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**Original Research Article**

# Physicochemical Properties of Biodiesel from Congolese Non-Edible Oleaginous Plant *Allanblackia floribunda Oliv*

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## Abstract

Vegetable oil-based fuels are promising alternative fuels for diesel and light fuel engines because of their environmental and economic strategic advantages. In this study, we prepared the biodiesel from Congolese underutilized, nonedible Oleaginous Plant *A. floribunda Oliv*, and we determined its physicochemical properties according to the international standards for biodiesels and petro-diesels. *A. floribunda* oil was extracted and transesterified by alkali-catalyst using methanol with KOH as catalyst. The maximum yield of the obtained fatty acid methyl ester FAME (biodiesel) was 87.23%. The GC-MS Chromatography of the obtained fatty acid methyl ester FAME (biodiesel) showed the presence of Stearic acid, Oleic acid, Palmitic acid, Linoleic acid and Alpha-linolenic acid in the quantity of 61.68, 35.20, 1.15, 1.27 and 0.68%, respectively. Physicochemical properties (density, viscosity, flash, cloud and pour point; ash, water and sulfur contents; and corrosion on copper) values of Biodiesel (B100), and B20 (FAME blend in Gasoil), were within the range of values set by the international standards specifications of Petro-diesel and biodiesel (American Standard: ASTM D-6751 and European Standard- EN 14214). Thus, these two types of biodiesel (B100 and B20) could be used in a diesel engine in substitution of the Petro-diesel (Gasoil).

**Keywords:** *Allanblackia floribunda* oil; physicochemical parameters; Fatty Acid Methyl Ester (FAME); non-edible feedstock; transesterification; GC-MS.



## I. INTRODUCTION

Because of frequent crisis in petroleum supply, possibility of its scarcity, prices instability and negative effects to the environment, the interest in alternative energy sources has increased in the last years [1]. Global warming brought the discussion about corporations and countries social responsibility in adopting feedstocks which generate clean energy. The use of clean energies can be a solution for shortage in petroleum supply, since they are generated from natural cycles, do not cause alterations in the planet thermal balance, and are profitable, because they offer the companies the possibility of negotiating in the carbon credits market.

Vegetable oils are considered as a long-term promising source of renewable energy because of their potential to solve problems of environmental safety caused by constant dependence of the world on fossil fuels [2-7]. Their impact to environment is positive and they are biodegradable.

It was reported that, in diesel engines, vegetable oils can be used directly as a fuel or as a mixture with petrodiesel [8]. Nevertheless, their high viscosity and low fuel atomization in engines lead to an inaccurate fuel-air mixing and inefficient combustion [8, 9]. Problem occurs also during the coking in the injector, deposits in the engine, and thickening lubricants during prolonged operating of the engine [10, 11]. Hence, prior to their use as biodiesel, these vegetable oils have to be subjected to a pretreatment, such as transesterification with short alcohols [10, 11].

This transesterification reaction can be carried out in the presence of different types of catalysts (Homogeneous or heterogeneous). Furthermore, comparison of transesterification catalyst methods for the production of biodiesel from vegetable oils and fats was studied in order to reduce vegetable oils viscosity [11-13].

In order to avoid competition against food supply, the use of inedible oilseeds in the production of biodiesel is strongly recommended [11, 14]. Oilseed from *Allanblackia floribunda* is one of them.

*Allanblackia floribunda* which is the subject of the present work belongs to the family of the *Clusiaceae*. It is a species of the tropical Africa and its surface of distribution extends from Sierra Leone to Cabinda and to Uganda. *Allanblackia floribunda* is widely distributed in the pluvial forest zone from Nigeria to Centrafrique and to Democratic Republic of the Congo and towards the south and the north of Angola [15]. It is forests tree which can rich 30 m in height and 80 cm in diameter. The Fruits of *Allanblackia floribunda* are the berries in the shape of ellipsoid, capable to contain 40 to 100 seeds containing 60-65% oil [16].

The present work concerns the production of a biodiesel under the international standards of quality set by ASTM (American Society for Testing and Material) starting from the oil of *A. floribunda* followed by the comparison of the physicochemical properties between the produced biodiesel and the reference diesel.

## II. MATERIALS AND METHODS

### 2.1. Plant materials

Fruits of *A. floribunda* were collected surrounding areas located in Luki, Kongo-central Province, and Western Democratic Republic of Congo (DRC). The material was authenticated at the herbarium of INERA (Institut National de Recherches Agronomiques), Department of Biology, Faculty of Sciences, University of Kinshasa, DRC. The seeds drowned in the pulp are separated, dried in an oven at 106°C during 24 h and finely ground into powder.

### 2.2. Methods

#### 2.2.1. Extraction of Oil

The dried and crushed seeds were introduced into a Soxhlet extractor. After 5 h of extraction with cyclohexane as solvent, the extract was dried with sodium sulfate. The solvent was evaporated in a rotary vacuum evaporator and the solvent traces were eliminated by drying oil in an oven at 103°C for 6 h. The mass of the fat matter has been measured and the content in lipids calculated by the following formula:

$\%\ \text{lipids} = \frac{(M1 - M0)\times 100}{M}$ With, $M_1$: Mass of flask containing oil $M_0$: Mass of empty flask M: Mass of dried and crushed seeds used.

#### 2.2.2. Preparation of Methyl Esters from *A. floribunda* Oil

The transesterification is the most convenient method for the preparation of methyl esters (MTE) or ethyl esters (ETE). This process is chemically balanced and is done in three stages. The two first steps are slowly and the last one is very rapid [15-18].

Synthesis of methyl ester of *A. floribunda* oil was carried out by transesterification with potassium hydroxide as catalyst.

In a 1 L round-balloon flask (immersed in a temperature controlling silicon oil bath, equipped with a magnetic stirrer, a thermometer, and a condenser with a balloon filled of helium) containing a known amount of methanol (6 : 1 molar ratio to oil), *A. floribunda* oil and then potassium hydroxide (6 : 1 molar ratio to oil) were added. The reaction mixture was stirred vigorously and heated at 70◦ C for 2 h. Upon cooling, it separated into two layers. The ester mixture formed the upper layer and glycerin formed the lower layer. Excess methanol from upper layer was recovered, using a rotary evaporator. Ethyl acetate (30 mL) was then added to the residue, and the resulting solution was washed with water and neutralized with few drops of aqueous HCl 1 N followed by extraction with ethyl acetate (three times). The organic extracts were dried over anhydrous magnesium sulfate, and the solvent was removed under reduced pressure. Methyl ester yields and composition were determined by the GC and GC-MS respectively. For comparison purpose, a mixture of 90% of fossil fuel diesel and 10% of B100 was prepared; the latter was called B10.

#### 2.2.3. Analysis of Fatty Acid Methyl Ester (FAME, biodiesel) using Gas Chromatography method

The fatty acid methyl ester (FAME, biodiesel) content of the reaction mixture was determined by capillary gas chromatography (GC). The GC was a Varian CP-3800 gas chromatograph equipped with FID (flame ionization detector) and Varian 8400 auto-sampler. The column was a wall-coated open tubular (WCOT) fused silica capillary column of 100 m length with a 0.25 mm inside diameter (CP-Sil 88 for FAME; (Varian/Agilent Technologies, Santa Clara, CA, USA). The carrier gas was nitrogen and the split ratio was 10:1. The chromatographic separation conditions were: initial oven temperature, $140^o$ C; this was held for 6 min, then ramped at $4^o$ C/min to $225^o$ C and held for 15 min. Injector temperature was $270^o$ C and the detector temperature reached $300^o$ C. Fatty acid methyl ester (FAME's) identification was based on comparison of retention times of standard FAME (Supleco quantitative standard FAME 37).

### 2.2.4. Determination of the physicochemical properties of oil, B100, and B20.

The physicochemical properties of oil and the three types of biodiesel prepared starting from the oil of *A. floribunda*, were determined in the respect of the standard characterization of the ASTM (American Society for Testing and Material) [19]. The determined parameters were: the density at 20°C, the kinematic viscosity at 40°C, the flash point, the ash content, and the residual carbon, the corrosion on the blade of cooper, the flow point, the water content and the curves of distillation.

## III. RESULTS AND DISCUSSION

### 3.1. Extraction of Oil from *A. floribunda*

Table 1 gives the percentage T (%) of the oil extracted from the pulp of the fruit of *A. floribunda* using cyclohexane as solvent. The extraction yield found in this work is similar to that reported by Loumouamou and Mouni when using the Congo's and Cameroonian species, respectively [16, 20].

**Table-1: Extraction yield of *A. floribunda* oils.**

| Experience | Percentage T (%) |
|---|---|
| 1 | 63.66 |
| 2 | 63.18 |
| 3 | 62.83 |
| Average | 63.22±0.34 |

### 3.2. Biodiesel yield from *A. floribunda* oils after transesterification reaction.

The yield of biodiesel produced via basic homogeneous catalysis of *A. floribunda* oils is shown in Table 2. This yield is calculated using the following relation:

$$\% = \frac{Vi}{Vd} \times 100 \quad ,$$

Where, V$i$ represents the volume (mL) of the synthetized biodiesel (methylesters) and V$d$ the corresponding volume of *A. floribunda* oil used in the transesterification reaction.

The dependence of the biodiesel yield on the reaction time was investigated. The reaction time was varied in the range of 30 to 120 min. Fig.1 showed that at the beginning (30 min), the reaction was slow due to the mixing and dispersion of the methanol into the oil and the biodiesel yield increased fast in the reaction time range of 30 and 60 min. The miscibility plays a significant role because the starting materials (oil and methanol) are not sufficiently miscible [21].

The maximum yield of biodiesel (87.23%) was obtained after two hours of reaction. These results demonstrated the impact of time in the transesterification reaction.

**Table-2: Biodiesel yield from *A. floribunda* oil using a basic homogeneous catalyst**

| Volume of oil V*d* (mL) | Ratio | T(°C) | Time (min) | Volume of biodiesel V*i* (mL) | Yield (%) |
|---|---|---|---|---|---|
| 500 | 6:1 | 70 | 30 | 291.45 | 58.29 |
| 500 | 6:1 | 70 | 45 | 320.1 | 64.02 |
| 500 | 6:1 | 70 | 60 | 382.3 | 76.46 |
| 500 | 6:1 | 70 | 75 | 420.25 | 84.05 |
| 500 | 6:1 | 70 | 90 | 436.15 | 87.23 |

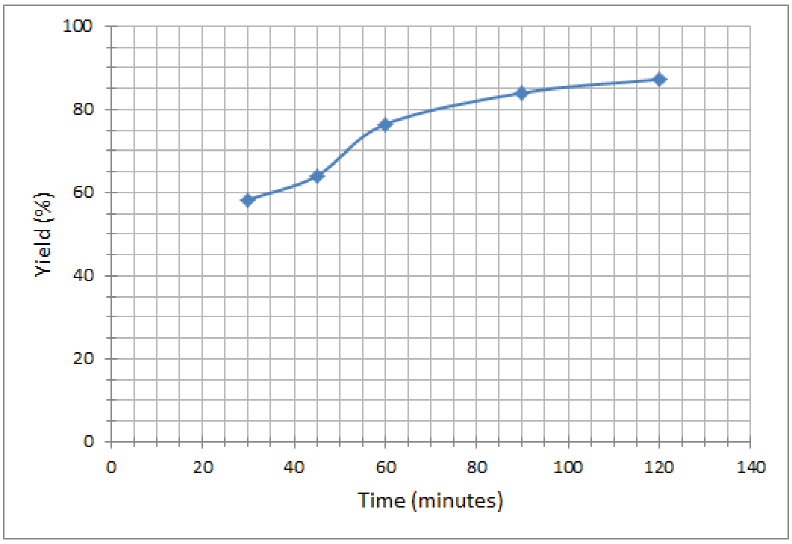


**Fig-1: Effect of reaction time on the biodiesel yield: methanol to oil molar ratio 6:1, reaction temperature 70°C.**

### 3.3. Analysis of Methyl ester (FAME, biodiesel) using Gas Chromatography method

The GC data (Table 3) and the chromatogram of the biodiesel (Figure 2) produced from *A. floribunda* oils show that the dominant fatty acids are stearic acid (C18:1; 61.68%), and oleic acid (C18:1; 35.20%). Other minor components present were linoleic acid (C18:2; 1.27%), palmitic acid (C16:0; 1.15%), and alpha-linolenic acid (C18:3; 0.68%). This agreed with results reported earlier by Loumouamou and Mouni when using the Congo's and Cameroonian species, respectively [16, 20].

The overall content of unsaturated and saturated fatty acids was 37.15% and 62.83% w/w, respectively.

**Table-3: Contents of Fatty acid of *Allanblackia floribunda* oil**

| Fatty acids | Brute formula | Amount (%) |
|---|---|---|
| Palmitic acid (C16:0) | $C_{16}H_{32}O_2$ | 1.15 |
| Stearic acid (C18:0) | $C_{16}H_{36}O_2$ | 61.68 |
| Oleic acid (C18:1) | $C_{18}H_{34}O_2$ | 35.20 |
| Linoleic acid (C18:2) | $C_{18}H_{32}O_2$ | 1.27 |
| Alpha-linolenic acid (C18:3) | $C_{16}H_{30}O_2$ | 0.68 |

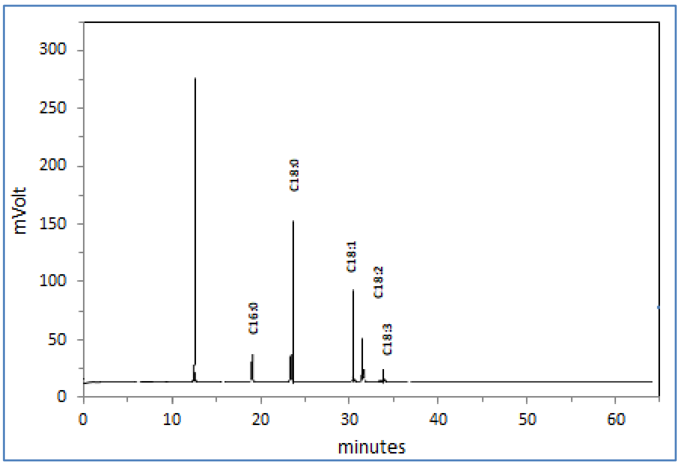


**Fig-2: GC chromatogram of biodiesel produced from *A. floribunda* oil.**

### 3.4. Determination of the physicochemical properties of oil, B100, and B20.

Since the biodiesel produced will be used in diesel engines, it is important that its quality be tested with standard methods (American Standard:-ASTM D 6751 and European Standard- EN 14214) [19, 22].

#### 3.4.1. Density

The physiochemical properties of biodiesel produced from *A. floribunda* oil (B100), B20 and Gasoil with international standards are listed in table 4.

Density is one of the principal fuel properties used to estimate the quantity of fuel injected by the injection systems to provide proper combustion. The density of the biodiesel fuels depends on several factors like feedstock used, method of biodiesel conversion and methyl ester profile [22]. A density in the range 810 < d < 890 $Kg/m^3$ ensures good running of engine [19, 23, 24].

The results of the study showed that the densities of 913, 870 and 868.8 $Kg/m^3$ for the *A. floribunda* oil, its biodiesel B100 and B20, respectively. The densities of B100 and B20 are less than 890 $Kg/m^3$ (the maximum required by the standard).

#### 3.4.2. Kinematic viscosity

Viscosity is one of the vital characteristics of a fuel which signifies the ability of fuel to flow. Being resistance to the flow, viscosity plays a major role in spray atomization and spray penetration. The kinematic viscosity of biodiesel is measured using ASTM D-445 (2.0–6.0 $mm^2/s$) and EN ISO 3104 (3.5–5 $mm^2/s$) [19, 22].

In this study, by comparing the results presented in Table 4; *A. floribunda* oil has a viscosity value of 6.73 which is higher than the limit fixed by the standard ASTM D445. However, those of biodiesels (B100: 4.31 $mm^2/s$ and B20 4.21 $mm^2/s$) are within the range of values set by the specifications.

#### 3.4.3. Flash Point

Flash point (FP) is the lowest temperature at which the vapors of the volatile fuel starts ignite when exposed to an ignition source. The conventional petro-diesel has a flash point of about 50–65 °C, whereas biodiesel possesses flash point of more than 150 °C. This signifies that biodiesel has better safety aspects in storage and in transit when compared to petro diesel. Generally, straight vegetable oil has a higher flash point than its methyl esters due to its poor volatility [19].

Flash Point is measured by the procedure prescribed in ASTM D-93 (60°C minimum) and EN ISO-3679(120°C minimum).

From table 4, it found that *A. floribunda* oil shows uppermost flash point value of 265°C, whereas its methyl esters (Biodiesel: B100 and B20) show the flash point value of 152 and 78°C, respectively. Thus, by comparing all flash point values (biodiesel B100, B20 and Gasoil: GO), we find that biodiesel B100 is less dangerous than B20 and Gasoil (GO).

### 3.4.4. Cloud Point

Cloud point is the lowest possible temperature at which the wax in the fuel is first seen to crystallize and form a cloudy appearance. Cloud point is the most common criterion used to set the low-temperature fuel controls [25]. From table 4, it found that *A. floribunda* oil cloud point value of +5°C, whereas its methyl esters (Biodiesel: B100 and B20) show the cloud point value of +2 and -10°C, respectively. These values of cloud point are within the range of values set by the specifications (+6°C maximum).

### 3.4.5. Pour point

Pour point of a liquid fuel is the minimum temperature at which the fuel loses its flow characteristics. Pour Point is also a crucial parameter in cold flow operation since the fuel is suitable for operation only above the pour point value. By comparing the results as presented in Table 4 with the standards of ASTM D97, the two biodiesels (B100 and B20) revealed pour point values in conformity to the ASTM specification (+5°C maximum).

### 3.4.6. Total sulfur (% weight).

Nowadays emission regulations strictly restrict the amount of sulfur in the fuel due to the threat of acid rain caused by the sulfur oxide emission. Sulfur content in a fuel directly influences the magnitude of sulfur oxides emissions during the combustion of fuel. It has been observed that the biodiesel synthesized from vegetable oils have very low levels of sulfur content [26]. From table 4, it found that gasoil content 0.043 % of sulfur, whereas *A. floribunda* oil and its methyl esters (Biodiesel: B100 and B20) content 0.021, 0.002 and 0.018 % of sulfur, respectively.

### 3.4.7. Copper strip corrosion (3 hours at 50°C), Water and ash content (%).

Water and ash contents present in the biodiesel portrays the cleanliness of biodiesel fuel. The presence of water content in biodiesel reduces the calorific value of fuel and corrodes components of engine fuel system. In addition, high water content induces hydrolysis reaction which converts biodiesel to free fatty acids [19, 26]. From table 4, it found that gasoil content 0% of water, whereas *A. floribunda* oil and its methyl esters (Biodiesel: B100 and B20) content 0.04, 0.04 and 0.01 % of water, respectively. These values are within the range of values set by the specifications (0.05% maximum). The test of corrosion on the blade copper is used to control the corrosiveness of fuel toward the copper metal, which is used in diesel engines. According to the standard method, corrosion should not exceed level 1 (light yellow) in a reference scale. In this study, all biodiesels and crude oil have the maximum recommended value of 1. They do not corrode the engine neither during operation during storage.

**Table-4: Physicochemical properties of *A. floribunda* oil, B100, B20 and GO**

| Properties (units) | ASTM Limits | EN 14214 Limits | Methods | *A. floribunda* oil | B100 | B20 | Gasoil(GO) |
|---|---|---|---|---|---|---|---|
| Density at 15°C (Kg/m3 ) | 810–890 | 860-900 | ASTM D-4052 | 913 | 870 | 868.2 | 867,0 |
| Viscosity at 40°C (cSt or $mm^2/s$) | 2.0 à 6.0 | 3.5 à 5.0 | ASTM D-445 | 6.73 | 4.31 | 4.21 | 4,09 |
| Flash Point (°C) | 60 min | 120 min | ASTM D-93 | 265 | 152 | 78 | 73 |
| Cloud Point (°C) | +6 max | - | ASTM D97 | +5 | +2 | -10 | -15 |
| Pour point (°C) | +5 max | - | ASTM D-97 | +14 | +4 | -8 | −10 |
| Total sulfur (% weight) | 0.05 max | 0.02 max | ASTM D4294 | 0.021 | 0.002 | 0.018 | 0.043 |
| Copper strip corrosion (3 hours at 50°C) | 1 max | 1a max | ASTM D130 | 1b | 1a | 1a | 1a |
| Ashes (%) | 0,01 max | 0.02 max | ASTM D-482 | 0.003 | 0.018 | 0.014 | 0.01 |
| Water content (%) | 0.05 max | 0.05 max | ASTM D-95 | 0.04 | 0.04 | 0.01 | 0 |

## IV. CONCLUSION

The increasing demand for energy and the limitation of the oil resources lead to the development of renewable energies including fat content (oils vegetable and animal) or their derivatives. This utilization of raw materials can contribute to a diversification of the energy resources. The biodiesel is an environmental friendly biodegradable alternative fuel that can be used directly in the engine without any major engine modification.

This study revealed that high quality light fuel oil (biodiesel) can be produced successfully from underutilized, nonedible *A. floribunda* oil as a starting biomaterial through alkali-catalyst transesterification using methanol with KOH as catalyst.

The physicochemical properties of two obtained biodiesels (B100 and B20) were within the international standards (American Standard:-ASTM D-

6751 and European Standard- EN 14214). Thus, fatty acid methyl esters (FAME) of *A. floribunda* oil appears to be promising alternative fuel for conventional light biofuel engines.

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