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# Production and Characterization of *Chrysophyllum Albidum* Seed Oil Derived Bio-lubricant for the Formulation of Oil-Based Drilling Mud

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***Abstract:*** *The application of diesel oil as the continuous phase of drilling mud is widespread when drilling through sensitive producing formation and troublesome shale zone. Due to the increase in environmental issues associated with the disposal of used fossil diesel, drilling companies are in recent times exploring options of use of environmentally friendly degradable oil as based for drilling fluids formulation. In this study, bio-lubricant also called polyol ester was formulated from biodiesel also called CAOFAME (chrysophyllum albidum oil fatty acid methyl ester) synthesized from vegetable oils extracted from chrysophyllum albidum (African star apple) fruit seed. The study also characterized the oils extracted against other vegetable oils. In addition, physiochemical properties of petroleum diesel oil, CAOFAME and polyol ester were examined against ASTM standards for purposes of characterization. The findings showed pH of acidic mixture on all four fluid types within the range of 4-6. Likewise, all four fluids densities and viscosities compared favourably with standards in the range of 0.83-0.93 g* $cm^{-3}$ *and 50.3-60.6* $mm^2s^{-1}$ *respectively. Other properties of pour point, flash point compared favourable with standards in comparison to diesel oil. In addition, the polyols produced with the oils from chrysophyllum albidum seed possesses lowest of toxicity and coupled with its biodegradable qualities, hence more environmentally friendly. Consequently, it is appropriately recommended as most suitable alternative to diesel oil as the continuous phase component in the formulation of oil-base drilling fluid.*

***Keywords:*** *chrysophyllum albidum seed oil, biodiesel, bio-lubricant, environmentally friendly*

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## I. Introduction

The use of petroleum diesel lubricants as base component for oil-based mud in the drilling industry continues to negatively impact the environment and this has resulted into both acute and chronic impacts on the ecosystem. The need to find a safer, more environmental friendly, economical, renewable lubricant that meets drilling industry lubrication standards becomes imperative. Plant-based oils are showing great potential and are highly attractive candidate to replace the conventional mineral oils for the use in lubricant production because they are structurally similar to hydrocarbons mineral oils but with renewable characteristics and lesser costs on product's life-cycle due to less maintenance, storage and disposal requirements. Reduces public exposure to toxins in the environment, consequently enables improved water quality for human and aquatic life. Also has no contribution to greenhouse gas emissions[1].

Recent studies are exploring the synthesis of plant-based oils to bio-lubricants for drilling fluid production[1-4]. Bio-lubricants, also known as bio-based lubricants or bio-lubes, are lubricants made of biological products or biomass from a variety of vegetable oils, such as rapeseed, canola, sunflower, soybean, palm, and coconut oils[5,6]. Vegetable oils are chemically triglycerides of fatty acids, and have excellent qualities like enhanced flash and fire points , higher viscosity and viscosity index, high biodegradability, high lubricity and very less toxicity[7]. There products are renewable, genetically changeable and nontoxic to humans and aquatic lives. Bio-lubricants applies to all lubricants that biodegrade rapidly and which are non-toxic for human beings and aquatic habitats. They may be based on oils extracted from seeds of plants such as palm kernel oil, canola oil, soybean, castor oil, moringa oil, and jatropha seed oil or synthetic esters produced from modified oils mainly derived from seeds of plants[3]. The use of bio-lubricants is particularly relevant to certain environments in which environmental protection is a constant concern. This is particularly true of aquatic, agricultural and forest environments. Bio-lubricants costs are lower due to the fact that they are locally produced and environmentally accepted[1-3]. Thus the study was aimed at syntheses and characterization of bio-lubricant from modified oils of *chrysophyllum albidum* (African star apple) seeds to reduce the effects of environmental impact of waste generated from drilling operations.

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**African star apple**

The African star apple (c*hrysophyllum albidum*) (commonly known as white star apple or African apple) is a forest fruit tree found throughout tropical Africa significantly in western, eastern and southern parts of Nigeria. It is a large berry containing 4-5 flattened seeds or sometimes fewer due to seed abortion. While the fruit is edible the seed is often discarded and randomly thrown away[8].

**Figure 1: The African star apple fruits (left) and seeds (right)**

**Bio-lubricant production**

There are two key feedstock for the bio-lubricant production. They are the vegetable oils extracted from seed plants of rapeseed, sunflower, soybean, palm oil, coconut, jatropha, karanja, rubber and this in study the *chrysophyllum albidum* and alcohol of either methanol, ethanol or butanol depending on cost, sustainability and performance characteristics[6,9,10]. However, methanol is much cheaper than the others and can easily be recycled[11]. In bio-lubricants production, fatty acid mono-alkyl ester (FAME) otherwise known as biodiesel is first synthesized from the free fatty acids (FFA) in the vegetable oils with methanol, after which the mixture is formulated into a bio-lubricant.

**Biodiesel synthesis**

The process of biodiesel also called fatty acid mono-alkyl ester (FAME) synthesis is known as transesterification. It involves a chemical reaction between vegetable oils FFA and the excess short-chain alcohol in the presence of a suitable catalyst (acid, base or enzymes) yielding glycerol as by-product[10] according to the reaction in Figure 2.

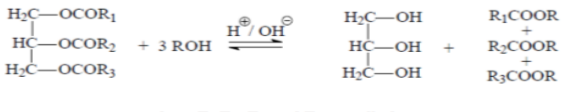


**Figure 2: Transesterification of triglycerides in oil to Biodiesel/FAME (Onoji *et al*[17])**

In oils with low FFA (~ 0.8wt. %), homogeneous base catalysts such as KOH or NaOH are applicable for transesterification. While if the FFA is higher, the homogeneous base catalysts will form soap which is not a desired product[12,13]. Hence, esterification will be required and an acid catalyst such as $H_2SO_4$, HCl or $H_3PO_4$ to initially reduce the FFA content according the reaction in Figure 3, before proceeding with transesterification in Figure 2 using either KOH or NaOH catalyst to produce FAME and glycerol[14].

$$\underset{\text{FFA}}{RCOOH} + \underset{\text{Methanol}}{CH_3OH} \xrightleftharpoons[]{\substack{H_2SO_4 \\ \text{(Catalyst)}}} \underset{\text{Methyl ester}}{RCOOCH_3} + \underset{\text{Water}}{H_2O}$$

**Figure 3: Esterification of FFA to Methyl Ester (Ebtisam *et al*[1])**

This study was thus aimed at developing an environmentally friendly bio-based oil from vegetable oils of locally sourced c*hrysophyllum albidum* seed suitable as an alternative to the conventional fossil diesel oil with adequate potentials as the continuous phase component in oil-based drilling fluid formulation.

## II. Methodology

The materials used in the study included seeds of African star apple purchased from domestic market, n-hexane 98% (analytical grade BDH chemicals), methanol 99.8% (analytical grade BDH chemicals), potassium hydroxide pellet 95% (analytical grade BDH chemicals), phenolphthalein indicator, Soxhlet apparatus, a muslin cloth, 0-100$^o$C thermometer, rotary evaporator model N-1110S (Rikakikai, Tokyo), measuring cylinders, separating funnels, pycnometer, titration kit and whatman filter paper.

**_Chrysophyllum albidum_ seed oil production**

Fresh seedsobtained from*chrysophyllum albidum* fruits were washed for 3–4 times in warm water to get rid of impurities such as dust, a covering creamlike colloidal fluid and an outer transparent film coating material. The seeds were sun dried at ambient temperature for about 24 h to remove moisture prior to crushing. About 500 g washed seed was cracked open and de-shelled manually removing its outer brown coloured hard pericarp using laboratory mortar and pestle to free the creamy coloured kernels from the shells. 100 g of oval shaped inner kernels was dried for 4 h at 105 $^o$C in an oven to flake for grinding as described in Bhuiya *et al*[15]. Dried *chrysophyllum albidum* seed kernels were ground and milled to about 2.5mm mesh size prior to extraction.Solvent extraction was carried out using50 g of powdered *chrysophyllum albidum* seed packed in a muslin cloth and placed in a thimble of the Soxhlet extractor as described in Awolu & Layokun[16]. The extraction was carried out at 60 $^o$C using n-hexane charged in round bottomed flask. The mixture was thereafter concentrated at 65 $^o$C using a model N-1110S rotary evaporator, to recover the extracted oil as described elsewhere[17].

**_Chrysophyllum albidum_biodiesel synthesis**

The materials required for the synthesis of *chrysophyllum albidum*oil biodiesel (CAOBD) also known as *chrysophyllum albidum*oil fatty acid methyl esters (CAOFAME) included*chrysophyllum albidum* seed oil, 98% $H_2SO_4$ as catalyst for FFA reduction in esterification reaction and 99% KOH catalyst for transesterification reaction**.** Based on the free fatty acid (FFA) value of oil> 1%, the *chrysophyllum albidum* seed oil required esterification for FFA reduction and avoid soap formation. Consequently, esterified *chrysophyllum albidum*oil ester was prepared using 100 mL *chrysophyllum albidum* seed oil with 45 mL of analytical grade methanol (>99%) in the presence of 1.5 % vol/vol $H_2SO_4$ catalyst at 60 $^o$C for 1 h as described in Betiku & Ajala[12]. The esterified oil was separated from the water and excess methanol by a model N-1110S rotary evaporator. CAOFAME was prepared using purified esterified oil and methanol with a homogeneous catalyst of potassium hydroxide in a transesterification process, followed by several purification steps as described elsewhere [1,18]. The esterified oil-to-methanol ratio was 3:1 in the presence of 0.5% w/w of the oil KOH catalyst at 60$^o$C for 2h. The prepared CAOFAME was separated from glycerol and any unreacted methanol in the mixture using a separating funnel.

**Bio-lubricant synthesis experimental procedure**

The materials used for the synthesis of bio-lubricants also referred to as polyol esters included a trimethylolpropane (TMP), sodium methoxide and the major feedstock *chrysophyllum albidum*oil fatty acid methyl esters (CAOFAME) from *chrysophyllum albidum* seed oil.Polyol ester synthesis was achieved by transesterification of the CAOFAME with TMP using sodium methoxide (in 30% methanol) as catalyst at 150$^o$C for 3 h as described in Ebtisam *et al*[1]. The weight ratio of CAOFAME-to-TMP was 3.5:1, while the amount of catalyst used was 0.8% w/w of the total reactants. After the reaction mixture was cooled to room temperature, the catalyst vacuum filtered and polyol obtained via fractional distillation. The polyol (biolubricant) was analyzed using ASTM standard method[19].

**Fluid property characterisation**

The characteristics of the *chrysophyllum albidum* seed oil, fatty acid methyl ester, and bio-lubricant produced and for comparative purposes that of petroleum diesel oil, were evaluated using their physiochemical properties determined. These properties which would provide indications on the desirability of materials, were all determined with ASTM standard procedures. Physical appearances of colour was based on ASTM D2622, while density/specific gravity at 15°C was based on ASTM D4052. Kinematic viscosities in cSt measured at 40 and 100 °C were based on ASTM D445 and D446 respectively, while flash point (°C), pour point (°C), acid value (mg KOH/mg) and % FFA were based on the ASTM methods D92, D97 and D664 respectively[1,3,18].

## III. Results and Discussions

**Table 1. Demographic Data Distribution on Sampled Teachers**

| Parameters | Refs.[1, 5, 10, 20, 21] | *chrysophyllum albidum* oil[22] |
|---|---|---|
| Colour | Golden yellow to dark brown | Light brown |
| Yield (%) | - | 30.6 ± 0.1 |
| pH | 3-7 | 6.0 ± 0.14 |
| Density, g/cm$^3$ @ 25 $^o$C | 0.857–0.945 | 0.924 ± 0.012 |
| Specific gravity @ 15 $^o$C | 0.857–0.945 | 0.924 ± 0.012 |
| $^o$API gravity @ 15 $^o$C | 33.61–18.24 | 21.64 ± 0.11 |
| Iodine value, g $I_2$/100 g oil | 40–146 | 68.26 ± 0.13 |
| Saponification value, mg KOH/g oil | 183.91–235.28 | 205.42 ± 0.28 |
| Acid value, mg KOH/g oil | 1.68–42.41 | 2.30 ± 0.02 |
| Free fatty acid (%FFA) | 0.84–42.41 | 1.15 ± 0.011 |
| Kinematic viscosity, mm$^2$/s @ 40 $^o$C | 6–66 | 60.38 ± 0.31 |
| Flash point, $^o$C | 72–295 | 88.5 |
| Pour point, $^o$C | –15 to +23.6 | 4 |

Ebitisam*et al*[1]; Karmakar*et al*[5]; Onoji *et al*[10];Aravind *et al*[20]; Reshad *et al*[21]; Igbafe[22] (This study)

**Physiochemical property analysis of *chrysophyllum albidum* seed oil**

The physiochemical properties of the *chrysophyllum albidum* seed oil are shown in Table 1. The colour of the seed oil was light brown which falls within the colour range with other vegetable oils from seed plant. The pH value of the oil was 6.0 which compares favourably with castor oil (6.8) and luffa cylindrica seed oil (3.93) (Asuquo *et al*[23]) as well as others in the 3-7 range shown in Table 1. This observed pH compares with other vegetable oils having significant amount of free fatty acids capable of forming soap, hence esterification was required[13]. The density at 25 $^o$C was 0.924 g/cm$^3$, which agrees with similar oils with values less than that of water. In like manner the specific gravity at 15 $^o$C and the $^o$API gravity of 0.924 and 21.64 are both of satisfactory levels and similar to values of other vegetable oils.The oil iodine value of 68.26 g $I_2$/100 g oil was less than the range specified for semi-drying oils[23]. This may be attributed to low amount of unsaturated fatty acid content in the oil. The saponification value indicates the amount of alkali (KOH or NaOH) required to convert the oil into soap and it is an index of the average molecular mass of fatty acid in the oil[24,25]. A saponification value of 205.42 mg KOH/g oil obtained for *chrysophyllum albidum* seed oil lies between those of sunflower (186 mg KOH/g oil), coconut oils (265 mg KOH/g oil) and palm oil (195–205 mg KOH/g oil )[20,26,27]. The acid value of *chrysophyllum albidum* seed oil was 2.30 mg KOH/g oil, which indicates acceptable free fatty acids suitable to be consumed as food being ≤ 10 mg KOH/g oil allowed for edible oils and no de-acidification refining required[10].The average kinematic viscosity of the oil determined was 60.38cp mm$^2$/s. This is lower compared to oils of jatropha curcas L. (77.4 mm$^2$/s) but higher than that of conventional diesel (<5 mm$^2$/s)[21]. Hence the resistance of *chrysophyllum albidum* seed oil to flow is higher than required. The flash point is high enough to reduce its volatility at ambient conditions but not adequate for operations at 90$^o$C and above. The pour point is the lowest temperature at which the oil starts to solidify and become deprived of its flow ability[28]. The *chrysophyllum albidum* seed oil had a value of 4 $^o$C which is same as for jatropha oil (4 $^o$C)[21,29]. This suggests that the oil can perform adequately under temperate climatic conditions.

**Table 2: Physicochemical properties of FAME from vegetable seed oil**

| Parameters | Biodiesel [1, 2, 17] | *chrysophyllum albidum* (CAFAME)[22] |
|---|---|---|
| Colour | Golden yellow – brown | Light brown |
| Yield (%) | - | 38.3 ± 0.14 |
| pH | 6–8 | 6.12 ± 0.10 |
| Density, g/cm$^3$ @ 25 $^o$C | 0.820–0.92 | 0.835 ± 0.021 |
| Specific gravity @ 15 $^o$C | 0.820–0.92 | 0.835 ± 0.02 |
| $^o$API gravity @ 15 $^o$C | 41.06–22.30 | 37.30 ± 0.13 |
| Kinematic viscosity, mm$^2$/s @ 40 $^o$C | 4.58–66.13 | 59.60 ± 0.22 |
| Pour point, $^o$C | –3 to 6 | 5 |

| | | |
|---|---|---|
| Flash point, °C | 72–295 | 82 |

Ebitisam *et al*[1]; Falode & Adegoke[2]; Onoji *et al*[17]; Igbafe[22](This Study)

**Physiochemical property analysis of FAME from vegetable seed oil**

The physiochemical properties ofFAME from *chrysophyllum albidum* seed oilor CAFAME in comparison with ASTM[19] are shown in Table 2. The colour of standard biodiesel was golden yellow while that of the CAFAME was light brown. The pH value of CAFAME was 6.12 which also compares favourably with that for standard FAME (~6.0)[17]. Likewise the density at 25 °C was 0.84 g/cm$^3$, which agrees with standard values and by correlations, the specific gravity at 15 °C and the °API gravity were 0.84 and 37.3 respectively. Both values fell within satisfactory levels of FAME standards.Other rheological and fuel properties such as oil viscosity, pour point and flash points determined were 59.6 mm$^2$/s, 5 °C and 82°C respectively. All three properties values adequately attained standard and with observations of Onuh *et al*[4]on FAME as against the raw vegetable oil.

**Table 3: Physicochemical properties of base component for drilling fluids formulation**

| Parameters | Petroleum Diesel Oil[22] | Bio-lubricants | | | | Motor lube (SAE40)[1] |
|---|---|---|---|---|---|---|
| | | *Chrysophyllum albidum* oil (CAOBL)[22] | Jatropha oil[1] | Palm oil[1,5] | Castor oil[18] | |
| Colour | Brown | Light brown | - | - | - | Light brown |
| Yield (%) | - | 24.5 | - | - | - | - |
| pH | 6.18 | 4.21 | - | - | - | - |
| Density g/cm$^3$@ 25 °C | 0.8538 | 0.9196 | 0.9044 | 0.919 | 0.8522 | 0.8755 |
| Specific gravity@ 15 °C | 0.8538 | 0.9196 | 0.9044 | 0.919 | 0.8522 | 0.8755 |
| °API gravity @ 15 °C | 34.23 | 22.371 | 24.957 | 22.47 | 34.541 | 30.122 |
| Viscosity, mm$^2$/s@ 40 °C | 50.25 | 52.64 | 51.89 | 38.25 - 50.33 | 256.17 | 130.22 |
| Viscosity, mm$^2$/s@ 100 °C | 4.1 | 8.25 | 8.53 | 7.58 - 10.87 | 22.78 | 14.42 |
| Pour point, °C | -12 | 4 | -3 | 5 | -12 | -12 |
| Flash point, °C | 87 | 84 | 296 | 240-253 | 296 | 260 |

Ebitisam *et al*[1]; Karmakar*et al*[5]; Dibal and Okonkwo[18]*;* Igbafe[22] (This Study)

**Physiochemical property analysis of base component for drilling fluids formulation**

The physiochemical properties of the two fluid types on comparative assessment as adequate most environmentally friendly suitable as base component fluids for formulation as drilling fluids are shown in Table 3. On colour, both fluid types were brownish, hence no significant discrepancy. In the case of pH, although both fluid were obviously acidic, the acidity was pronounced with *chrysophyllum albidum* oil derived polyol or bio-lubricant (CAOBL) as against petroleum diesel oil. This may be attributed to residual unreacted $H_2SO_4$ during esterification to reduce the %FFA prior to transesterification process[12,13]. In a similar fashion the densities of both fluids are less than that of water, however the CAOBL appears to be denser than petroleum diesel oil hence the CAOBL has more potentials to settling on water. Also similar characteristics can be deduced from the specific gravity at 15 °C and the °API gravity of 0.93 and 22.4.On the viscosity, polyols have kinematic viscosities between 40 – 250 cSt at 40 °C and 4 – 20 cSt at 100 °C. In this study, the CAOBL is more viscous than petroleum diesel oil with over 10 mm$^2$/s. The CAOBL viscosity though higher, would most certainly compensate and enhance the overall rheological property of the drilling fluid during formulation. The pour points and flash points of bio-lubricants and other lubricant types are within ASTM standards. However, bio-lubricants have higher pour point compared to non-biodegradable lubes such as petroleum diesel oil and automobile lube oils, hence any solidification or waxy precipitation would be rapid during transportation in pipelines, especially in cold weather[3,18]. However, CAOBL are renewable, genetically changeable nontoxic compared to petroleum diesel oil. In the context of contributions to environmental pollution and decreasing world petroleum reserves, vegetable oil-based lubricants are emerging as future green products because of their nontoxic and biodegradable nature[7].

## IV. Conclusion

Non-edible low-cost vegetable oils obtained from abundant waste *chrysophyllum albidum* seeds in Nigeria was exploited for the production of bio-lubricants. The oil was chemically extracted from seeds locally source in open markets, using n-hexane solvent in a Soxhlet extractor. The physicochemical properties of raw oils, FAME synthesised, polyol formulated and conventional fossil diesel, yielded pH of acidic an mixture on all four fluid types. Also densities and viscosities ranged between 0.83 and 0.93 g cm$^{-3}$ as well as 50.3 and 60.6 mm$^2$s$^{-1}$ respectively. Similarly pour points and flash points observed all compared adequately with ASTM (1995) standards. But for pour point property, the polyols (CAOBL) produced with the oils from *chrysophyllum albidum* seed showed considerably insignificant difference in characteristic properties to the conventional petroleum diesel oils. On the other hand for its biodegradability property compared to diesel oil, it is suitable and more appropriate as an environmentally friendly continuous phase component for the production of oil-base drilling fluid.

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