Original Research Article

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

ABSTRACT

As the oils used for biodiesel production face food competition, many scientific research is now focusing on the upgrading of non-conventional oils, which are not edibles.
This study consisted in revalorizing of Congolese nonedible *Afzelia bella* seeds oil as raw materials for the biodiesel production. *Afzelia bella* seeds oil was extracted with oil yield of 26.38±0.22% and subsequently transesterified by alkali-catalyst using methanol with KOH as catalyst. The maximum yield of the obtained Diacetylenic Light Fuel Oil was 96.53% and FTIR spectroscopy showed the presence of fatty acid methyl esters in the produced biodiesel. The FTIR spectrum of Diacetylenic Light Fuel Oil revealed the following characteristics functional groups: Carbonyl group stretching (C=O), Methyl asymmetric bending (CH_3) and Methoxy group stretching (O-CH_3) with the peaks at 1743.94, 1436.11 and 1197.38 cm^{-1}, respectively. Physicochemical properties (Kinematic viscosity, density, flash point, Cetane number, cloud point, pour point, water contents, ash and sulfur contents) 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 diesel and biodiesel (American Society of Testing Materials and European Standard). Thus, these three produced biodiesels (B100, B50 and B20) from the Congolese *Afzelia bella* seeds oil could be used as a substitute for diesel without damaging the engine.

Keywords: Afzelia bella seeds oil; Diacetylenic Light Fuel Oil; transesterification; FTIR spectroscopy, biodiesel; fatty acid methyl esters.

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 edibles. Being biodegradable and clean to the environment, vegetable oils have always been considered as good alternatives that can solve problems related to environmental pollution [2–6]. However, since their densities and viscosities are high, they cannot be used directly in diesel engines and must be pre-treated through the transesterification reaction, in which a short chain alcohol reacts with the oil in order to produce the alkyl esters of fatty acids called "Biodiesel" [1].
Several studies have already evaluated biodiesel production from different oilseeds such as palm oil, soybean oil, sunflower oil, rapeseed oil, etc. in the presence of different catalysts (acids, bases, etc.) as well as their physicochemical properties almost similar to those of fossil diesel [7,8,9].
Thus, there was competition between the use of these oleaginous seeds in biodiesel production and food demand.
Hence, the need to use non-conventional oilseeds.
Unconventional oilseeds are a very important part of the Congo Basin Forest Non-timber Forest Products (NTFPs) [10–13]. One of these unconventional Congo (DRC) plants is *Afzelia bella*.
Afzelia 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 Liberia east, eastern Guinea to the Central African

Republic, to Democratic Republic of the Congo and Angola (Cabinda) [14, 15]. *Afzelia 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 *Afzelia bella* seeds oil in the production of biodiesel according to American Society of Testing Materials (ASTM) and European Standards.

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.

Fig. 1. *Afzelia bella* seeds

2.2 Extraction of Oil and Physicochemical Properties

Afzelia bella seeds oil was extracted by the Soxlet extraction using Cyclohexane as solvent according to method describe by mulula and al[1]. Cyclohexane was selected as an extraction solvent because it is apolar and especially less toxic compared to n-hexane [6].
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.
Physicochemical chemical properties of *Afzelia bella* were done according to [16]. Here are some physicochemical parameters determined: Kinematic viscosity, color, density at 28°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 [17-18].

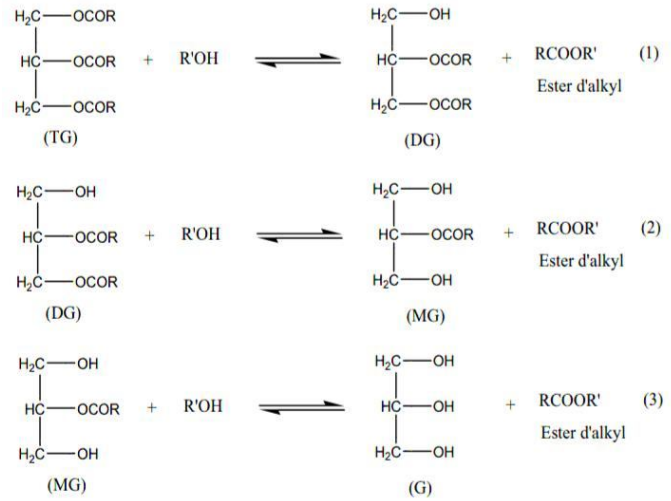

Fig. 2. Transesterification reaction.

The biodiesel form *Afzelia bella* oil was prepared by by transesterification reaction with potassium hydroxide as catalyst according to the method described by Mulula and Al [1].
A mixture of 80% of fossil diesel with 20% of *Afzelia bella* oil biodiesel called "B20" as well as another mixture consisting of 50% of fossil diesel with 50% of *Afzelia bella* oil biodiesel (called "B50") were prepared to compare their physicochemical properties.

Fig. 3. *Afzelia bella* oil Biodiesel separation.

2.4 Analysis of *Afzelia bella* oil Biodiesel using FTIR Spectroscopy.

The characteristic functional groups of *Afzelia bella* oil biodiesel were determined by 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 8cm^{-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 *Afzelia bella* oil biodiesel and their mixtures were determined according to American Society of Testing Materials (ASTM) and European Standards [19].
Here are some physicochemical parameters determined: Kinematic viscosity at 40°C, the density at 28°C, Cetane number, the flash point, Pourpoint, 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 *Afzelia bella* seeds oil are shown in Table 1.
The percentage of oil extracted was 26.38±0.22%. This value is higher than that found by Kabele and al. during their study on non-conventional oleaginous plants of Zaire in 1979 [13].
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 [19]. 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 [19, 20].
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) [19]. Thus, this oil must undergo a transesterification reaction in order to improve these parameters.
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 [21, 22].

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 *Afzelia bella* seeds oil biodiesel using FTIR Spectroscopy.

The yield of *Afzelia bella* seeds oil biodiesel is calculated using the following relation:

$\% = \frac{Vi}{Vd} \times 100$, Where, Vi represents the volume (mL) of the synthetized *Afzelia bella* seeds oil biodiesel and Vd the corresponding volume of *Afzelia bella* seeds oil used in the transesterification reaction. The obtained yield of biodiesel was 96.53% after two hours of reaction.
The *Afzelia bella* seeds oil biodiesel spectrum is shown in Figure 4 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 Methyl asymmetric bending CH_3) and 1188–1200 (for Methoxy group stretching O-CH_3), in the spectra, clearly demonstrated the transformation of vegetable oils into biodiesel according to the literature [23,24].

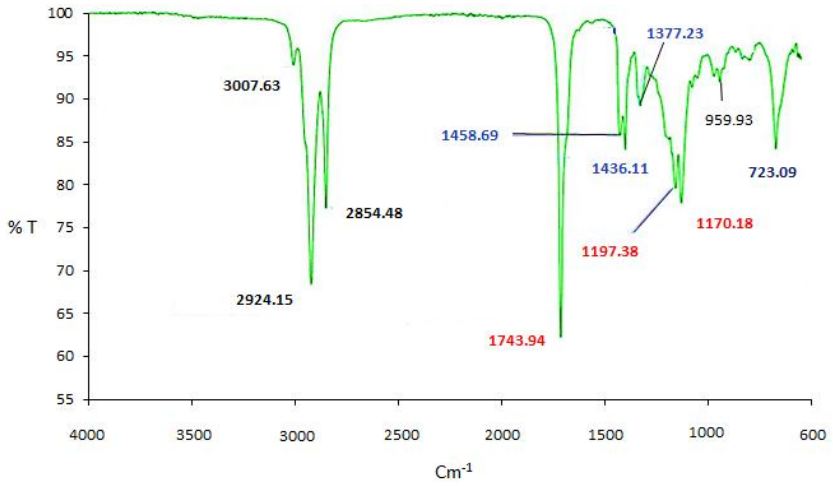

Fig. 4. FTIR spectra of *Afzelia bella* Fatty Acid Methyl Ester (FAME, biodiesel).

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 [24, 25, 26]. 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-CH_3 stretching vibration of a terminal methoxy group of the ester (biodiesel) respectively [26, 27].

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 CH_3 and CH_2 bending vibrations of the ester, respectively. The same goes for the peak that absorbs at 1377.23 cm^{-1}.

The characteristics 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 [24-27].

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 [24-27]. 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 olefenic double bonds [24-27].

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 –(CH_2)n methylene groups (cis disubstituted alkenes and aromatic)

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

The physiochemical 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, 19, 28].

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 [28].
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 [22]. 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/m3)	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

As the oils used for biodiesel production face food competition, many scientific research is now focusing on the upgrading of non-conventional oils, which are not edibles.
The purpose of this study was to contribute to the revalorization of *Afzelia bella* seeds oil in the production of biodiesel according to American Society of Testing Materials (ASTM) and European Standards.
Biodiesel from *Afzelia bella* seeds oil (B100) and their mixture with diesel fossil (B20 and B50) were prepared and analyzed according to American Society of Testing Materials (ASTM) and European Standards.
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.

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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