

## **Production of Biodiesel by Transesterification Reaction of Waste Cooking Oil**

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### **Authors' contributions**

*This work was carried out in collaboration among all authors. Authors DRTT, SBLN and GNNA designed the study, performed the statistical analysis, wrote the protocol and wrote the first draft of the manuscript. Authors SBLN, SGA, RDTN and TNK managed the analyses of the study. Authors DRTT, PANK, GNNA, IHTK, AB and SBLN managed the literature searches. All authors read and approved the final manuscript.*

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### **ABSTRACT**

Waste cooking oils are an agro-food waste with adverse effects on the health of living organisms and the environment. The main objective of this work is to valorize waste cooking oil for the synthesis and physicochemical characterization of biodiesel. The method used is based on the

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transesterification reaction of the oils using methanol and a basic homogeneous catalyst. In this study we employ waste from refined palm oil used for frying doughnuts. After optimization a reaction time of 2 hours, KOH catalyst, and a molar ratio of 9:1 were selected to obtain a good quality biodiesel. Physicochemical characterization was performed on the biodiesel to obtain its density, viscosity, calorific value, acid number, saponification index and IR spectral features. The analysis shows that the biodiesel obtained after transesterification has physicochemical characteristics similar to those of diesel and is consistent with American standards.

*Keywords: Biodiesel; viscosity; transesterification; basic catalysts; waste cooking oil.*

## 1. INTRODUCTION

Nowadays, the industrial and transport sectors are increasingly intensive and require use of fossil fuels. The increase in the consumption of these fuels is linked to a more rapid depletion of valuable reserves and risk for the future. Moreover, these sectors are considered the main sources of greenhouse gas emissions (CO<sub>2</sub>, SO<sub>2</sub> and NO<sub>x</sub>) [1] and therefore linked to global warming [2]. These factors as well as the fluctuation in the cost of oil have prompted researchers to urgently embark upon finding alternative and more sustainable sources to replace crude oil. The solutions to be implemented are not only those aimed at making a transition to renewable, inexhaustible, and non-polluting energy sources, but also the use of energy production methods by recycling [1]. Indeed, the return to biodiesel initiated more than a century ago proves to be one of the possible solutions. Biodiesel, which is biodegradable and non-toxic, has the added advantage of reducing oil consumption and greenhouse gas emissions [3,4].

The history of biofuel began in 1892 when Rudolph Diesel, the inventor of the diesel engine, first tested peanut oil in the internal combustion engine [5]. The major difficulty with the direct use of vegetable oils is related to their high viscosity which is about 11 - 17 times higher than that of diesel fuel) and the low volatility that leads to the formation of deposits in engines [6]. In order to change their physico-chemical properties to make them similar to those of petrodiesel, triglycerides are transformed into alkyl esters by a transesterification reaction with methanol or ethanol in the presence of acidic, basic or enzymatic catalysts [7]. Transesterification is a chemical process in which a vegetable or animal oil is mixed with an alcohol in the presence of a catalyst to obtain an alkyl ester, commonly known as biodiesel. The change from an oil to an ester reduces the molecular weight to one-third

of that of the oil, reduces viscosity by a factor of eight, reduces density, and increases volatility [8].

The use of waste frying oils, slaughterhouse fats, or fish oils is very interesting from an ecological point of view and it avoids the dumping of these oils into the environment [9]. In addition, used oils cause health problems in humans such as respiratory tissue irritation, and carcinogenic effects on the prostate and lung [10]; hence, the idea of converting them into biodiesel. According to statistics provided by the Groupement d'Initiative Commune (GIC-Bellomar), hotels in Douala (Cameroon) dispose of an average of 30 litres per week of used frying oil into the environment. The main objective of this work is to optimize experimental conditions for triglyceride transesterification in order to produce biodiesel from used frying oils meeting international standards. These biodiesels are assessed for performance in a diesel engine.

## 2. MATERIALS AND METHODS

### 2.1 Presentation of Samples

The raw material used to carry out this work was refined palm oil, which had been used for frying fritters but not exceeding five times. The reagents used for the experiments were sodium hydroxide (NaOH) and potassium hydroxide, both from Fisher Scientific, and methanol (CH<sub>3</sub>OH) supplied by Louis Dreyfus Commodities.

### 2.2 Biodiesel Production and Testing in an Engine

#### 2.2.1 Production method

The used frying oil was filtered in order to remove any particulate impurities and solids, and then placed in an oven at a temperature of 105°C to remove any surplus of water. The experimental device was a three-necked round

bottom flask with a capacity of 1000 mL placed on a hot plate/magnetic stirrer, with a fitted condenser as shown in Fig. 1.

100 g of waste frying oil (WCO) was introduced into the three-necked round bottom flask and heated to 60°C with magnetic stirring. Once the temperature was stabilised, a previously prepared potassium methylate solution CH<sub>3</sub>OK (MeOH + KOH) was added to the oil under stirring. The masses of methanol used were calculated from the equation below as a function of the molar ratio of alcohol to oil used.

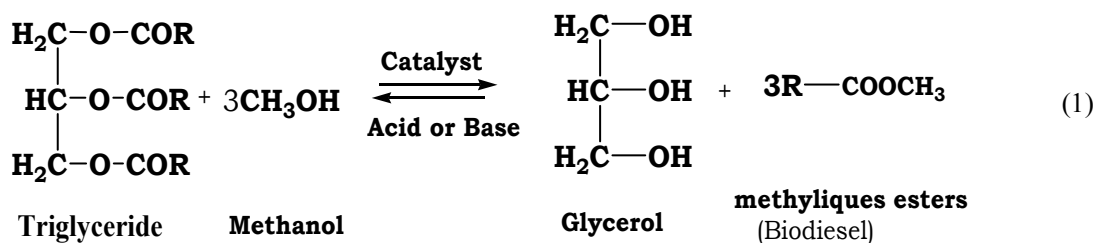
The reaction temperature was maintained at 60°C (to avoid evaporation of methanol with a

boiling point at 65°C) for a given time. The following studies were carried out: the influence of the reaction time (1 h, 2 h, 3 h), the molar ratio of alcohol/oil (6:1, 9:1, 12:1), and the type of catalyst used (KOH, NaOH), the yield was determined in each case. The catalyst/oil mass percentage used was 1%. Once the reaction was completed, the mixture was introduced into a separating funnel to separate the biodiesel from the glycerol. The biodiesel was washed several times (to remove impurities such as residual glycerin, excess alcohol, traces of catalysts, soaps and salts) with hot water and then dried in an oven at a temperature of 110°C. The following figures illustrate the separation and washing steps.



Fig. 1. Experimental device for transesterification reactions

The transesterification reaction can be described by the following equation:





**Fig. 2. Separation operation**



**Fig. 3. Washing biodiesel**

### 2.2.2 Characterization

The resultant biodiesel was characterized by determining the density, viscosity, acid number (AFNOR) and saponification index (AFNOR). An infrared spectroscopy analysis was carried out to investigate the nature of the functional groups present in the product. For the different properties, the following methods and protocols were used.

#### 2.2.2.1 Density

The relative density of a substance is the density of the substance divided by the density of the reference body at the same temperature. We measured the masses of a known volume of oil and biodiesel with a sartorius scale. The knowledge of mass and volume allows us to calculate the density and deduce the density of the sample by the following relationships:

$$\rho = \frac{m}{V} \quad (2)$$

and

$$d = \frac{\rho}{\rho_e} \quad (3)$$

m: mass of the sample  
V: sample volume  
 $\rho$ : density of the sample  
 $\rho_e$ : density of water.

#### 2.2.2.2 Viscosity

Viscosity is defined as the resistance to uniform flow without turbulence occurring. Viscosity was

determined using an NDJ-5S digital rotary viscometer.

#### 2.2.2.3 Acid number

The acid number is the mass in mg of basic solution that can neutralize one gram of ester. The acid number determination consisted of dissolving 1 g of fat in 10 mL of ethanol, adding 3 drops of phenolphthalein, then titrating the mixture with an ethanolic solution of 0.1 M potassium hydroxide until the pink coloration persists [11]. The acid number was calculated with the following formula.

$$I_a = 56,1 \frac{VC}{m} \quad (4)$$

C: Concentration of the ethanolic KOH solution used for titration.

V: Volume of KOH ethanolic solution used (mL)

m: mass (g) of the sample

#### 2.2.2.4 Saponification index

The saponification number determines the mass of KOH in mg required to saponify the combined fatty acids in a gram of fat. It is a reverse titration [11]. A fatty acid solution was reacted with an excess of KOH under heat. This excess was then back-titrated with a solution of hydrochloric acid (HCl). The saponification index was calculated with the following relationship.

$$I_s = 56,1 \frac{(V'-V)N}{m} \quad (5)$$

V': volume in mL of the 1 mol/L hydrochloric acid (HCl) solution for the blank titration.

V: Volume in mL of the 1mol/L hydrochloric acid (HCl) solution for sample titration.  
 N: normality of the hydrochloric acid solution  
 m: sample mass.

### 2.2.3 Testing biodiesel in a diesel engine

After characterizing the waste oil methyl ester (WOME) in terms of acid index and saponification, density, viscosity, we tested the performance with a KIPOR model KM 178F diesel engine connected to a dynamometer as

shown in Fig. 4. The dynamometer displays the torque value and rotation speed for this engine. These displayed values were used to calculate the engine power for each type of fuel using the following equation.

$$BHP = \frac{TN}{5250} \quad (6)$$

Where,

BHP: Brake horsepower, T: Torque in lbs.ft, N: Motor rotation speed, 5250: Constant.



Fig. 4. Dynamometer connected to the diesel engine

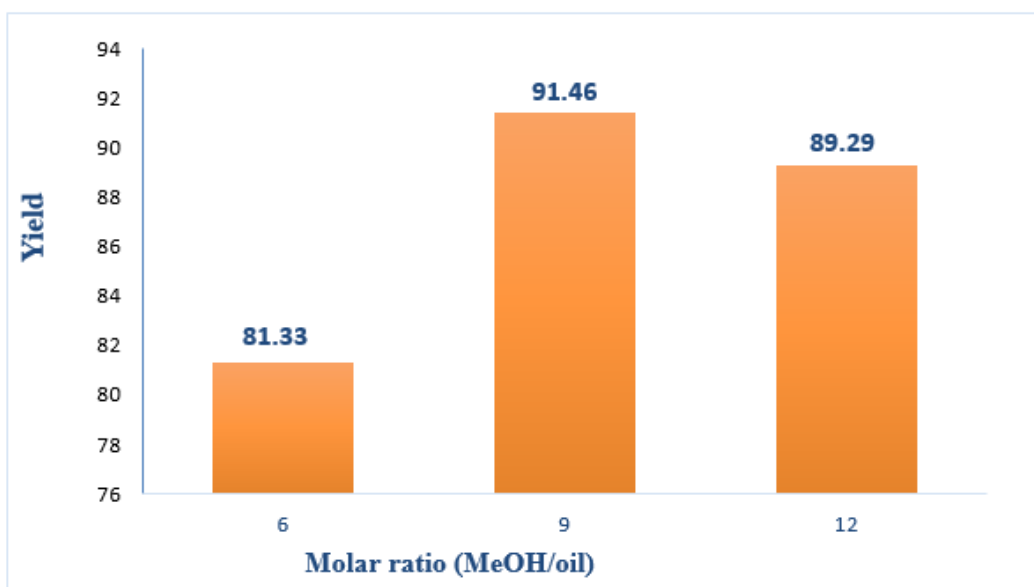


Fig. 5. Evolution of biodiesel yield as a function of the molar ratio MeOH/ WCO

### 3. RESULTS AND DISCUSSION

#### 3.1 Influence of the MeOH/WCO Molar Ratio on the Performance of the Transesterification Reaction

The molar ratio MeOH/WCO is one of the important factors influencing biodiesel yield. Transesterification of triglycerides is usually carried out with an appropriate amount of alcohol to shift the balance to the product alkyl esters. The study of the influence of the MeOH / WCO molar ratio (6:1; 9:1 and 12:1) was carried out at a temperature of 60°C for 2 hours of reaction with 1% by mass of KOH catalyst. Fig. 5 shows the evolution of the biodiesel yield (g biodiesel/g WCO) as a function of the molar ratio.

From the histogram in Fig. 5, it can be seen that the best biodiesel yield is obtained at a molar ratio of 9:1. However, the yield decreases for a molar ratio greater than 9:1. This could be due to the fact that the miscibility of glycerol in the ester-enriched phase increases with the molar ratio. Therefore, the separation process becomes more difficult [12]. This result is in line with the work of Jazie et al. [13]. Hayyan et al. [14] produced biodiesel from palm oil and found that the 10:1 ratio was the best ratio for generating good biodiesel quality. This result is also in agreement with that of Atadashi in 2016 who studied the effect of the molar ratio on the production of biodiesel from used frying oil [15].

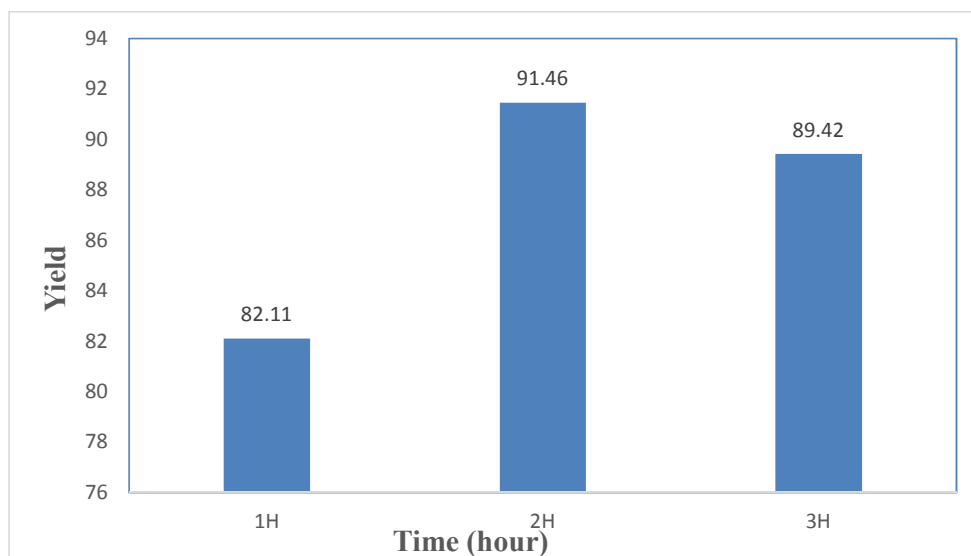
#### 3.2 Influence of Transesterification Reaction Time

The influence of various transesterification reaction times (1h, 2h, 3h) at temperature (60°C) was investigated (molar ratio MeOH/WCO of 9:1 and 1% by mass KOH). Fig. 6 shows the evolution of biodiesel yield as a function of reaction time.

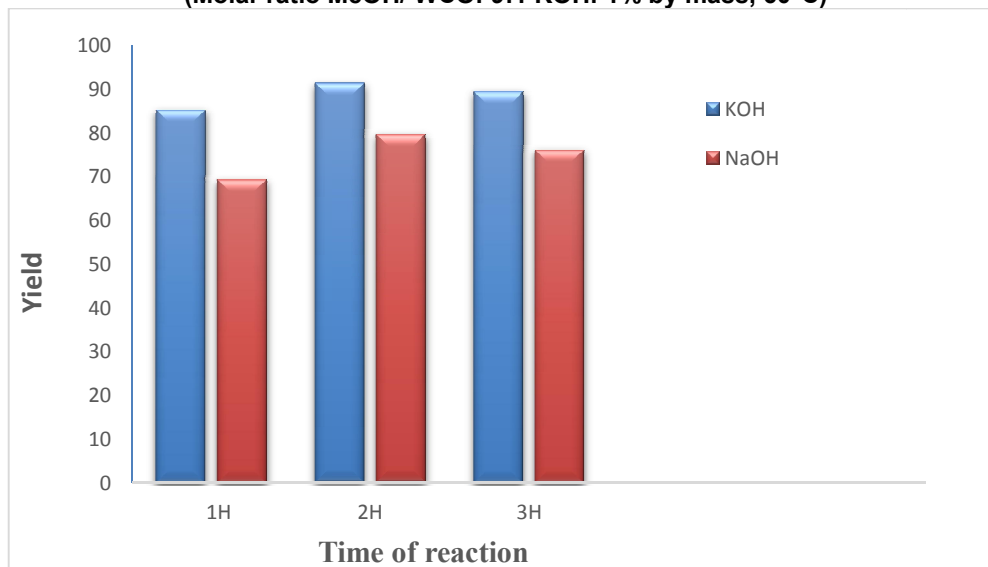
Fig. 6 shows that transesterification reactions at a temperature of 60°C produce the maximum biodiesel yield of 91.46% after 2 hours of reaction. After 2 hours of reaction, almost all the oil has been transformed into methyl esters leading to a maximum conversion. These results also show that the reaction is incomplete after one hour. On the other hand, a prolonged reaction time (more than 2 hours) leads to a decrease in yield because of saponification as a reverse reaction to transesterification. This reaction leads to a loss of esters and the formation of soaps.

#### 3.3 Influence of Catalyst Type on Biodiesel Yield

The influence of the two basic catalysts (KOH and NaOH) on the transesterification reaction was investigated using the molar ratio MeOH/WCO of 9:1 for 1 h, 2 h, 3 h and 1% by mass of catalyst. Fig. 7 shows the evolution of biodiesel yield as a function of time for these different types of catalyst.



**Fig. 6. Evolution of biodiesel yield as a function of reaction time (Molar ratio MeOH/ WCO: 9:1 KOH: 1% by mass, 60°C)**



**Fig. 7. Evolution of biodiesel yield as a function of time for different types of catalyst**

Examination of Fig. 7 shows that the better biodiesel yields were obtained with KOH catalysed reactions. This is because NaOH and KOH catalysts each contain sodium and potassium cations which affect reactivity. This result is in agreement with results reported by Chitra et al. [16] on optimization of experimental conditions for biodiesel production from alkali-catalyzed transesterification of jatropha curcus oil. KOH was found to always give the best yields.

### 3.4 Variation of Viscosity as a Function of Temperature

High viscosity is a major disadvantage in the direct use of oils as biofuel, because it is closely related to fluidity [17]. Fig. 8 shows that the viscosities of the biodiesels are lower than that of used cooking oil at all temperatures. This lower viscosity of biodiesel compared to that of waste cooking oil is consistent with formation of methyl esters and removal of glycerol. The main advantage of using esters compared to their oils is the lower viscosity. This lower viscosity is a major advantage due to better atomization by the injectors and therefore cleaner combustion [17]. We also note that the viscosities of the biodiesels are very close to those of diesel at different temperatures and that these viscosities comply with the European standard [18]. The viscosity values of the biodiesels obtained are

comparable to those reported by Ayhan et al. in 2008 [19].

### 3.5 Biodiesel Characterization by Infrared (IR) Spectroscopy

The IR spectra of our raw materials and reaction products were recorded following a well-established procedure with a Nicolet iS5 FTIR spectrophotometer located in the Department of Chemistry at the University of Dschang. The IR spectra below (Fig. 9) show the characteristic absorption bands of the functional groups in used frying oil and biodiesel (produced with a molar ratio of 9:1). The following information can be obtained from the interpretation of the IR spectra of biodiesel.

When we compare the spectra of biodiesel to that of cooking oil, we find that they are almost identical. This could be explained by the fact that spectra for waste cooking oil and for biodiesel are both dominated by alkyl esters. A slightly lower intensity in bands of biodiesel compared to those for waste cooking oil correspond to methyl esters compared to glycerides as shown in equation (1).

### 3.6 Physico-chemical Characterisation of Biodiesel

The physico-chemical characteristics (density, viscosity, acid number, and saponification number) of used frying oil, diesel oil and the

various biodiesels obtained by potassium (KOH) and sodium (NaOH) catalysed transesterification in the ratio 9:1 at a temperature of 60°C are given in Table 2.

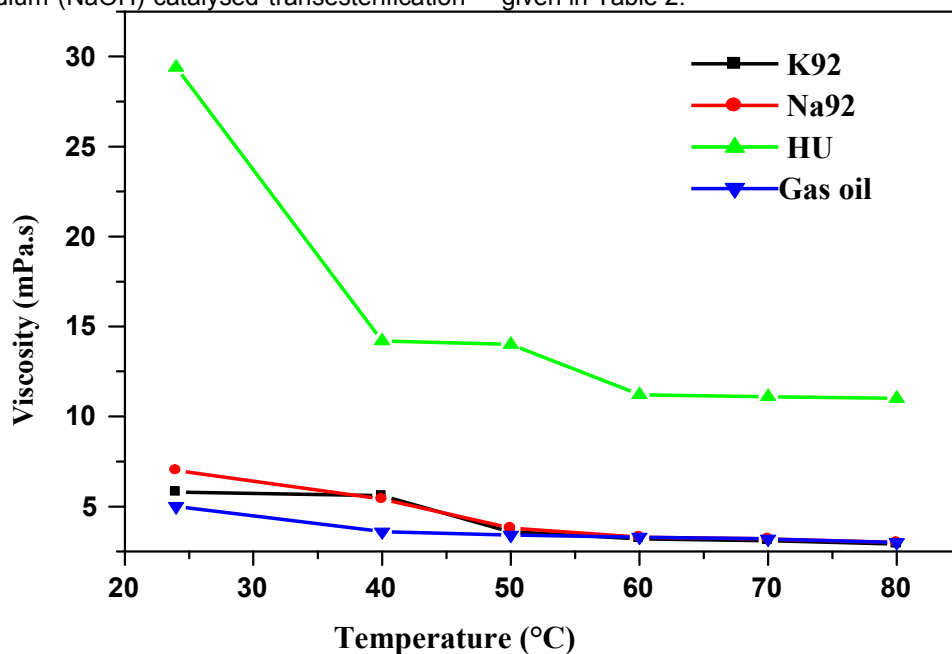


Fig. 8. Viscosity variation of biodiesels, waste oil and diesel as a function of temperature

Table 1. Functional groups present on biodiesel IR spectra (wave numbers in  $\text{cm}^{-1}$ )

Functional groups	C-H aliphatic	C=O	C=C	C-O	=C-H vinyl
Used frying oil	3000-2900	740	1745	1250-1150	735
Biodiesel	3000-2900	740	1750	1155	730

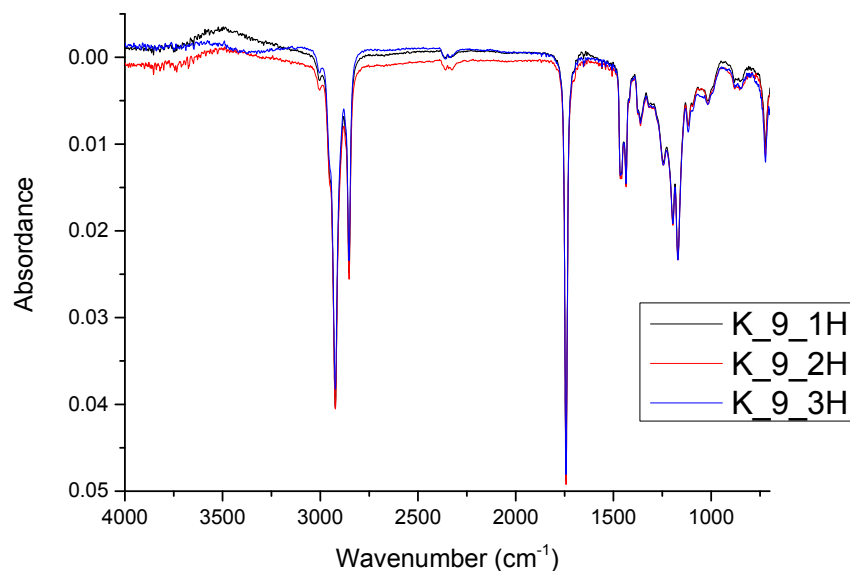


Fig. 9. IR spectra of biodiesels obtained by KOH catalysis (1%) in the ratio 9:1 for different reaction times (1h, 2h, 3h)



**Table 2. Physico-chemical characteristics of diesel and biodiesels**

<b>Characterization</b>	<b>Spent frying oil</b>	<b>Biodiesel (KOH)</b>	<b>Biodiesel (NaOH)</b>	<b>Petrodiesel (Diesel)</b>	<b>American Standards</b>
Relative density at 25°C	0.90	0.87	0.85	0.84	0.86-0.90
Viscosity at 30°C (mPa.s)	18.40	5.60	5.40	4.80	1.9 à 6.0 mm <sup>2</sup> /s
Saponification index (mgKOH/g)	193.00	184.33	185.12	/	170.4. – 196.20
Acid number (mgKOH/g)	2.2	1.10	1.40	0.98	0.11-0.50

**Table 3. Reading dynamometer parameters**

<b>ME</b>	<b>Quantity</b>	<b>Time</b>	<b>Load</b>	<b>B100</b>		<b>Calorific value (MJ/kg)</b>
				<b>Torque</b>	<b>Engine speed (rpm)</b>	
Diesel	500 mL	60 s	Low 25%	3	23	54.85
			Mid 50%	4.4	38	
			High 100%	6	48	
WOME (Waste oil methyl ester)	500 mL	60 s	Low 5%	0.8	15	41.90
			Mid 50%	3.4	23	
			High 100%	5.5	40	

*B100: not blend fuel*

From the results shown in Table 2, it is clear that biodiesel has a lower density than waste cooking oil. This lower density of biodiesel relative to waste cooking oil is linked to formation of methyl esters by removing glycerol. Moreover, the biodiesels obtained have a relative density that varies between 0.854 and 0.867. These results are close to those given by the European standard and similar to those found by Firestone et al. [20]. However, the work of Abollé et al. has shown that the high density of oils can also have adverse effects on diesel engines [21].

The acid number is a function of free fatty acids and it characterizes the state of alteration of oils by hydrolysis. Table 2 shows that biodiesel has a relatively low acidity compared with used oil. Indeed, during cooking or storage the oils are oxidized and free fatty acids are formed, which could justify the high acid number values for waste cooking oil. Furthermore, the values of the acid indices of the biodiesel produced is greater than  $0.8 \text{ mgKOH.g}^{-1}$ , which is the limit value required by standard ASTM D675. Felizardo et al. (2006) also studied the optimal conditions for the production of biodiesel from used frying oil [22, 23]. They suggested that a pretreatment step would be necessary to obtain a higher percentage of good quality fatty acid methyl ester.

In Table 2, we note that the saponification indices of biodiesel samples are lower than those of waste cooking oil ( $193.2 \text{ mgKOH/g}$ ). This could be explained by the fact that biodiesel is a fuel that has been refined, treated by transesterification (equation 1) to lower its acidity and to eliminate undesirable constituents (GLA, phospholipids, etc.). A high saponification index results in a high level of short-chain fatty acids and a higher glycerol content [24].

### 3.7 Biodiesel Performance in Diesel Engines

Table 3 shows the effects of two fuel types, Diesel and Waste oil methyl ester on the performance of a diesel engine at different load rates 25, 50 and 100%. It shows that regardless of the fuel type, the engine speed and torque were highest with a diesel engine at running at 100%. With the same diesel, the engine torque and speed logically increase with increasing engine load. The same observations are made for a biofuel feed from frying oil residues, although the values are lower.

From these results, it can be seen that diesel fuel naturally gives the better engine performance, which is directly proportional to the power of the engine. Indeed, the higher the engine torque and speed, the greater the power developed by the engine. Since the engine used for the tests is diesel-powered, it is natural that its performance is optimal for its reference fuel.

It can also be seen that biodiesel obtained from waste cooking oil presents results that are generally not significantly different from those of conventional diesel except for results at 25% load. At a low engine load, the starting temperature would be low. In diesel engines, a relatively low compression ratio leads to a low piston falling speed in the combustion chamber.

In the context of the fight against global warming, biofuels produced from waste cooking oils are of great importance. Indeed, they could be added to obtain a diesel-biodiesel blend on one hand, or even used directly as a substitute for diesel for the reduction of the use of fossil fuels on the other hand. Diesel engines in this sector work at load rates of between 75 and 90%. These values make it possible to calculate the engine power for each fuel used and these values are recorded in Table 3. Examination of Table 3 show that the calorific values obtained are close for the two types of fuel. This shows that biodiesel produced from waste cooking oil has acceptable energy parameters for powering a diesel engine.

## 4. CONCLUSION

Biodiesel is one of the most important renewable energy sources. It is biodegradable and its use reduces greenhouse gas emissions. In our study, we carried out transesterification reactions for the production of biodiesel from waste cooking oil and methanol mixtures in basic medium (KOH and NaOH). The influence of operating parameters, reaction temperature, reaction time, and MeOH/ WCO molar ratio was studied. The performance of biodiesel on a diesel engine was also evaluated. This study shows that biodiesel produced by transesterification reaction of waste cooking oil at  $60^\circ\text{C}$  has physical and chemical properties comparable to those of diesel and thereby meets international standards. Analysis of the optimization results shows that a molar ratio MeOH/WCO of 9:1, a reaction time of 120 min and the use of KOH as a catalyst at  $60^\circ\text{C}$  led to a maximum efficiency of 91.46%. The biodiesel produced has been tested in an engine, it follows from this test that biodiesel has slightly

lower energy properties than conventional diesel. The overall results showed that waste cooking oil could be used to produce biodiesel from waste oil as a contribution to waste management and recycling processes.

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## COMPETING INTERESTS

Authors have declared that no competing interests exist.

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