

Physicochemical Characterization of a Biodiesel Produced from Oil Extract from the Pulp of *Raffia Sese de Wild* Collected in Democratic Republic of Congo

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Abstract

Biodiesel are produced and characterized from the oil extract from the pulp of *Raphia sese de Wild* collected in Democratic republic of Congo. The transesterification reaction was used in homogeneous phase with acid and base catalysts. The reaction was carried out in volume ratio 6:1 of ethanol to oil using 1% in volume of the concentrated sulfuric acid (H₂SO₄) or sodium hydroxide (NaOH). The yield of the reaction was 73% in transesterified oil for the acid catalyzed reaction runned during 3 hours at 60°C, and 99.2% for the base catalyzed reaction runned during 2 hours, at the same temperature. The physicochemical properties were determined for B100 (pure biodiesel), B10 and B5 (blended biodiesel with the fossil gazole). The results show that these three types of biodiesel can be used in a diesel engine in replacement of the traditional gazole.

Keywords: *Raffia sese of Wild* oil; Transesterification; acid catalyst; base catalyst; physicochemical parameters.

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1. Introduction

It is demonstrated that the use of fossil energy pollutes the environment. However, on the other hand, the fossil energy crisis will slow down the development process in the world with multiple consequences. To dam up these environment and development consequences, it is necessary to find out alternative solutions that can target both problems. The sustainable development demands more guaranteed than the solutions, regarding to each local problem which affects the implementation of local resources.

There is no development without more accessibility to energy resources. In this world in full transfer, contradictorily upset by the rapid demographic evolution of the populations and the rarefaction of the traditional fossil energy reserves (petroleum, charcoal, naturel gas, etc.), the increasing consumption of energies coupled with the phenomena of the climatic change, constitutes the challenges of energy which are numerous and whose solutions are complex [1-6].

Vegetable oils can be considered as one of the promising sources of renewable and sustainable energies. According to its availability, vegetable oils can be used to solve the problems of environmental impact and security caused by the constant dependence of the man with respect to fossils fuels.

These last decades, several researches have been undergone on various vegetable oils which can produce biofuels presenting good physicochemical proprieties compared to diesel fuel. Regarding to these proprieties, biofuels are promoted to replace, partially or totally, the fossil fuel. Several oils extracted from the seeds of vegetable plants such as sunflower, cartage, soya, colza, crowd, etc. have been tested with success in diesel engines [6-14]. To ameliorate their physicochemical proprieties, vegetable oils have been transesterified using homogenous or heterogeneous catalysis [6-10]. It has been noted that biodiesel, as an alternative fuel, is made from renewable materials such as vegetable oils and animal fats. Their impact to environment is positive because they are biodegradable and non-toxic [6]. Moreover, different ways to produce biofuels from different kind of raw materials (refine, crude or frying oils) in the presence of different types of catalysts (basic, acid, ion exchange resins, lipases and supercritical fluids) have been studied [7]. From these studies, the ratio of 6/1 between the volume of alcohol and the oil, respectively, gave an important transesterification conversion using 0.5 to 1% w/w of the base catalyst. The other important parameter determined by the above studies was the reaction temperature that is fixed at 60°C [7]. For the acid catalysis, to find a very high yield of the oil conversion into biodiesel, since the reaction velocity is very low, a molar ratio of 30/1 between the alcohol and the oil was used in 50 hours reaction to get a conversion of 99% [7]. Furthermore, comparison of transesterification methods for the production of biodiesel from vegetable oils and fats was studied in order to reduce vegetable oils viscosity [8]. This parameter is the most important in the choice of a fuel depending on the type of engines. This study revealed that sodium hydroxide is very adapted to be used due to its low cost and high yield [8]. In another study using the methanolic sulfuric acid for the acid catalysis, a very rapid transesterification conversion was obtained with 10% of sulfuric acid mixed to methanol. Here, the reflux temperature is first reached before adding the oil [8]. In view to not cause stress to food commodities, it was proposed the utilization of the second generation raw materials in the production of biofuels than the first generation biofuels. The second generation raw materials are not competitive to the food supply [9].

This situation explains the increasing use of wasted edible material as raw materials. The less or no exploited oil seeds is very promoted because of their availability and sustainability [15]. The transesterification reaction combines oil, alcohol and catalyst. Several families of vegetable plants are known in the Democratic Republic of Congo (DRC). Many of them are used in food supply such as palm oil which is the most important with a significant contribution. However, other species exist in DRC and need to be promoted such as *Raffia sese de Wild* (R.S.W) that belongs to the family of palmaceae.

It was reported that in the diesel engine, vegetable oils can be used directly and/or blended to the petrodiesel (16). Nevertheless, because of the high viscosity of oils, the atomization of carburized in the engines is weak. That causes an inaccurate fuel-air mixture with an ineffective combustion like primary consequence (16). The problem also appears during the coking of injectors that contribute to the apparition of the deposits and the thickening of the lubricants during the engine operation. Due to that, a preliminary process needs to be done on vegetable oils before their utilization as fuels in replacement of diesel fuel, partially or totally (17).

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 *Raffia sese de Wild* followed by the comparison of the physicochemical properties between the produced biodiesel and the reference diesel. In this aim, different parameters of crude oil of *Raffia sese de Wild*, its transesterified oil blended or not to diesel (B100, B10 and B5) and the reference diesel were compared. In this study, the homogeneous catalyst (acid and base) is used to produce biofuels by reacting ethanol with *Raffia sese de Wild* 's oil in the ratio of 6/1 during 3 hours.

2. Materials and Methods

2.1. Materials

2.1.1. Vegetable material

The fruits of *Raffia sese de Wild* used in this study were collected alongside the Congo River in Nganda sese village (February, 2017). This village is located in the City of Kinkole at the east part of Kinshasa/ Democratic Republic of Congo (DRC).

The plant material was identified at the National Institute of Research and Agronomic Studies (INRA) located at the Faculty of Sciences of the University of Kinshasa under voucher. The collected fruits were peeled and the pulps were detached from the almonds using a knife. Then, the pulps were dried in a drying oven at 50°C during 24 hours. The dried pulps were pulverized in a Butterfly crusher model: B-592. The obtained powder was kept in a desiccator prior to its utilization.

2.2. Methods

2.2.1. Extraction of oil

Several extraction methods of oils and greases from vegetable materials exist and depend on the raw material. However, different factors have to be taken into account in the choice of the extraction method in order to increase its velocity and the yield (18-19).

These parameters are the nature and the composition of the solvent, the particle size, the penetration capacity and diffusion of the solvent, the temperature, the duration of the extraction and the degree of agitation.

Since, the *Raphia Sese de Wild* oil is located into the pulp, the extraction by a soxhlet using petroleum ether solvent is adapted. This solvent has a boiling point between 40-60°C at atmospheric pressure.

2.2.2. Preparation of biodiesel starting from vegetable oil

Among the four principal methods of biodiesel synthesis starting from vegetable oils, such as transesterification, pyrolysis, dilution and microemulsion; the transesterification method was used due to different reasons explained before [6-14].

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 [20-21]. Figure 1 shows the diagram of transesterification in three stages.

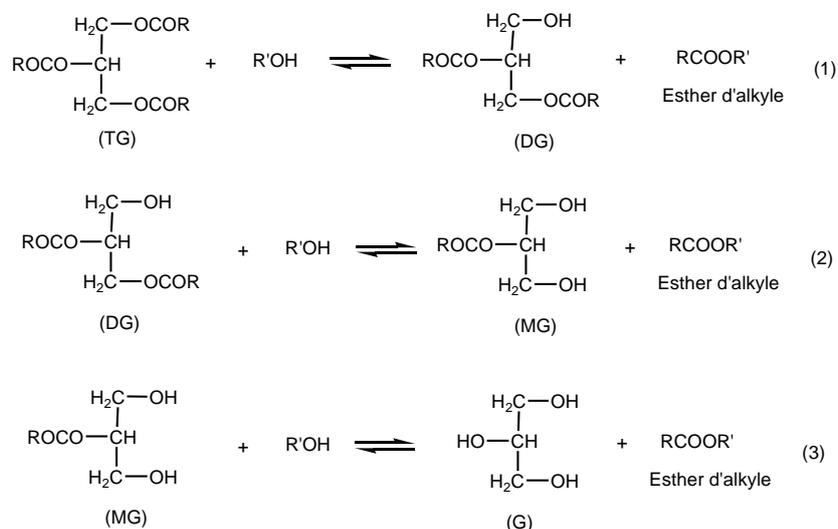


Figure 1: Successive steps of transesterification reaction

However, there are some factors that affect mainly the output of the reaction. Among them, the relationship between the quantity of catalyst and of alcohol, the temperature, the pressure, the response time, as well as the quantity of free fatty acids (FFA) and water content in oil (18, 19).

The transformation is complicated when oil contains large quantity of FFA, which can, easily, contribute to the

formation of the soap during the basic catalysis. The soap production prevents the separation of the produced biodiesel and glycerin [22-24].

To avoid the saponification reaction by decreasing the rate of FFA, the homogeneous catalysis is allowed. This method is improved in two steps; the first one is the acid catalysis and the last one is basic one [18-20].

The transesterification reaction is done by reacting the Raphia Sese de Wild oil and ethanol [18-19]. The choice of ethanol is due to its nontoxicity and availability (good price) in DRC.

2.2.2.1. Acid catalysed

In this catalysis, the free fatty acids should be transformed into esters. However, if the reaction duration is very long; some molecules of triglycerides will be also transformed into esters. If methanol is used as alcohol, the reaction products are methylesters; otherwise, it will be ethylesters, while using ethanol.

The principal factors which affect the effectiveness of the transformation by acid catalysis are the response time (between 2 and 3 hours), the molar reported ratio of alcohol (ethanol: 99.9%) and oil which are, generally, between 3:1, 6:1, 9:1 or 15:1. For this study, we considered a molar ratio of 6:1 [18 -20]. The preparation of the ethyl esters consisted in putting the oil of Raphia Sese de Wild in a balloon of 2 liters (volume) with 1% of concentrated H_2SO_4 mixed with ethanol (99.9%). The mixture was heated by reflux during three hours. Then after, it was putted in a funnel separating during 24 hours for separation.

2.2.2.2. Base catalysed

After the separation of the product obtained by acid catalysis, the dense phase was used in the basis catalysis. In a balloon of 2 liters, ethanol (99.9%) was mixed with 1% of concentrated sodium hydroxide (NaOH). The mixture was carried to boiling until the complete dissolution of the base. Then, the dense phase containing the non-reacting triglycerides of R.S.W. oil was mixed with the heated phase (the same report/ratio of 6:1 between the alcohol and the catalyst was used). The whole homogeneous obtained phase was heated with backward flow during 2 hours.

At the end of the reaction, the obtained mixture was placed in a funnel separating and then washed several times with distilled water. This operation was done when the mixture was still hot to avoid saponification reaction.

The dense part was discarded and the less dense part was constituted by the transesterified product (biodiesel).

2.2.3. Preparation of B100, B10 and B5 starting from the oil of R.S.W

The pure biodiesel named B100 and the mixtures biodiesel/reference diesel (B10 and B5) were prepared as follow:

- B100 (100% of biodiesel) was obtained by mixing the biodiesel from acid and basis catalysis of the oil of R.S.W.

- B10 and B5 were obtained by mixing (V/V) 90% and 95% of usual gas oil, called reference diesel in this work, with 10% and 5% of biodiesel (B100) of R.S.W, respectively.

2.2.4. Determination of the physicochemical properties of oil, B100, B10 and B5

The physicochemical properties of oil and the three types of biodiesel prepared starting from the oil of R.S.W, were determined in the respect of the standard characterization of the ASTM (American Society for Testing and Material) (29). The determined parameters were: the density at 20°C, the kinematic viscosity at 40°C, the flash point, the cetane number, the ash content, the residual carbon, the corrosion on the blade of cooper, the flow point, the water content and the curves of distillation.

3. Results and discussion

3.1. Content of oil extracted from fruit pulps of R.S.W

Table 1 gives the content of the oil extracted from the pulp of the fruit of R.S.W. using petroleum ether as solvent and calculated according to the following relation:

$$T (\%) = \frac{m_o}{m_p} \times 100 \quad (1)$$

Where m_o and m_p represent the mass in gram of the extracted oil, and the mass in gram of the dry RSW powder, respectively. T(%) is the extraction yield of the RSW oils.

The extraction yield found in this work is similar to that reported by Silou and his colleagues when using the Congo's species [25].

Table 1: Extraction yield of *Raphia Sese de Wild* oils collected in Nganda sese/Kinshasa/DRC

Experience	Dry powder : m_0 (g)	Extracted oil : m_p (g)	Extraction yield : T (%)
1	1,800	700.992	38.944
2	660	244.998	37.121
3	850	324.997	38.235
Average	3310	1270.987	38.398±0.28

The results presented in Table 1 are the average of 5 extraction operations. To synthetize the Table, we consider that experience 1 is an average of 5 operations.

3.2. Efficiency of the ethyl ester synthesized starting from the oil of R.S.W

Table 2 gives the total synthesis efficiency with homogeneous catalysis (acid and basic) realized from the oil of

Raphia Sese de Wild.

This output is calculated using the following relation:

$$R (\%) = \frac{V_i}{V_o} \times 100 \quad (2)$$

Where V_i represents the volume, in ml, of the synthesized biodiesel (ethylesters) and V_o the corresponding volume of *Raphia Sese de Wild* oil used in the transesterification reaction, in ml.

Table 2: Efficiency of the ethylesters obtained from homogeneous catalysis of *Raphia Sese de Wild* oil

RSW oil initial volume, V_o (ml)	1,500
Acid catalysis V_{a1} (ml)	1,100
Acid catalysis V_{a2} (ml)	400
Base catalysis V_{b1} (ml)	396
Base catalysis V_{b2} (ml)	4
Both catalysis Efficiency (%)	99.73

Where: V_{a1} is the volume of the biodiesel obtained from acid catalysis; V_{a2} is the volume of the dense phase from acid catalysis; V_{b1} represents the volume used for base catalysis from the dense acid catalysis phase and V_{b2} represents the residual volume from base catalysis and finally V_o represents the *Raphia sese de wild* oil initial volume used for the reaction.

The acid catalysis gives, after 3 hours, 73% of biodiesel calculated from the initial volume of *Raphia Sese de Wild* oil used in the reaction. For the base catalysis, the efficiency is 99% calculated from the residual volume from the acid catalysis (non reacted part).

To determine the total yield of the homogeneous catalysis, the efficiency of the two catalyzes (acid and base) are summed and gives 99,733%. In our knowledge, no work has been reported in the literature on the transesterified *Raphia Sese de Wild* oil for the biodiesel production.

3.3. Physicochemical characteristics of the oil and the biodiesels of R.S.W

The physicochemical characteristics of the oil and the biodiesel (ethylesters) from RSW were determined in accordance with the standards ASTM which are the reference specifications in DRC.

Table 3 gives the values of the whole parameters and the specify ASTM method used for their determination.

Table 3: Comparison of the characteristics of the oil and biodiesels prepared starting from the oil of the fruit pulp of *Raphia Sese de Wild*

Characteristics	ASTM Limits	Methods	Crude oil	B100	B10	B5	G0
Density at 15°C (Kg/L)	0.8200-0.8800	ASTM D4052	0.8808	0.8715	0.8558	0.8533	0.8495
Viscosity at 40°C (cst)	2.0 à 6.0	ASTM D445	6.284	5.511	4.186	4.000	3.9
Flash Point (°C)	60 Min	ASTM D93	129	52	67	74	70
Cloud Point (°C)	+6 Max	ASTM D97	+4	00	-9	-18	-15
Carbon corand residue dist. residue	10% 0.15 Max	ASTM D189	0.254	0.248	0.100	0.060	0.05
Ashes (%)	0.01 Max	ASTM D428	0.003	0.006	0.005	0.005	< 0.01
Total Sulfur (%)	0.05 Max	ASTM D4294	0.022	0.023	0.050	0.062	0.043
Water content (%)	0.05 Max	ASTM D95	0.000	0.000	0.000	0.000	0.000
Distillation recovery at 360°C	90 Max	ASTM D86	-	94	92.5	93	96.2
Copper strip corrosion (3 hours at 50°C)	1a	ASTM D130	1b	1a	1a	1a	1a
Cetane number	45 Min	ASTM D976	-	42	49.5	50	48.5

Legend:

Crude oil represents the pure *Raphia Sese de Wild* oil obtained by soxhlet extraction; B100 represents the pure biodiesel obtained from both homogeneous catalysis (acid and base); B10 is the mixture between 10% of the biodiesel and 90% of the reference diesel; B5 is the mixture between 5% of the biodiesel and 95% of the reference diesel; G0 is the reference diesel (fossil gazole); 1a is the acceptable limit of the color of the copper strip compared to the standard specifications where 1b denotes the beginning of its corrosion in the presence of the considered fuel.

3.3.1. Density

According to the result as shown in Table 3, the oil of *Raffia sese of Wild* with a density of 0.8808 deviates slightly from the ASTM limits. Several scientific researchers relate that the high density of oils has a harmful effect on the diesel engines [26 , 27].

Indeed, oils have a high inertia than the reference diesel for the same injection pressure. Thus, the high density of vegetable oils will lead to an increase length of the fuel jets, entraining them at the bottom of the combustion chamber. On the other hand, the same Table shows clearly the fact that the biodiesel (B100) and its mixtures (B10 and B5) with the reference diesel present density values situated in the interval of the ASTM standards limits and are between 0, 8533 and 0,8715.

3.3.2. Viscosity

The viscosity has a direct relation with the fluidity of the oils. In our case, by comparing the results presented in Table 3; the oil of R.S.W. has a viscosity value of 6,284 which is higher than the limit fixed by the standard ASTM D445. However, after transesterification, its values are in the average fixes by this specification for the three biodiesel, although higher than that of the reference diesel.

It should be noticed here that the interest of the utilization of ethylesters, compared to their oils, is their lowest viscosity values. Indeed, viscosity decreases by the command of a factor from 11 to 17 times for all oils. This decreasing is very important because it allows a better atomization by the injectors and thus, a better combustion [18, 27].

3.3.3. Flash Point

The flash point is the lowest temperature at which the application of a flame causes the ignition of a vapor portion under specific conditions of test. The value of the flash point of oil is 129°C whereas those of the ethyl esters are between 52 and 74°C. These values are similar to the standards of ASTM D93 that fix the minimal value at 60°C, as indicated in Table 3. While comparing all the values (reference diesel, biodiesel B100, B10 and B5), the biodiesel B100 and B5 are less dangerous to handle than the reference diesel and B10.

3.3.4. Cloud Point

The flow point represents the lowest temperature at which the biodiesel starts freezing when it is cooled, without agitation, under standardized conditions. By comparing the results as presented in Table 3 with the standards of ASTM D97, the three biodiesels revealed cloud point values in conformity with the reference diesel and the ASTM norms.

3.3.5. Cetane number

The cetane number is used to appreciate the autoignition aptitude of a diesel fuel on a scale of 0 to 100. It measures the aptitude for the lighting of a fuel under the pressure effect. This parameter characterizes the time between the injection and the combustion of a fuel. Its highest value shows that the fuel is easily flammable even in a cool condition. Usually, vegetable oils present low values of cetane number compared to diesel [28].

Indeed, more the value of the cetane number is high; more the autoignition time of fuel is short. In this condition, the combustion is reached easily with a high percentage of burned matters (hydrocarbons, monoxide of carbon, etc.).

The results indicated in Table 3, show clearly that, cetane numbers of biodiesel (B100) and the blended biodiesel (B10 and B5) are acceptable with that of the standards ASTM D976 value as compared to the reference diesel.

The other parameters such as the ash content, the sulfur total, the water content, the corrosion on the copper blade and the residual Conradson carbon agree well with the standards specifications by comparison with the ASTM value of the reference diesel.

However, except the density and the viscosity values compared to the reference diesel, the RSW oil can be used as fuel in replacement of diesel in the compression ignition engine.

3.3.6. Distillation recovery

Figure 2 shows the distillation curves of the Raphia Sese de Wild oil and the biodiesels in comparison with the reference diesel.

The characteristics of distillation (volatility) of hydrocarbons have a significant effect on their safety and their handling in the case of the fuels and solvents. The range of boiling provides information on the composition, the property and the behavior of fuel during their storage and their utilization. Volatility is the determining principal cause of the tendency of a mixture of hydrocarbons to produce the potentially explosive vapor.

The characteristics of distillation are very significant for the gasoline engines and of planes. They affect the engine start, the heating and the tendency to the formation of the vapor buffers at a high temperature. The operation characteristics of such engines at high altitude are also due their capacity of volatility. The presence of the chemical compounds with high boiling point can, significantly, affect the degree of formation of the solid particles during the combustion.

It is necessary to include the distillation characteristics in the main parameters improving the petroleum products quality. For the trade agreements, the applications of process of refinery control and for the compliance with the rules of standardization, the distillation parameters must to be taking into account (29). Referring to the ASTM D86, 100 ml of the sample was placed in distilling flask closed of a cork stopper transpierced of a thermometer. The first value of the temperature of which the first vapor condensed corresponds with the initial point of distillation. The end of this test corresponds to 90% of distilled volume at 360°C. Figures 2 (a, b, c and d) represent successively the various curves of distillation of crude oil, B100, B10 and B5 compared to the reference diesel (G0). The different values indicating the end of the distillation for all the studied samples are presented in Table 4. It puts forward the distilled volume of B100, B10 and B5 at 360°C compared to the volume of GO. We note that for the crude oils, at this temperature, distilled volume is largely lower than 90%.

Table 4: Distilled volumes at 360°C

Sample	Volume (ml)
B100	94
B10	92.5
B10	93
GO	96.2

Distillation gives significant ideas of the proposals relating to light, average hydrocarbons and heavy contents in a fuel.

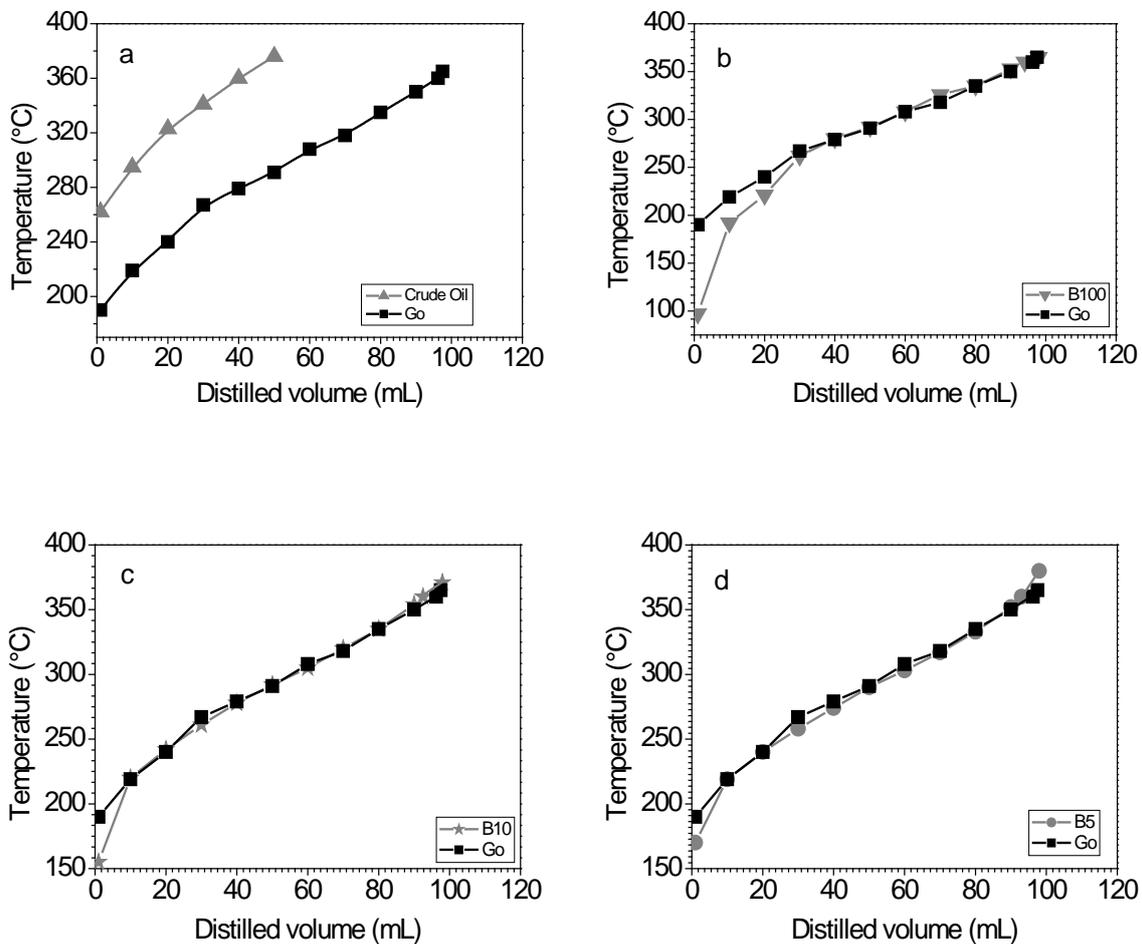


Figure 2: Curves of distillation of crude oil and biodiesels compared with the reference diesel (GO)

The item 50% of distilled volume is the average point of the level of volatility of the whole of the product and gives an indication on the normal rate of the engine. This point allows the determination of the calculated cetane index. Due to the results as shown in the previous figures, the appearance of heavy products is observed. The item 90% is the point which indicates the high content in heavy hydrocarbons at high boiling point. Referring to the ASTM D86 standards, 90% of the volume of the fuel must be, at 360°C, in the vapor state or must be vaporized. We note clearly that the three prepared biodiesel agree satisfactory with this standard. According to the results presented in Table 4, 96.2, 94, 92.5 and 93% v/v are the percentage of volume reached at 360°C for the reference diesel, B100, B10 and B5, respectively. Considering these similarities, the biodiesels produced from the oil of *Raphia Sese de Wild* can be used in a diesel engine in replacement of traditional diesel successfully.

4. 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. Biodiesel or biofuel is one among the different renewable and sustainable energy resources and must be promoted. This biofuel is biodegradable, its calorific value is significant and its utilization allows the reduction of the gas emission for purpose of greenhouse.

This study showed that the transesterification of the oil of *Raffia sese de Wild*, gives a biodiesel presenting good physicochemical properties compared to the reference diesel (fossil fuel), according to the standards of ASTM. The three types of biodiesel (B100, B10 and B5) can be used in replacement of the traditional fossil fuel. Furthermore, a mixture including more proportion than 10% of biodiesel can be also promoted due to the results obtained in this study. Finally, due to the cloud point value illustrated in this work, all these fuels can be also used in the winter period, even though, DRC has a tropical and equatorial climate.

5. Recommendations

The results obtained in this study show that the three types of the biofuels produced from the pulp of *Raphia sese de Wild* (B100, B10 and B5) can be used in a diesel engine. The authors recommend a complementary study of their combustion characterization in a diesel engine to provide more supplementary results for their utilization as fuels. The variation of the amount of the catalyst (base and acid) and the ratio between the alcohol and the oils to improve the transesterification reaction of by using an heterogeneous catalyst.

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