

Original Research Article

Fourier Transform Infrared (FTIR) Analysis of Fatty Acid Methyl Ester from Congolese Non-Edible *Azelia bella* seeds oil.

ABSTRACT

The increasing demand for energy and the limitation of the oil resources as raw materials for the production of biodiesel lead to identify and valorize unconventional vegetable oils, which are not edibles. This study consisted in revalorizing of Congolese nonedible *Azelia bella* seeds oil as raw materials for the biodiesel production. *Azelia bella* seeds oil was extracted with oil yield of $26.38 \pm 0.22\%$ and subsequently transesterified by alkali-catalyst using methanol with KOH as catalyst. The maximum yield of the obtained fatty acid methyl ester FAME (biodiesel) was 96.53% and FTIR spectroscopy showed the presence of fatty acid methyl esters in the produced biodiesel. The FTIR spectrum for the biodiesel revealed the functional groups with characteristics bands C=O stretching, CH₃ asymmetric bending, and O-CH₃ stretching in the spectrum with the peaks at 1743.94, 1436.11 and 1197.38 cm⁻¹, respectively. Physicochemical properties (density, viscosity, flash, cloud and pour point; Cetane number, ash, water and sulfur contents; and corrosion on copper) values of Biodiesel (B100), and biodiesel blend in Gasoil (B50 and B20), were within the range of values set by the international standards specifications of Petrodiesel and biodiesel (American Standard: ASTM D-6751 and European Standard- EN 14214). Thus, these three produced biodiesels (B100, B50 and B20) from the Congolese *Azelia bella* seeds oil could be used in a diesel engine as a substitute for petro-diesel.

Keywords: *Azelia bella* seeds oil; transesterification; biodiesel; fatty acid methyl esters (FAME); FTIR spectroscopy.

1. INTRODUCTION

For some time, the production of biodiesel, as a substitute for petro-diesel, has faced an obstacle related to the raw material or biomass to be used because most of the vegetable oils used compete with local food consumption [1]. Thus, it is very important to identify and use unconventional vegetable oils, which are not edible. 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 [2–6]. Their impact to environment is positive and they are biodegradable. Before their use as biodiesel, these vegetable oils have to be subjected to a pretreatment, such as transesterification with short alcohols [1].

Unconventional oilseeds are a very important part of the Congo Basin Forest Non-timber Forest Products (NTFPs) [7–10]. One of these unconventional Congo (DRC) plants is *Azelia bella*.

Azelia bella belongs to the family Caesalpiniaceae. It is known as “Bolengu” in local Kongo central in Democratic Republic of the Congo (DRC). It is widespread occurring from eastern Guinea and Liberia east to the Central African Republic, and south to DR Congo and Cabinda (Angola) [11, 12]. *Azelia bella* is a non-edible, non-toxic and biodegradable substance. To our knowledge, there are no studies yet relating to the production of biodiesel from this Congolese plant. The purpose of this study is to contribute to the revalorization of *Azelia bella* seeds oil in the production of biodiesel under the international standards of quality set by ASTM (American Society for Testing and Material).

Comment [WP1]: Very less introduction, did not reflect the authors interest of research.

2. MATERIAL AND METHODS

2.1 Plant materials

Fruits of *Afzelia bella* were collected surrounding areas located in Mayombe, Kongo-central Province, 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 Extraction of Oil and Physicochemical Properties

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:

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

Physicochemical chemical properties of *Afzelia bella* were done according to [13]. The determined parameters were color, density at 28°C, Viscosity at 40°C, saponification value, iodine value, acid value, ash content, and moisture content.

2.3 Preparation of Methyl Esters from *Afzelia bella* seeds 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 [14-15].

Synthesis of methyl ester of *Afzelia bella* oil was carried out by transesterification with potassium hydroxide as catalyst according to the method described by Mulula and Al [1].

For comparison purpose, a mixture of 80% of fossil fuel diesel and 20% of B100 was prepared; the latter was called B20. Another mixture of 50% of fossil fuel diesel and 50% of B100 was prepared and called B50.

2.4 Analysis of Fatty Acid Methyl Ester (FAME, biodiesel) using FTIR Spectroscopy.

The conversion of *Afzelia bella* oil to the biodiesel (*Afzelia bella* methyl ester) was defined by the shape of the peaks in the carbonyl region of the spectrum using a PerkinElmer Spectrum 100 FTIR spectrometer fitted with a Universal Sampling Accessory. Measurement was recorded in Transmittance mode and the range was between 4000 cm^{-1} and 600 cm^{-1} . Biodiesel sample (about 0.1mL) was dropped on the diamond ATR probe aperture, and spectra were recorded at a resolution of 8 cm^{-1} using 4 scans under 1 minute against air as the background.

2.5 Determination of the physicochemical properties of B100, B50 and B20.

The physicochemical properties of oil and the three types of biodiesel prepared starting from the oil of *Afzelia bella*, were determined in the respect of the standard characterization of the ASTM (American Society for Testing and Material) [16]. The determined parameters were: the density at 28°C, the kinematic viscosity at 40°C, the flash point, pourpoint, Cetane number, the ash content, and the water content.

3. RESULTS AND DISCUSSION

3.1 Extraction and Physicochemical properties of *Afzelia bella* seeds oil.

The results of physicochemical properties of the extracted seed oil samples are shown in Table 1. The percentage of oil extracted was 26.38±0.22%. This value is higher than that found by Kabele in 1979 [10].

The acid value is a measure of total acidity of system, which may involve contributions from all the constituent fatty acids that make up the glyceride molecule. It correlates to the fuel's long-term stability and corrosiveness, the smaller the acid value, the higher the quality of the fuel [16]. The acid value of the *Afzelia bella* seeds oil was 2.96. This value is higher than the oil (2.174 – 2.610) and biodiesel standards (0.8 max), respectively [16, 17].

The same applies to the density of this oil, which is 911.3 (Kg/m³) whereas the biodiesel standards require a value of 810–890 (Kg/m³) [16]. Thus, this oil must undergo a transesterification reaction in order to improve these parameters.

Comment [WP2]: More discussion is required to compare with the results.

However the Saponification and iodine values were 124.16 and 120.86, respectively. These results are almost similar to those reported by Ogbu and Otori when using another *Afzelia* species (*Afzelia africana*) in Nigeria [18, 19].

Table 1. Extraction yield of *Afzelia bella* seeds oils.

Parameters	Values
Yield (%)	26.38±0.22
Color	Yellow
Density at 28°C	911,3
Viscosity at 40°C (cSt or mm ² /s)	6.23
Saponification value (mg KOH/g)	124.16
Iodine Value (g/100 g)	120.86
Acid value (mgKOH/g)	2.96
Ash content (%)	0.04
Moisture content (%)	1.69

3.2 Biodiesel Yield and analysis of Fatty Acid Methyl Ester (FAME, biodiesel) using FTIR Spectroscopy.

The yield of biodiesel produced via basic homogeneous catalysis of *Afzelia bella* oils is calculated using the following relation:

$\% = \frac{V_i}{V_d} \times 100$, Where, V_i represents the volume (mL) of the synthesized biodiesel (methyl esters) and V_d the corresponding volume of *Afzelia bella* oil used in the transesterification reaction. The obtained yield of biodiesel was 96.53% after two hours of reaction.

The Fatty Acid Methyl Ester (FAME, biodiesel) spectrum is shown in Figure 1 and the lists of functional groups identified were shown in Table 2.

The presence of infrared bands in the region 1425–1447 cm⁻¹ (for CH₃ asymmetric bending) and 1188–1200 (for O-CH₃ stretching), in the spectra, clearly demonstrated the transformation of vegetable oils into biodiesel according to the literature [20,21].

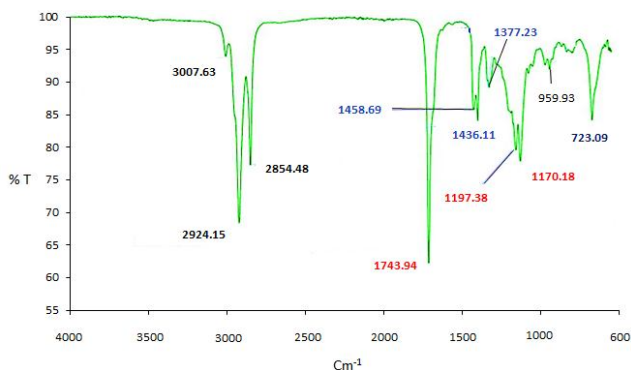


Fig. 1. FTIR spectra of *Afzelia bella* Fatty Acid Methyl Ester (FAME, biodiesel).

According to the literature, the region 678.55 cm⁻¹ – 960 cm⁻¹ indicates the presence of =C-H functional groups which are attributed to olefinic (alkenes) functional groups in the biodiesel and they are unsaturated [21, 22, 23]. Thus, the specific peak at 723.09 cm⁻¹ could be attributed to =C-H (cis) groups, whereas the peak at 959.93 cm⁻¹ could be attributed to =C-H (trans) groups.

According to the literature, the peaks at 1170.18 and 1197.38 cm⁻¹ could be allocated to C-O-C and O-CH₃ stretching vibration of a terminal methoxy group of the ester (biodiesel) respectively [23, 24].

The band region of 1377.23 – 1465.03 cm⁻¹ can be ascribed to the bending vibration of C-H methyl groups in the fuel [23,24]. Thus, absorption peaks at 1436.11 and 1458.69 cm⁻¹ could be attributed to asymmetric CH₃ and CH₂ bending vibrations of the ester, respectively. The same goes for the peak that absorbs at 1377.23 cm⁻¹.

The characteristic peak at wavenumber 1743.94 cm^{-1} which is strongest in the spectrum is attributed to C=O groups with the stretching mode of vibration. These groups indicate the presence of carbonyl functional groups in the biodiesel and the conversion of triglycerides in the oil to methyl esters [21-24].

Intense sharp peaks at 2854.48 cm^{-1} and 2924.15 cm^{-1} could be attributed to aliphatic symmetric CH_2 stretching and aliphatic asymmetric CH_2 stretching vibrations, respectively [21-24]. However, the small peak at the shoulder of the intense asymmetrical CH_2 peak at 3007.63 cm^{-1} could be represented unsaturation due to olefinic double bonds [21-24].

Table 2. FTIR peaks of *Afzelia bella* Fatty Acid Methyl Ester (FAME, biodiesel).

Wavenumber (cm^{-1})	Types of vibration	Functional groups
3007.63	Asymmetrical stretching	=C-H (alkenes)
2924.15	Asymmetrical stretching	C-H (alkanes)
2854.48	Symmetrical stretching	C-H (methylene)
1743.94	Stretching	C=O (ester carbonyl functional group in biodiesel)
1458.69	Asymmetric Bending	CH_2
1436.11	Asymmetric Bending	CH_3
1377.23	Bending	CH_3 (C-H)
1197.38	Stretching	O- CH_3 terminal methoxy group of the ester (biodiesel)
1170.18	Stretching	C-O-C
959.93	Bending of alkenes	=C-H (trans) groups.
723.09	Bending of alkenes and overlapping of rocking	=C-H and $-(\text{CH}_2)_n$ methylene groups (cis disubstituted alkenes and aromatic)

3.3. Determination of the physicochemical properties of B100, B50 and B20.

The physicochemical properties of biodiesel synthesized from *Afzelia bella* oil (B100), B50, B20 and Gasoil according to international standards (American Standard:-ASTM D 6751 and European Standard- EN 14214) are listed in table 3.

Engine efficiency is significantly linked with the fuel viscosity (one of the vital characteristics of a fuel) which plays a major role in spray atomization. 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) [1, 16, 25].

The kinematic viscosity values at 40°C (mm^2/s) of *Afzelia bella* Fatty Acid Methyl Ester were found to be 4.58, 4.41, and 4.28 mm^2/s for Biodiesel 100 (B100), B50 and B20, respectively. These results indicate that the kinematic viscosity values of *Afzelia bella* biodiesels are within the range of values set by the specifications.

The flash point is an essential fuel property which expresses the fuel risk flammability because of the presence of extremely flammable and volatile constituents. Higher flash point usually eliminates the risk of fire. The flash point (°C) values of *Afzelia bella* biodiesels were 118, 83, and 78 °C for Biodiesel 100 (B100), B50 and B20, respectively. Thus, by comparing all flash point values (biodiesel B100, B50 and B20), we find that biodiesel B100 is less dangerous than B50 and B20.

These values are within the range of values set by the specifications (60°C minimum for ASTM D-93 and 120°C minimum for EN ISO-3679).

Cloud point is the most common criterion used to set the low-temperature fuel controls. High cloud point usually results in fuel line clogging [1,2]. However, the pour point of a liquid fuel is the minimum temperature at which the fuel loses its flow characteristics [25].

By comparing the results as presented in Table 3 with the standards, the three *Afzelia bella* biodiesels (B100, B50 and B20) revealed cloud point and pour point values in conformity to the ASTM specifications.

Cetane number is a measure of ignition quality of diesel fuel. The higher the cetane number, the easier the fuel will ignite when it is injected into the engine the better the fuel [19]. The Cetane number values of *Afzelia bella* biodiesels were 51.2, 50.7 and 49.5 for Biodiesel 100 (B100), B50 and B20, respectively.

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 [1,2]. From table 3, it found that gasoil contents 0.046 % of sulfur, whereas *Afzelia bella* biodiesels (B100, B50 and B20) content only 0.023, 0.032 and 0.039% of sulfur, respectively.

Table 3. Physicochemical properties of Biodiesel 100, B50, B20 and GO.

Properties (units)	ASTM Limits	EN 14214 Limits	Methods	B100	B50	B20	Gasoil (GO)
Density (Kg/m ³)	810–890	860-900	ASTM D-4052	877.2	872.8	869.4	867,0
Viscosity at 40°C (cSt or mm ² /s)	2.0 à 6.0	3.5 à 5.0	ASTM D-445	4.58	4.41	4.28	4,09
Flash Point (°C)	60 min	120 min	ASTM D-93	118	83	78	73
Cloud Point (°C)	+6 max	-	ASTM D97	+2	-7	-11	-15
Pour point (°C)	+5 max	-	ASTM D-97	+4	-3	-7	-10
Cetane number	45 Min	-	ASTM D976	51.2	50.7	49.5	48.6
Total sulfur (% weight)	0.05 max	0.02 max	ASTM D4294	0.023	0.032	0.039	0.046
Copper strip corrosion (3 hours at 50°C)	1 max	1a max	ASTM D130	1a	1a	1a	1a
Ashes (%)	0,01 max	0.02 max	ASTM D-482	0.007	0.005	0.003	0.002
Water content (%)	0.05 max	0.05 max	ASTM D-95	0.03	0.02	0.01	0

4. CONCLUSION

The increasing demand for energy and the limitation of the oil resources as raw materials for the production of biodiesel lead to identify and valorize unconventional vegetable oils, which are not edibles.

This study revealed that high quality light fuel oil (biodiesel) can be produced successfully from nonedible *Afzelia bella* oil as a starting biomaterial through alkali-catalyst transesterification using methanol with KOH as catalyst. The FTIR spectrometry showed the presence of fatty acid methyl esters in the produced biodiesel.

The physicochemical properties of three obtained biodiesels (B100, B50 and B20) were within the international standards (American Standard:-ASTM D-6751 and European Standard- EN 14214). Thus, with an oil yield of 26.38±0.22%, the Congolese *Afzelia bella* seeds oil is promising to contribute to the production of biodiesel as a substitute for petro-diesel.

COMPETING INTERESTS DISCLAIMER:

Authors have declared that no competing interests exist. The products used for this research are commonly and predominantly use products in our area of research and country. There is absolutely no conflict of interest between the authors and producers of the products because we do not intend to use these products as an avenue for any litigation but for the advancement of knowledge. Also, the research was not funded by the producing company rather it was funded by personal efforts of the authors.

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Comment [WP3]: Some references are not as per the journal.

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