *Allanblackia parviflora* oil (49%) per seed was comparatively high when compared with other oils (non-edible) for biodiesel. Typically, *Acacia Senegal* and cypress with oil contents of 9.80% and 10% respectively [25, 26]. The *Allanblackia parviflora* oil in addition exhibited a kinematic viscosity which is low (28.97 mm²/s) in comparison to those of jatropha, soyabeans and sunflower of 30 or 40 mm²/s at 40 °C [27]. The saponification number and the molecular weight were 198.84 and 889.3 g respectively. And since the *Allanblackia parviflora* oil contained free fatty acid of 0.58% and 0.84% water content (figure less than 1), alkaline-catalyzed transesterification was preferred [28–30].

3.2 Optimizing *Allanblackia parviflora* oil reaction parameters

3.2.1 Influence of molar ratio of methanol to oil

The primary reactant for the transesterification of biodiesel is the alcohol. For this research, variation of methanol was between 2:1 and 8:1 (Fig. 2) at catalyst (NaOH) amount of 1% (wt), temperature of the reaction at 60 °C and time of the reaction at 1.5 h. At low methanol to *Allanblackia parviflora* oil ratio (2:1), the biodiesel yield was equally low (73.2%). The optimum yield of biodiesel (94.6%) was acquired at 6:1. Above this molar ratio (6:1), the additional methanol had negative impact on the yield. Even though, there was reaction at ratio of 2:1, much more ratio was needed to obtain higher yield. Ratio of 8:1 also produced relatively higher yield (92.3%) (bit close) to that of molar ratio of 6:1, this molar ratio will not be economically feasible for the purpose of cost minimization [26]. This findings agrees with those of Meher et al. [31]. From the study it was obvious that to increase the yield of *Allanblackia parviflora* biodiesel, the actual methanol to oil molar ratio needed to increase the esters yield to maximum (94.6%) was 6:1. Any methanol to oil ratio above the limit of maximum could result in decrease in the yield, and this was possibly, as a result of dilution of the oil with the methanol. Henceforth 6:1 molar ratio of methanol to oil was selected as the optimum.

3.2.2 Influence of temperature on the yield of biodiesel

Influence of temperature of the reaction on the reactions process was undertaken at ratio of methanol to oil at 6:1, NaOH amount of 1% (by wt) and reaction time of 1.5 h. Figure 3 shows variation in the rate of *Allanblackia parviflora* oil conversion at various temperatures (30 °C to 75 °C). With increase in reaction temperature from 30 °C to 60 °C, the biodiesel yield correspondingly rose from 70.1% to 94.6%. A further upward adjustment in the temperature of the reaction (from 60 to 65 °C and subsequently to 70 °C) resulted in the fall in yield (92.2% and subsequently to 89.1%). Generally, high temperatures (above 50 °C) favours reaction. But, in this case, the reaction temperature/s (above 60 °C) might have resulted in the vaporization of methanol and this might have inhibited the reaction on the methanol-oil phase. The observations in this study however, agree with previous works [31–33]. It was observed that even at temperature of 30 °C, there was reaction taking place; however, it could not be completed. This was possibly due to high viscosity of the *Allanblackia*

Fig. 2 Effect of Methanol: *Allanblackia parviflora* oil molar ratio (reaction temperature 60 °C, catalyst amount 1% and reaction time of 1.5 h)

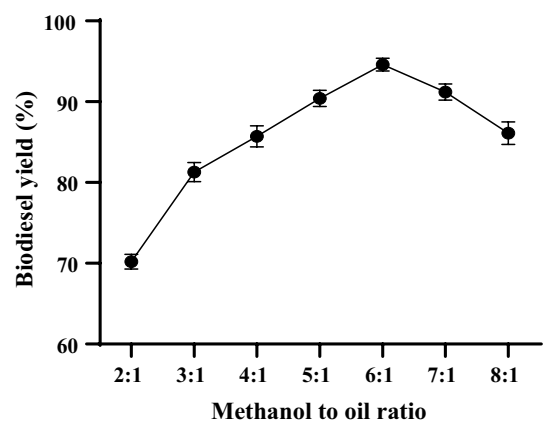
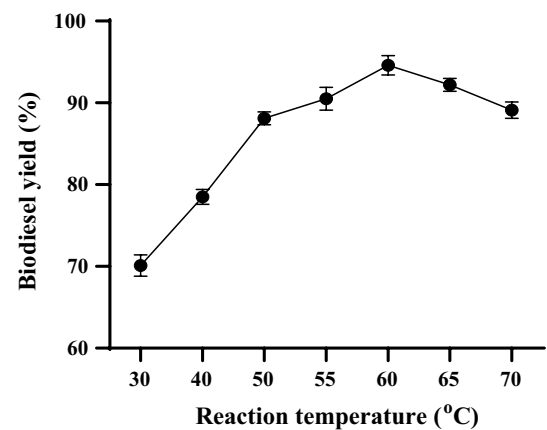


Fig. 3 Influence of the temperature of the reaction (1.5 h of reaction time, 6:1 methanol to oil molar ratio and 1%NaOH amount)



parviflora oil at the room temperature leading to incomplete reaction within the reactant (sodium hydroxide-alcohols) [34]. The phenomenon in this case contradicts earlier studies on similar subject [35]. However, the phenomenon agrees with others previous studies on related works [36].

3.2.3 Influence of the amount of catalyst on the yield

Figure 4 shows the influence of the amount of catalyst on the yield of biodiesel. Ideally, when the amount of catalyst is less, optimum yield of biodiesel cannot be achieved. Meanwhile, a concentrated amount of catalyst can also lead to saponification reaction. In this study, the effect of the amount of NaOH was investigated at 1.5 h, 6:1 methanol to *Allanblackia parviflora* oil molar ratio and 60 °C of reaction temperature. The amount of catalyst was varied from 0.25% to 2.0%. The figures in terms of the percentages were calculated based on the weight of the oil used for the study. When the amount of catalyst was adjusted from 0.25 to 1%, the yield of the biodiesel also risen from 70.7% to 94.6% respectively. However, additional upward adjustment of the amount of NaOH (above 1%) slightly reduced the biodiesel yield.

Previous studies [31, 32] have indicated that relatively, high amount of alkaline catalyst lead to saponification reaction in the presence of high free fatty acids which subsequently result in reduced yields. In the present study, this observation was noted at 1.25% amount of catalyst; which was attributed to the fact that the *Allanblackia parviflora* oil used was a bit crude and had little free fatty acid concentration (< 1%). The phenomenon recorded in this study agrees with previous study on similar subject [37].

3.2.4 Influence of duration of the reaction of the biodiesel yield

Figure 5 shows the influence of biodiesel yield on the duration of the reaction using *Allanblackia parviflora* seed oil. The duration of the reaction was varied in the range of 0.5 h to 2.5 h. From Fig. 5, it can be deduced that the yield of the

Fig. 4 Influence of the amount of NaOH (6:1 methanol to oil molar ratio, 60 °C reaction temperature and 1.5 h reaction time)

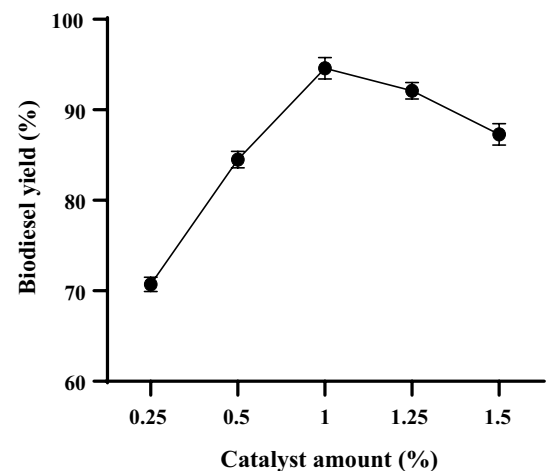
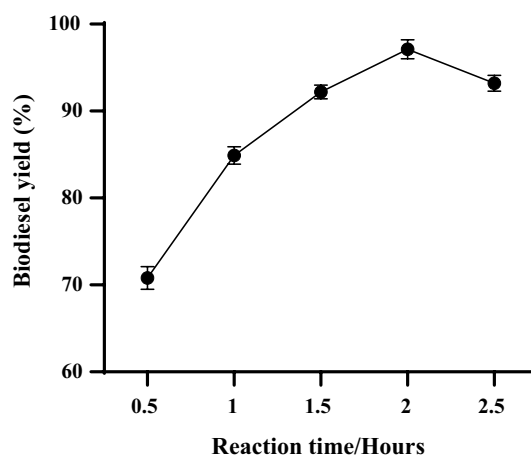


Fig. 5 Influence of reaction time (methanol to oil molar ratio of 6:1, reaction temperature of 60 °C and catalyst amount of 1%)



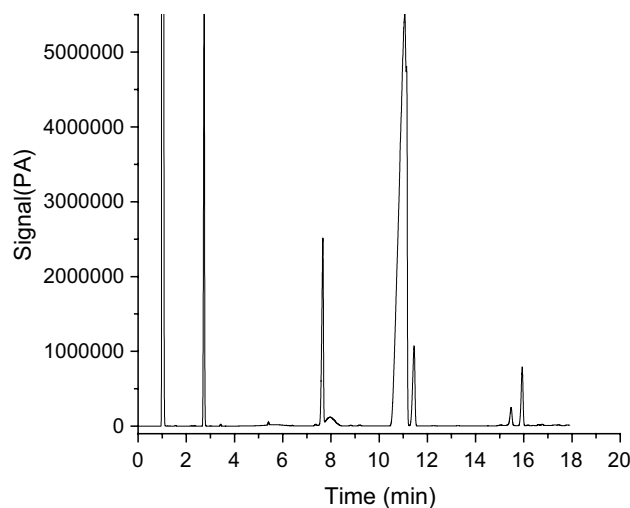
biodiesel was very much dependent on duration of transesterification reaction. At the beginning of the reaction (0.5 h), the yield was low due to mixing and the dispersion of methanol onto the oil. The maximum yield (97.1%) was obtained in 2 h. Further upward adjustment in the duration of the reaction of *Allanblackia parviflora* oil (after the optimum was reached) led to the reduction in the yield. The reduction in the yield after the optimum duration of 2 h was probably due to reversible reaction which might have taken place resulting in slight loss of esters [38]. The optimum duration of the reaction was therefore 2 h. Figure 6 shows the chromatogram of *Allanblackia parviflora* methyl esters at optimum reaction conditions while Fig. 7 shows the TGA curves of *Allanblackia parviflora* methyl esters in dry air atmosphere.

3.3 Characterization of *Allanblackia parviflora* methyl ester (APME)

The APMEs that were obtained at optimum conditions (reaction temperature of 60 °C, methanol: *Allanblackia parviflora* oil molar ratio of 6:1, reaction time of 2 h, catalyst amount of 1% NaOH (by wt) were subjected to characterization using GC. There was appearance of signal in the typical GC spectra (Fig. 6) after transesterification during the following time (min) and the corresponding peak (Intensity-Signal): 1 min—petroleum ether, 2.4 min—tetradecane, 7.6 min—C16:0, 11.0 min—C18:2, 11.2 min—C18:1 trans, 15.5 min—C18:0, 16 min—C20:0.

Fuel's ignition quality is affected by properties such volatility and thermal stability [39]. On the basis of the indication of the thermogram (Fig. 7), the thermal stability of APMEs were especially, between 100 and 160 °C. However, there was loss of mass between 160 and 900 °C. At 700 °C there was an optimum speed of mass loss. The mass loss happened due to combustion or by evaporation of the methyl esters specifically; methyl linoleate and oleate that were basically the major compounds. Similar behaviour has been shown by *Jatropha curcas* methyl esters [40].

Fig. 6 Gas Chromatogram of *Allanblackia parviflora* methyl esters (APME) (methanol to oil molar ratio of 6:1, reaction temperature of 60 °C, reaction time of 2 h and catalyst amount of 1%)



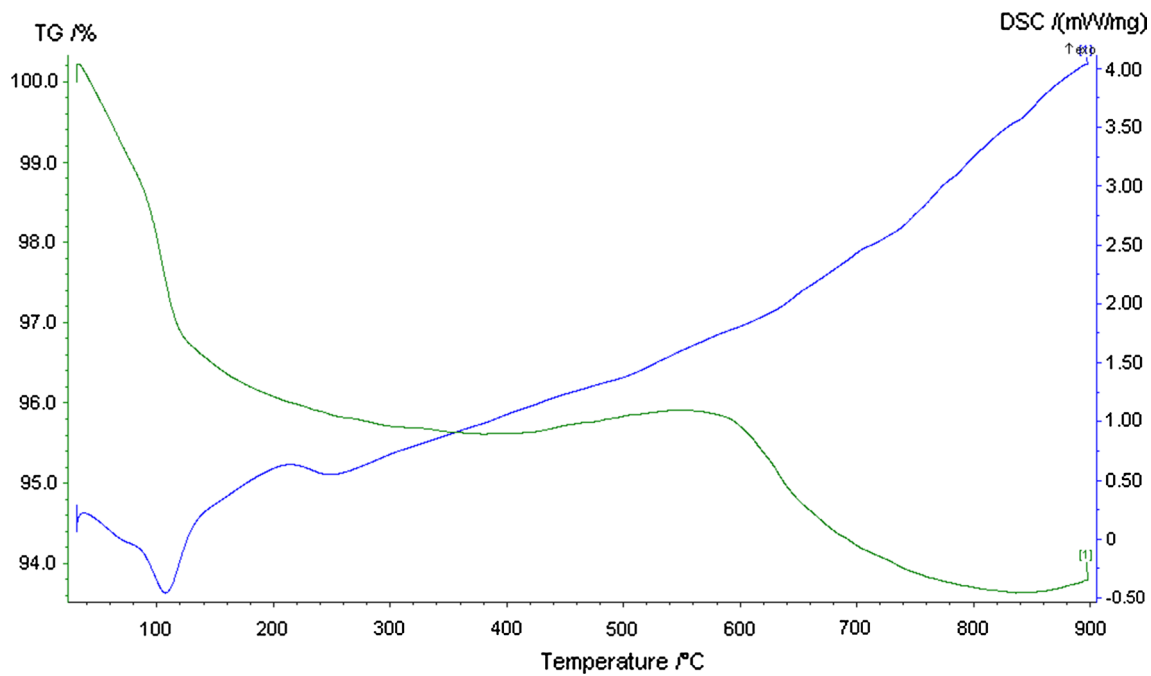


Fig. 7 TGA curves of *Allanblackia parviflora* methyl esters atmospheric condition

3.4 Properties of *Allanblackia parviflora* methyl ester (APME)

Fuel characteristic of the APME after determination were identified to conform to the international standard and are shown in the Table 1. The determination of the properties was to examine whether APMEs have sufficient values that could function as a candidate methyl ester. The movement of biodiesel through pipes, injector nozzles and orifices are affected by density [24]. The value of the density for APMEs was 867 kg/m^3 and this was considered lower when compared with the values of other internationally accepted oils such as jatropha. The density of the APMEs (867 kg/m^3) was lower than the values that were reported for other popular biodiesel such as rapeseed (882 kg/m^3), soybean (884 kg/m^3) and palm (876 kg/m^3) methyl esters [41].

Kinematic viscosity is a significant property of biodiesel since it influences the fluidity of the fuel. When biodiesel has a high viscosity, it simply indicates that there can be incomplete burning and this can lead to the formation of engine deposit and subsequently, soot formation [42, 43]. From the study, the viscosity of the biodiesel was reduced from $35.2 \text{ mm}^2/\text{s}$ (viscosity of the oil) to $4.74 \text{ mm}^2/\text{s}$. This value was within the range specified by ASTM D 6751 and EN 1425 ($1.9\text{--}6 \text{ mm}^2/\text{s}$) and was relatively less than viscosity values of biodiesels including sunflower ($4.43 \text{ mm}^2/\text{s}$) and soybean ($4.03 \text{ mm}^2/\text{s}$) [42, 43]. This implies that APMEs with this low viscosity can easily flow through the fuel system of diesel engines.

Table 1 Comparing the properties of the *Allanblackia parviflora* biodiesel to United States and Europe standards

Fuel property	<i>Allanblackia parviflora</i>	ASTM D6751	EN 14,214
Cetane number	50	≥ 47	≥ 51
Kinematic viscosity (mm^2/s ; $40 \text{ }^\circ\text{C}$)	4.74	1.9–6.0	3.5–5.0
Oxidative stability (h)	2.42	≥ 3	≥ 6
Pour point ($^\circ\text{C}$)	3	–	–
Flash point ($^\circ\text{C}$)	155	≥ 93	≥ 120
Sulfur value (% w/w)	0.002	≤ 0.05	0.020
Acid amount (KOH mg/kg)	0.422	≤ 0.5	≤ 0.5
Water content (mg/kg)	0.03	≤ 0.05	≤ 0.05
Density ($20 \text{ }^\circ\text{C}$)	867	–	860–900

Flash point of fuel is one of the most important property. Biodiesel's vapour will burst into flames immediately when exposed to fire or very high temperature (above 150 °C). The lower the biodiesel flashpoint, the less risky it is to transport. Table 1 indicates that the methyl esters obtained from the *Allanblackia parviflora* oil had a flashpoint of 155 °C, which is within the recommended range of ASTM D 6751 and EN 1425 (130 °C least) and this value is closer to those of soybean (178 °C), olive (178 °C) and peanut (176 °C) methyl esters [41].

The cetane number determines ignition quality of biodiesel. The higher the cetane number, the more ignitable the biodiesel will be. In this study, the cetane number of the biodiesel was 50, which is higher than the least acceptable cetane number of 47 by ASTM D 6751 and EN 1425. The cetane value of 50 in this case was higher than that of sunflower (45) biodiesel (49) [41].

The ash value is the measure of proportion of non-ignitable matter contained in fuel [5]. The *Allanblackia parviflora* biodiesel had negligible ash content of 0.002% by wt. The sulfur and carbon values of 0.003% and 0.02% respectively were within the range of ASTM D 6751 and EN 1425 [31].

4 Conclusion

Allanblackia parviflora oil was obtained from RYTE Aid Chemical (Ho, Ghana) with the amount of oil per seed determined to be 49%. The crude oil was transesterified to biodiesel using methanol in the presence of NaOH as the catalyst to obtain *Allanblackia parviflora* methyl esters (APMEs). Maximum yield of 97.1% was obtained at methanol to oil ratio of 6:1, reaction temperature of 60 °C, catalyst amount of 1% and reaction time of 1 h. The properties of the APMEs were within international standards. *Allanblackia parviflora* oil could be proposed for use as an oil feedstock for producing biodiesel in Ghana. When compared with other vegetable oil feedstocks such as, soybean, rapeseed and sunflower *Allanblackia parviflora* oil could be cheap non-food oil for biodiesel production.

Authors' contributions MT and PKE wrote the manuscript and RK prepared the tables and figures. All authors read and approved the final manuscript.

Availability of data and materials All the data for this research are embedded in the manuscript. The authors want to thank Micmod Foundation for their support and also acknowledge Dr Michael Miyittah for polishing the English language.

Competing interests The authors declare that there are no conflicts of interest.

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