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# Evaluation of African Star Apple (*Chrysophyllum albidum*) Seed Oil as a Potential Feedstock for Industrial Application

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***Authors' contributions***

*This work was carried out in collaboration among all authors. Author OMA designed the study, wrote the protocol and wrote the first draft of the manuscript. Authors ASC and IC managed the analyses of the study. Authors OMA, ASC and GKA managed the literature searches. All authors read and approved the final manuscript.*

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## ABSTRACT

**Aims:** Evaluate possible application of African Star Apple seed oil as industrial raw material geared towards possible conversion of waste to wealth with no negative impact as it borders on food scarcity. Also unveiling the possibility of a sustainable environment via the eradication of waste from the environment, thereby creating a clean environment.

**Place and Duration of Study:** Fresh ripped fruits of African Star Apple were bought from some local market sellers at Agbarha-Otor market which is located in Ughelli North Local Government Area of Delta State, Nigeria on longitude 6° 2' 54" E /5° 30' 40" N, between November, 2019 and March, 2020.

**Experimental Details:** Soxhlet extraction with n-hexane as solvent was used for the oil extraction of 100 g per batch of extraction. Pretreatment procedure of oil was done prior to transesterification. Homogenous base-catalyzed transesterification reaction was used in this study under controlled experimental conditions such as temperature, reaction time, methanol to oil molar ratio and

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catalyst type and concentration were examined under varied ranges to ascertain optimum experimental conditions for the reaction. Extracted seeds oil were analysed for its physicochemical properties using standard methods via: specific gravity, acid value, refractive index, saponification value, iodine value and free fatty acid using standard methods.

**Results:** The results showed an oil yield of 14.9%, an indication that the seed reflects a poor source of abundant oil. Results obtained for the physiochemical properties, revealed that; specific gravity, refractive index, saponification value, acid value, free fatty acid and iodine value showed values of 0.896 kg/m$^3$, 1.549, 231.32 mgKOH/g, 3.23 mgKOH/g, 2.07% and 47.63 mg/100 g respectively. Also, results for Methanol: Oil ratio showed that maximum yield was obtained at 6:1 molar ratio. Maximum conversion efficiency for molar concentration was achieved at 0.75% for both catalysts. While a reaction time of 125 min projected better yield.

**Conclusion:** The seed of African star fruit are discarded as waste, therefore its use as a source of oil for industrial feedstock and other domestic application, poses no challenge with regards to food security. Its non-drying potential based on its low iodine value, makes it suitable lubricating oil for industrial application. Similarly, potential application of the oil as feedstock for the production of biodiesel is justified on the basis of its low acid value. Conclusively, the seeds may not have sufficient oil volume potential to be used as edible (domestic) and industrial oil. Consequently upon its low yield, application for biofuel production in commercial scale becomes unrealistic.

*Keywords: Biodiesel; physiochemical; esterification; fatty acid; seed oil; esters; saponification.*

## 1. INTRODUCTION

Recent trend has shown that oil has become an important feedstock in industries for production of cosmetic, biofuel, bio-lubricant and other products [1,2]. This need has necessitated the search for alternative sources through biomass (oil seeds) [3,4]. Consequent upon the rising need in the demand for oil by both domestic and industries, the search for alternative underutilized seeds as sources of oil to supplement the already existing traditional sources of oil becomes necessary [5]. Fats and oils (lipids) are one of the important macromolecules of living organisms [6]. Its composition is made up of mixtures of organic molecules, which are mainly triacylglycerols, diacylglycerols, monoacylglycerols, free fatty acids and other minor components such as phospholipids, phytosterols, tocopherols and tocotrienols and hydrocarbons [7,8]. Based on the bond present, the fatty acid constituents are classified as saturated (no double bonds), monounsaturated (one double bond), and polyunsaturated fatty acids (multiple double bonds) [9]. Biodiesel also known as fatty acid methyl esters is considered an attractive alternative to conventional fuels based on its excellent fuel properties, biodegradable potentials, environmental compatibility, readily available and non-emission of poisonous gases [10-12]. Biodiesel is produced through a chemical process known as "transesterification" in which there is displacement of alcohol from an ester under acidic or basic catalytic conditions producing free glycerol and fatty acid esters of the respective alcohol as described in Fig. 1 [13,14].

Report has shown that both edible and non-edible oils have been successfully employed in biodiesel production [15-17]. But due to the rising challenge of food security across the globe, developing nations like Nigeria, whose crude oil is used mainly to produce conventional diesel, depend on alternative oil producing crops which can be utilized as feedstock, such as soybeans, groundnuts, cottonseeds, sunflower, rapeseeds, oil palm, coconut oil etc [18,19]. These oil sources form the major source of nourishment for the teaming populace, hence limiting its application. In southern Nigeria, *Chrysophyllum albidum* plant parts are used as remedy for bruises and wounds, due to its potency in stopping bleeding from fresh injuries, thereby preventing further contamination [20,21]. Sequel to the aforementioned, interest on the use of non-edible seed oil will provide a sustainable approach. African star apple (*Chrysophyllum albidum*) seed oil is an emerging competitor as a promising feedstock [22]. African star apple popularly called Udara and Agbalumo by native of Igbo and Yoruba respectively. *Chrysophyllum albidum*, belongs to the family of Sapotaceae which comprises of about 800 species [23]. The tree is evergreen with a projecting height up to 40m high [24]. Its straight and long fluted bole with small buttress at the base gives it a unique identity [25]. The fruit contains an average of three to five seeds which are non edible. The dark brown or blackish coat,

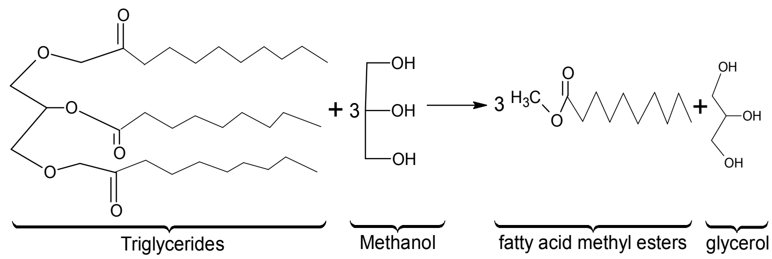


**Fig. 1. Transesterification reaction**

when broken reveals white coloured cotyledons [25]. The *Chrysophyllum albidium* seed oil is rich in both linoleic (36.0%) and oleic (37.6%) fatty acids [26]. The indiscriminate dumping of the seeds after consumption has posed severe environmental challenge of waste disposal, thereby creating breeding ground for harmful organism. Therefore this study aimed at possible conversion of waste to wealth with no negative impact as it borders on food scarcity upon its application in industrial production. Likewise the study unveils the possibility of a sustainable environment via the eradication of waste, thereby creating a clean environment.

## 2. EXPERIMENTAL DETAILS

### 2.1 Materials and Apparatus

Wij's solution, n-hexane and other chemicals were products of Sigma-Aldrich, USA. The methanol used (99% pure) is of analytical grade with boiling point of 78°C; while the NaOH, Potassium dichromate and Potassium iodide used were of analytical grade and purchased from Aldrich Chemical Co. Ltd. Sodium sulfate, Hydrochloric acid, starch, sodium thiosulphate, Phenolphthalein used were also an analytical grade product of Merck Co Ltd. Laboratory oven (DHG 9030) magnetic stirrer with hotplate (UNICON), three necks round bottom flask, measuring cylinder, beaker, separating funnel, burette, density bottle, funnels, pet-bottle thermometers and measuring flask were used.

### 2.2 Sample Collection and Treatment

Fresh ripped fruits of African Star Apple were bought from some local market sellers at Agbarha-Otor market which is located on longitude 6° 2' 54" E and Latitude 5° 30' 40" N in Ughelli North Local Government Area of Delta State, Nigeria. This is a seasonal fruit that is available during the dry seasons. The seeds after careful separation from the fleshy mesocarps, were first air-dried in the sun at an average temperature of 29°C for 5 days and then the dark brown shell of the separated seeds were cracked mechanically to de-coated the shell and subsequently unveiled the inner cotyledon which were sun-dried for 72 h, The dried cotyledon were further oven-dried at a temperature of 100°C for 24 hours in a laboratory oven. The dried cotyledon were ground into fine particle size using an electric blender and stored in an air-tight containers until needed for the experiment.

### 2.3 Extraction of Oil

The method employed by Betiku, et al. [27] was used for this study. Soxhlet apparatus (1-liter) and n-hexane as solvent were used for the oil extraction. One hundred grams (100 g) of the powdered *Chrysophyllum albidium* seeds were wrapped with whatmann filter paper and transferred into the thimble of the Soxhlet extractor. The thimble was carefully fixed on a 1-litre capacity round-bottomed flask. 700 ml of n-hexane (B.p. 40-60°C) was poured to about two-third of the volume of the flask and heated at 60°C on a thermostatically controlled heating mantle and allowed to reflux continuously for 4 hrs. Percentage oil yield was determined as expressed and replicate extraction processes were performed.

#### 2.3.1 Percentage oil content

$$\%\text{Yield} = \frac{\text{y1} - \text{y2}}{\text{y1}} \times 100$$

Where;

$y_1$ is the weight of *Chrysophyllum albidium seed powder* before extraction.
and $y_2$ is the weight of *Chrysophyllum albidium* seed powder after extraction.

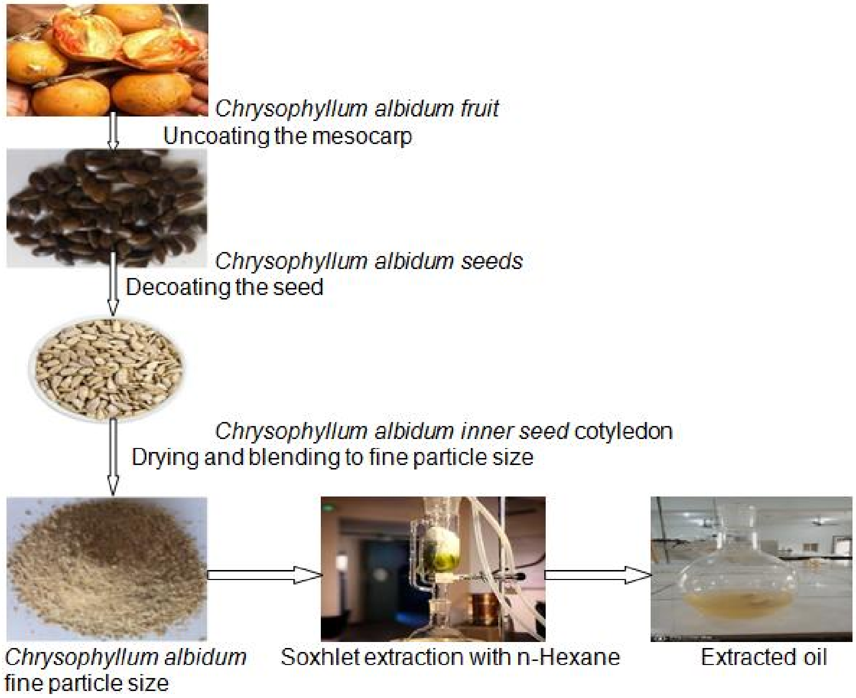


**Fig. 2. Flow chart for the extraction of *Chrysophyllum albidum* seed oil**

### 2.3.2 Pretreatment of the oil extract

This reaction was carried out using methanol and concentrated Sulphuric acid as catalyst prior to transesterification reaction. 40 ml of the oil was weighed into a 500 mL flat bottomed flask, 6 ml of methanol and 0.5% $H_2SO_4$ were added. The mixture was agitated at a high speed of 450 rpm and temperature of 60°C using magnetic stirrer within a reaction time of 2 hrs. The mixture was then transferred into a 50 mL separating funnel which later separated into three layers comprising water at the bottom, pretreated oil in the middle and methanol at the upper layer. The mixture was carefully separated by removing the water first, followed by the oil and finally the methanol. The pretreated oil was poured into a beaker and dried carefully in an oven regulated at a temperature of 105°C until the residual water evaporated off completely. After this process, the pretreated oil was made ready for transesterification [27].

### 2.3.3 Transesterification of oil

Homogenous base-catalyzed transesterification reaction was used in this study as described by Umer et al. [28]. The biodiesel production was carried out under controlled experimental conditions such as temperature, reaction time, methanol to oil molar ratio and catalyst type and concentration were examined under varied ranges to ascertain optimum experimental conditions for the reaction. The transesterification reaction was carried out using a 250 mL round bottomed flask equipped with thermostat, mechanical stirrer, sampling outlet and condensation system. About 50 g of the oil was initially heated to the set temperatures (30, 40, 50, 60 and 70°C) on a hot plate before the reaction. Freshly prepared methanolic solutions of NaOH and KOH at varying concentrations of 0.25, 0.50, 0.75, 1.00 and 1.25% (based on the weight of the oil) were added to the oil. And the experiment was conducted at a reaction time for 120 min at varying temperatures, methanol to oil molar ratio (2:1,4:1, 6:1,8:1,10:1 and 12:1). These parameters were adopted to determine the best experimental conditions for complete conversion of the seed oil into fatty acid methyl ester (biodiesel).

## 2.4 Phase Separation and Purification of Biodiesel

After a complete transesterification process, the mixture was allowed to stand for 24 hrs to ensure proper settling and easy separation of the various portion of the mixture. The ester left in the separating funnel was washed thrice with warm water to remove residual catalyst. Complete removal of the catalyst from the ester was made certain by determining the amount of potassium salt in the glycerin phase and the total potassium removed from the ester phase. The sum of these accounted for all of the potassium entering the reaction as a constituent of the catalyst. The washed ester (biodiesel) was poured into a 250 mL beaker, placed in an oven set at 105°C and dried for 2 hrs. Finally, volumes of the biodiesel and percentage yields were noted. The procedure was repeated using NaOH catalyst [27,28].

## 2.5 Physiochemical Properties

*Chrysophyllum albidium* seeds was analyzed for its physicochemical properties using standard methods via: specific gravity (ASTM D4052), acid value (ASTM D8045), saponification value (ASTM D5558), iodine value (ASTM D4607) and free fatty acid using standard methods [29].

### 2.5.1 Specific gravity

Capillary stopper relative specific gravity bottle (pyknometer bottle) of 50 ml capacity was used to determine specific gravity of biodiesel. This parameter helps to ascertain the weight of the oil in comparison to standard fluid.

$$\text{Specific Gravity at} \frac{30°\text{C}}{30°\text{C}} = \frac{\text{A} - \text{B}}{\text{B} - \text{C}}$$

Where:

A = Weight in g of the specific gravity bottle with sample at 30°C,
B = Weight in g of empty specific gravity bottle,
C = Weight in g of specific gravity bottle with distilled water at 30°C.

### 2.5.2 Acid value

The acid value is an important indicator of oxidation of the oil. The number of NaOH required to neutralizing the free fatty acid present in 1.0 gm of the sample.

$$\text{Acid Value} = \frac{5.61 \text{ x V}}{\text{W}}$$

Where;

V = Volume in ml of 0.5N NaOH required for titration in ml.

W = Weight in g of sample taken.

### 2.5.3 Saponification value

Saponifcation value (SV) is related to the average molecular mass of fatty acid in the oil sample. A known quantity of oil is refluxed with an excess amount of alcoholic KOH. After saponification, the remaining KOH is estimated by titrating it against a standard acid.

$$\text{Saponification Value} = \frac{28.05\ (\text{V2} - \text{V1})}{\text{W}}$$

Where:

$V_2$ = Volume in ml of 0.5N acid required for the blank.
$V_1$ = Volume in ml of 0.5N acid required for the sample.
W = Weight in g of the sample taken.

### 2.5.4 Iodine value

The iodine value is a measure of the degree of unsaturation of oils and determines the stability to oxidation. Standard AOAC [30] official methods of analysis and Wij's iodine method as described below was used: 0.52 g of oil sample was dissolved in 10 ml of cyclohexane. 20 ml of Wij's solution (Iodine monochloride) was added, the stopper flask was allowed to stand for 30 min in the dark at room temperature, and 20 ml of 10% potassium iodide solution was added. The resulting mixture was then titrated with 0.1 M $Na_2S_2O_3$ using starch as indicator. Iodine value was calculated using equation [31-33].

$$\text{Iodine Value} = \frac{[\rho\text{o} - \rho] \text{ x M x } 12.69}{\text{W}}$$

where

M = concentration of sodium thiosulphate used;
$\rho^o$= volume of sodium thiosulphate used as blank;
$\rho$ = volume of sodium thiosulphate used for determination.
W = Weight in g of the material taken for the test.

### 2.5.5 Determination of % free fatty acid (FFA)

The % FFA of the hydrolysis of the seed oil would be determined according to AOAC, [30]. Approximately 50 mL of isopropanol would be placed into the flask, and about 0.5 mL phenolphthalein was added and then neutralised by addition of 0.02 N sodium hydroxide (NaOH) until a permanent pink colour appeared. The neutralized isopropanol was then added to the 5 g of FFA, thereafter transferred into an Erlenmeyer flask, and about 0.5 mL of phenolphthalein was added, preceded by gentle shaking. The mixture was neutralised when the first permanent pink colour was obtained by the addition of 0.02N NaOH. The percentage FFA was calculated by using the equation.

$$\%\ \mathrm{FFA} = \frac{28.2 \times \mathrm{N} \times \mathrm{V}}{\mathrm{W}}$$

## 3. RESULTS AND DISCUSSION

### 3.1 Physiochemical Characteristics of Oil Feedstocks

The oil extracted from the African Star Apple seeds (*Chrysophyllum albidum*) with hexane using the soxhlet extractor was analyzed and the following physiochemical results are presented in Table 1.

The oil extracted, depict a honey-like colouration that is dark brown. The result showed that the oil yield was high with a value of 14.92%, higher than an average value of 10.71% reported by Peter et al. [34] and 12% by Akubugwo, Ugbogu [35]. Also in other findings, reports of 8.05% and 12.70% [36] was also below the reported value in this study. The oil yield 14.9% obtained for African star apple seed in this study was found to be lower than 16.8% reported by Agbede et al. [37]. Due to the low yield of oil, it shows a clear indication that the seed reflects a poor source of abundant oil. Consequently its application at industrial production scale becomes a limitation. The low oil yield as reported in other findings could be attributed to variation in several factors such as: climate, plant species, soil condition, and improper processing techniques [38].

The ability of oil to flow in an automobile engine is an important property ascertained based on its specific gravity. Therefore, considering the value of 0.896 obtained from this study showed that the results are within acceptable ranges for biodiesel according to report by Enweremadu, Alamu [39]. Also in other findings, result obtained at 30°C is in agreement with values of 0.89 at 25°C, 0.8269 at 25°C as reported by other authors [34,40], but fall below 0.92 as reported by Michael et al. [41].

The refractive index of the oil obtained at 25°C, indicated a value 1.549. This parameter indicates the level of optical clarity of the biodiesel oil sample relative to water [41]. Report from this study were above most non-drying oils with refractive index ranging between 1.475 -1.485 as reported by Akinhanmi et al. [39] and also above 1.464 at 30°C in other study [41]. This value is lower than 1.672 at 31.2°C reported in other findings [34]. The higher refractive index indicates thickness of the *Chrysophyllum albidum seed* oil over the non-drying once.

Saponification value (SV) unveils the average molecular mass of fatty acid in the oil sample that makes it a suitable parameter in determining its application in soap making [42,43]. The average saponification value obtained revealed 231.32 mg/KOH/g which is higher than 199.50 mg/KOH/g, 200 mg/KOH/g and 90.71 mg/KOH/g obtained for Adebayor, Orhevba [34], Michael et al. [41] and Ominyi, Ominyi [40] respectively. However research from other findings is within range with value of 228.4 mg/KOH/g [44] and 236.341 mg/KOH/g [45], but lowers than 327 mg/KOH/g in other report [37]. The results have attributed this property as good feedstock in production of liquid soap, shampoos, and lather shaving creams [46,47].

**Table 1. Physiochemical properties of the extracted *Chrysophyllum albidum* seed oil**

| Physiochemical property | Reported values |
|---|---|
| Oil content (%) | 14.92 |
| Specific gravity | 0.896 |
| Refractive Index | 1.549 |
| Saponification Value (mgKOH/g) | 231.32 |
| Acid Value (mgKOH/g) | 3.23 |
| Free Fatty Acids (%) | 2.07 |
| Iodine Value (mg/100 g) | 47.63 |

According to Michael et al. [41], low acid value (< 0.1) is attributed to low oxidation with no possibility of gum and sludge formation. The acid value from this study was found to be 3.23 mg/KOH/g. This value is slightly higher than 2.57 mg/KOH/g by Musa, Isah [44], but lower than 7.72 mg/KOH/g [41], 4.50 mg/KOH/g [34] and 19.70 mg/KOH/g in other findings [40]. A measure of freshness of oil based on its quality has been reported to be a function of the acid values, hence unveiling the degree of rancidity [44,45].

The result obtained for the free fatty acid content records 2.07 mg/KOH/g of the extracted oil. Result from this study is in agreement with 2.25 mg/KOH/g in other findings [34]. However result from this finding is below 9.90 mg/KOH/g and 8.75 mg/KOH/g as reported in other study [40,45]. The low free fatty acids content of this oil, reflect low possible enzymatic hydrolysis, thereby presenting a better shelve life for the oil as reported by Mahale, Goswami-Giri [46].

The degree of unsaturation is a property of oil based on the iodine value [47]. The iodine value of 47.63 mg/100 g for this study was higher than values of 35 mg/100 g and 29 mg/100 g [34,45]. Report from this findings was higher than values of 30 mg/100 g by Musa, Isah [44], however was lower than values obtained by Ominyi, Ominyi [40]. A low iodine value below 100 mg/100 g is an indication the oil is nondrying [47].

To optimize transesterification conditions, the influences of methanol to oil molar ratio, temperature, catalyst amount, and reaction time on the biodiesel yield were examined.

### 3.2 Effect of Methanol/Oil Molar Ratio on the Yield of Biodiesel of *Chrysophyllum albidum* Seeds Oil

The influence of molar ratio on transesterification reaction with *Chrysophyllum albidum* seed oil was studied using six different molar ratio of 2:1,4:1, 6:1,8:1,10:1 and 12:1 methanol/oil, represented in Fig 3. Both catalyst shows periodic level of increase, up to a molar ratio of 6:1. This result showed an attribute that reflect that methanol at the initial stage of the transesterification process was sufficient to hydrolyze the triglyceride linkage [48]. However, above molar ratios of 6:1, the result showed little or no impact on the biodiesel yield. The emulsifying potential of methanol could be responsible for the periodic decline in the ester yield at high volume due to its ability to bind both the ester and the water phase, subsequently resulting to difficulty in separating the ester layer from the water phase [49]. Results from this finding is in agreement with report from other study [50,51].

### 3.3 Effect of Catalyst Type and Amount

Base-catalyzed transesterification was done using KOH and NaOH at varied concentration. The effect of each alkali concentration was evaluated in projecting a comparative study on the best alkali to use. In this study, the amounts for each alkali were varied and tested with other factors maintained as constant to evaluate the impact of each component towards the yield in biodiesel production. Therefore results from this finding revealed that the biodiesel yield increased when the concentration of the catalyst increased. But beyond 0.75%, the yield begins to decline as shown in Fig 4. This decrease could be as a result of saponification since excess catalyst favoured saponification reaction, thereby enhancing the formation of an emulsion with subsequent decrease in the biodiesel yield [52-54]. Result from this study is slightly higher than maximum biodiesel yield of 0.6% NaOH reported in Soursop Seed Oil and 0.5% of NaOH in rubber seed oil in other study [52,55]. The maximum conversion efficiency is achieved at 0.75% for both catalyst. However the result revealed that NaOH showed better conversion efficiency that KOH.

### 3.4 Effect of Temperature on the Yield of Biodiesel of *Chrysophyllum albidum* Seed

Effect of temperature as it affects biodiesel production using *Chrysophyllum albidum* seed oil is presented in Fig. 5. Catalyst amount of 0.75% and a Methanol/oil molar ratio of 6:1 were held constant since it reflects the best condition in this study for biodiesel production. The figure shows that higher temperature reflects consequent increases in biodiesel yield of 78.54% yield. However subsequent decline in the yield was observed for both catalyst at higher temperature above 60°C. This trend is in agreement with report in other findings that explained the subsequent decrease in biodiesel yield at higher temperature based on the assertion that saponification reaction of triglycerides is favoured at such prevailing condition. [56,57]. Results from this study is in agreement with report in other findings, but falls below ranges of 90-95%

biodiesel obtained for Jatropha oil [58]. Some studies have shown that the saponification of triglycerides by alkali catalyst is much faster than alcoholysis at temperatures above 60°C [57,58]. Also possible evaporation has been reported to be responsible for loss of methanol, subsequently leading to reduced biodiesel yield [57]. Hoque et al. [51] in their study with cooking oil at 65°C showed a maximum biodiesel yield of 87.6% which was higher that reported value in this study.

### 3.5 Effect of Reaction Time on Biodiesel Yield

Fig. 6 showed the variation in the yields of *Chrysophyllum albidum* biodiesel with reaction time. At a reaction time of 25 min, the results

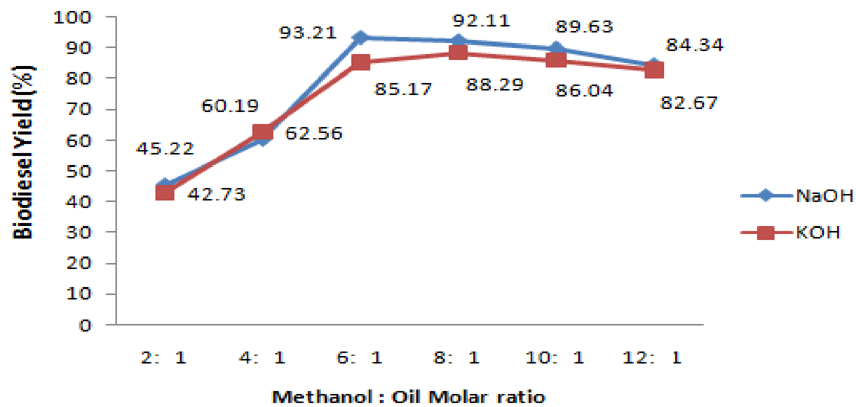


**Fig. 3. Effect of methanol: Oil molar ratio on biodiesel yield**

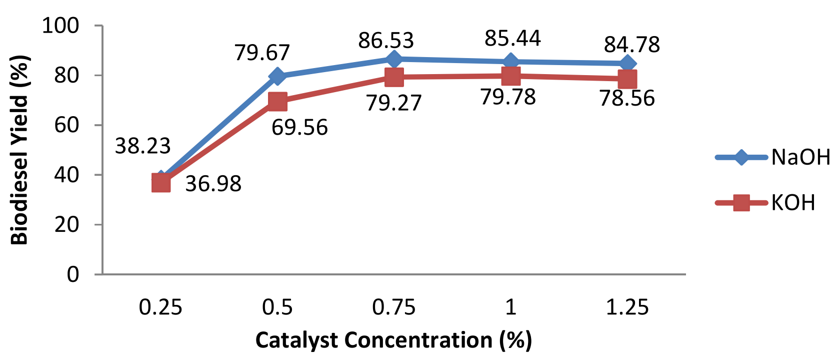


**Fig. 4. Effect of catalyst type and amount on biodiesel production**

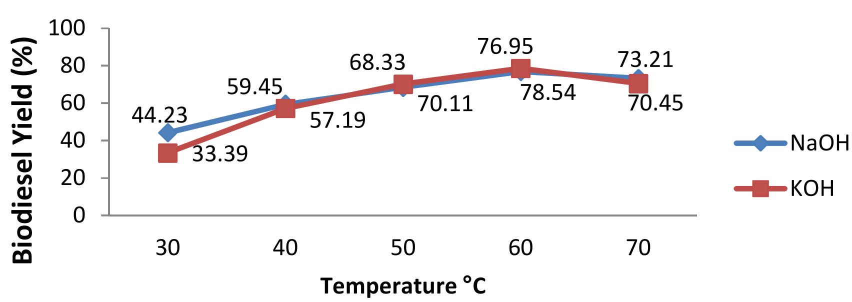


**Fig. 5. Effect of Temperature on the yield of biodiesel**

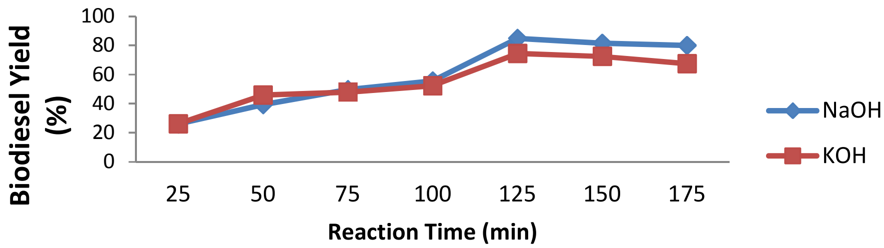


**Fig. 6. Effect of reaction time on biodiesel yield**

reveals a slow start up process of conversion rate of 25.97% biodiesel yield. This result follows similar trend as reported in other findings [50,59]. Gradual increase was observed with maximum yield recorded at 125 min. However at higher reaction time above 125 min, gradual decrease in the biodiesel yield was gradually been observed. This observation was in line with report findings that soap formation at higher reaction time could be responsible for the decline in biodiesel yield [50,57].

## 4. CONCLUSION

*Chrysophyllum albidum* seed oil show relatively low oil yield of 14.92%. The extracted oil has a consistent liquid phase at room temperature without any trace of precipitation unlike some other vegetable oil. The oil is also characterized with a dark-brown honey-like colour. The seed of African star fruit are discarded as waste, therefore its use as a source of oil for industrial feedstock and other domestic application, poses no challenge with regards to food security. Sequel to the aforementioned, report from this study has unveiled potential properties that can be harnessed towards divers applications. Its non-drying potential based on its low iodine value, makes it a suitable lubricating oil for industrial application. However, it may not be suitable for oil paint, and surface coatings due to its non-drying nature. Similarly, potential application of the oil as feedstock for the production of biodiesel, is justified on the basis of its low acid value. Conclusively, the seeds may not have sufficient oil volume potential to be used as edible (domestic) and industrial oil. This shows limitation in the application for diodiesel production in commercial scale.

## COMPETING INTERESTS

Authors have declared that no competing interests exist.

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