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Synthesis of Biodiesel by Transesterification of Used Frying Oils (UFO) through Basic Homogeneous Catalysts (NaOH and KOH)

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Abstract: The quest for an alternative sustainable source without petroleum technology and its refining has prompted the development of biofuels, such as biodiesel, from the transesterification of new or utilized vegetable oil. This work is devoted to the investigation of the transesterification of a used vegetable oil and optimization of the various parameters influencing the synthesis of biodiesel, such as the molar proportion (alcohol/oil), the amount of catalyst added and their weight percentage, the type of alcohol, the temperature T(°C) and the reaction time. From this standpoint, the current work's significant target is to propel the preliminary conditions of the transesterification response of fatty oils to create biodiesel from utilized vegetable oils. Diverse physicochemical characteristics were investigated (in terms of density, viscosity, acidity index, pour point, and flash point) to obtain biodiesel accordingly with international standards and commercial biodiesel.

Keywords: biodiesel; vegetable oil; alkyl esters; flash point; pour point; acidity index; viscosity.

1. Introduction

The manufacture of biodiesel represents an alternative source of fuel production; which is known as renewable [1-4], biodegradable [5,6], sustainable [7], clean and non-toxic [8], the resources are abundantly available [9], neutral in CO_2/GHG (greenhouse gases) [9-12], they cause negligible or zero SOx emissions [13,14], they provide lower NOx emissions [15,16], environmentally friendly [17,18], can be produced locally [19], they have a high flash point and less pollution [20-22], their production processes and/or biofuel farms are generally safe [23,24].

The applications of biofuels in life have been detailed in numerous publications [25–28]. These applications have radically changed and improved the lives of people all over the world, especially in the automotive field where biodiesel can be used both as an alternative to conventional diesel or as an additive, neat or blended [29], it has less amount of pollutants during and after the combustion process. Engine life can be improved thanks to biodiesel viscosity twice as high as petroleum diesel [30], also during the combustion of biodiesel, the reduction of total unburned hydrocarbons is greater than 90% [31]. Biodiesel is produced by converting vegetable oils, animal, or waste origin into alkyl esters [32-38]. The transesterification reaction is one of the most important methods used to convert vegetable oils into diesel fuel due to the process's low cost and simplicity. It is a process in which triglycerides are converted in the presence of an alcohol (methanol or ethanol), an ester, and glycerol catalyst [39, 40]. To minimize production costs, researchers are currently using leftover cooking oils and other inedible oils as raw materials [41-47].

This work aims to produce a biodiesel meeting the international quality standard, from used frying oil (UFO) by the reaction of transesterification. To achieve the best conversions of UFO, other parameters controlling the reaction of transesterification were studied, such as the molar ratio of alcohol to acid, the amount of catalyst, the time and temperature of the reaction. The biodiesel will be characterized and compared to petrodiesel and commercial biodiesel, which meets the international quality standard.

2. Materials and Methods

2.1. Starting materials.

The raw materials used to carry out this work are new vegetable oils of different origins (Sunflower, Soybean, Canola). Three types of used vegetable oils (HFU) with a frequency of use not exceeding 4 times.

2.2. Reagents.

The different reagents used for our experiments are sodium hydroxide (NaOH), potassium hydroxide (KOH), methanol (CH_3OH), ethanol (C_2H_5OH), and 2-Propanol (C_3H_8OH).

2.3. Biodiesel production.

A mixture of used frying oil from different sources (Soybean, Sunflower, Canola) and alcohol (Methanol, Ethanol, 2-Propanol) in the presence of a basic homogeneous catalyst (NaOH, KOH) undergone continuous stirring at different temperatures (ranging from ambient temperature to 72C°) in an Erlenmeyer flask. These working parameters can be changed in the manufacturing attempts of biodiesel by the transesterification reaction to optimize this reaction's conditions to increase the yield.

The solution resulting from the reaction is left to stand for the separation of the two phases; the glycerol, which has a greater density than biodiesel, is located at the bottom of the separating funnel after 24 hours of decantation.

The resulting biodiesel is placed in a separating funnel and then slowly rinsed with distilled water to remove the excess alcohol and catalyst. After rinsing biodiesel, it is recovered and dried with $Na_2\ SO_4$. The obtained product is weighed in order to determine its yield.

The yield of the reaction is calculated according to the following formula:

$$\eta(\,\%\,) = \frac{m_b}{m_h} * 100 \qquad (1)$$

With : m_b: weight of biodiesel;

m_h: weight of used frying oil;

The global chemical reaction is presented in Figure 1:

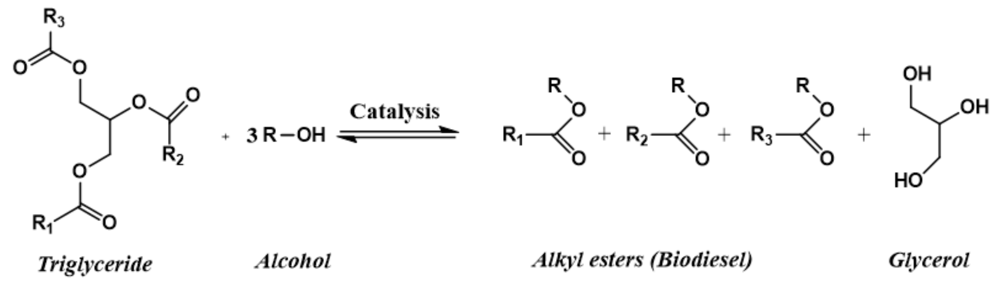

Figure 1. General reaction of the transesterification of triglycerides with alcohol (R-OH) where R1, R2, and R3 are the alkyl groups of fatty acids.

The transesterification reaction is affected by several varied parameters, and its degradation yield is limited by the temperature, the type of catalyst, the quantities, ratios of the used reagents, and the weight percentage of catalyst (KOH and NaOH)/UFO. Also, reaction time, type of alcohol, and source of oil are influencing parameters.

2.4. Physico-chemical characteristics of used frying oils, new oils, and biodiesel.

The physicochemical properties were determined according to international standards. In our study, we determined density (NFT 60-214), viscosity, acid index (NFT 60-204), open cup flash point (NF EN ISO 2592/01) for oils, a closed-cup flashpoint for biodiesel (ISO 2719/16), pour point (NF T60-105) and color (NF ISO 2049/98).

3. Results and Discussion

3.1. Study of the parameters influencing the transesterification reaction.

3.1.1. Temperature effect on the yield of biodiesel.

The used frying oil (UFO) transesterification reaction was performed using 1 wt% KOH and MeOH /UFO molar ratio: 6: 1. Temperatures of 18, 42, 54, and 72 °C were used for a reaction time of 60 min. Figure 2 exhibits the evolution of biodiesel's yield depending on the temperature of used frying oil from soybean.

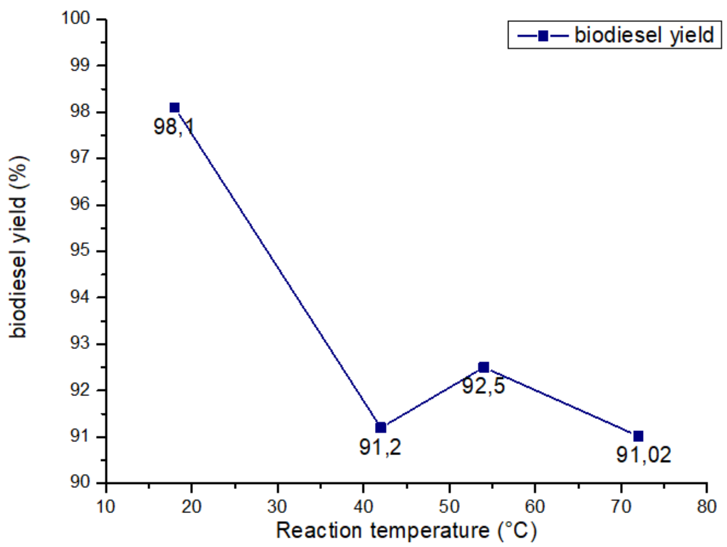

Figure 2. Evolution of biodiesel yield as a function of temperature (KOH: 1% by weight, MeOH/UFO molar ratio: 6: 1, 60 min).

Figure 2 shows that when the transesterification reaction is carried out at different temperatures, the maximum yield of biodiesel of 98% is obtained at a reaction temperature of 18°C (ambient temperature). Therefore, the reaction temperature seems to influence the yield of biodiesel is ambient temperature.

3.1.2. Influence of the reaction time on the yield of biodiesel.

The influence of various transesterification reaction times (10, 30, 60, 90, and 120 min) at room temperature (19 ° C) was studied (MeOH /UFO) molar ratio of 6: 1 and 1% by weight of KOH). Figure 3 shows the evolution of biodiesel yield as a function of reaction time.

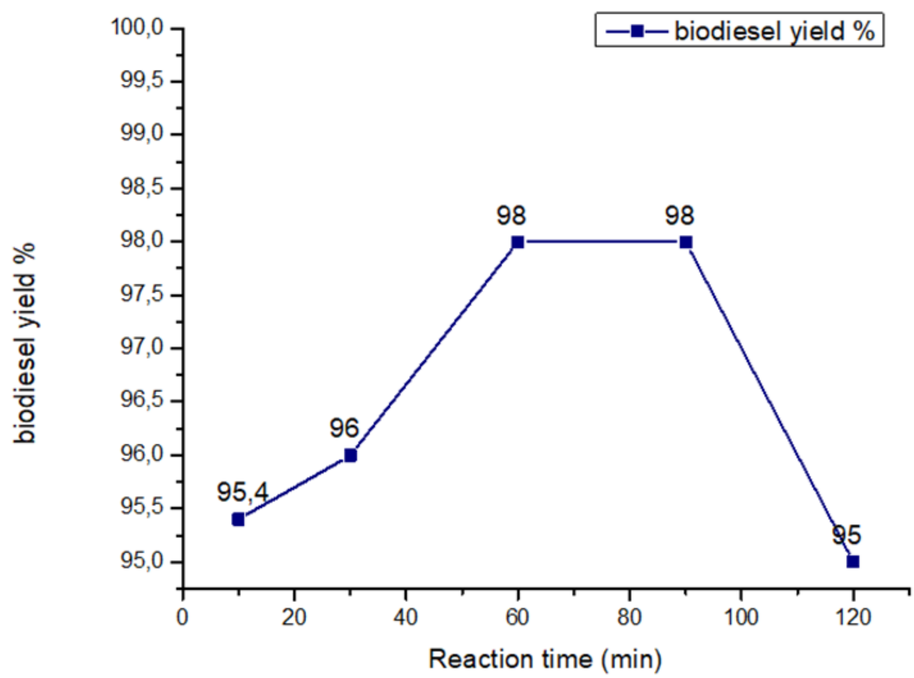

Figure 3. Evolution of biodiesel yield as a function of reaction time (KOH: 1% by weight, MeOH / UFO molar ratio: 6: 1, 19 ° C).

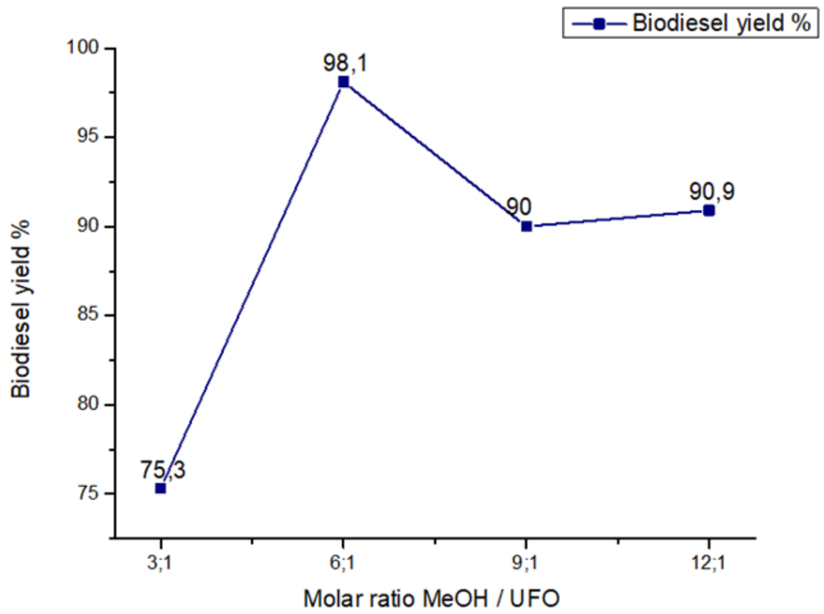

Figure 4. Evolution of the biodiesel yield as a function of the MeOH /UFO molar ratio (KOH: 1% by weight, 60min, 18 ° C).

Figure 3 shows that when the transesterification reaction is carried out at a temperature of 600°C, biodiesel's maximum yield is obtained after 60 min of reaction with a percentage of 98%. A yield of 95% is obtained after 10 min of reaction at room temperature. Therefore, the reaction time does not seem to have a great influence on the yield of biodiesel.

3.1.3. Influence of the MeOH / UFO molar ratio on the yield of biodiesel.

The study of the influence of the MeOH / UFO molar ratio (3: 1, 6: 1, 9: 1, and 12: 1) was carried out at room temperature (18 ° C) for 60 min of reaction with 1% by weight of KOH. Figure 4 shows the evolution of biodiesel yield as a function of reaction time.

The maximum biodiesel yield is of 98% corresponds to the MeOH /UFO molar ratio of 6: 1. The biodiesel yield is 75% for a 3: 1 ratio, 90% for a 9: 1 ratio, and 91% for a 12: 1 ratio.

The results in Figure 4 show that the MeOH / UFO molar ratio of 6: 1 provides the best performance from biodiesel. Subsequently, an increase in this parameter implies a decrease in the yield of the reaction.

3.1.4. Effect of the weight percentage of the catalyst (KOH and NaOH) /UFO on the yield of biodiesel.

The study of the influence of the weight percentage of the KOH and NaOH catalysts was carried out at room temperature (19 ° C) for 60 min reaction time with a MeOH /UFO molar ratio of 6: 1. The weight percentages, 0.5, 1, 1.5, and 2% of catalyst (KOH and NaOH), were tested.

Figure 5 shows the change in biodiesel yield as a function of the weight percentage of the NaOH and KOH catalyst.

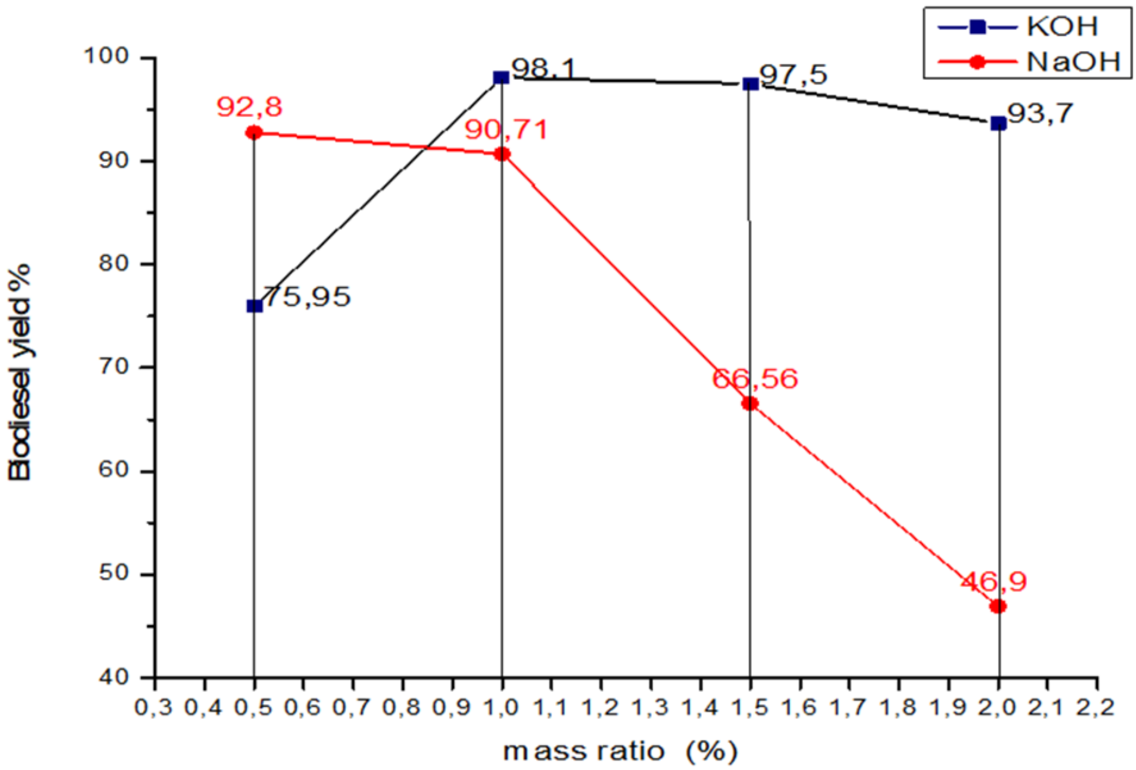

Figure 5. Evolution of biodiesel yield as a function of the percentage of KOH and NaOH catalysts (MeOH /UFO molar ratio: 6: 1, 60 min, 19 °C).

Figure 5 shows that biodiesel's maximum yield (98.1%) corresponds to 1% mass of KOH. The yield is 76% when using 0.5% by weight of catalyst and 97.5% for 1.5% and 93.7% by weight of KOH catalyst. Moreover, NaOH's maximum yield reached 92.8%, which corresponds to 1% by weight with a decrease in yield when the weight percentage increases caused an emulsion to form by the saponification reaction, which allows a loss of the product.

Figure 5 shows that the 1% weight percentage of KOH provides the best yield from biodiesel.

3.1.5. Effect of alcohol type on the yield of biodiesel.

The influence of alcohol type was investigated at room temperature (18 ° C) for 60 min reaction time with 1% wt;% of KOH and molar ratio 6: 1 with the use of methanol, ethanol, 2-propanol every time. Figure 6 shows the evolution of the biodiesel yield depending on alcohol type.

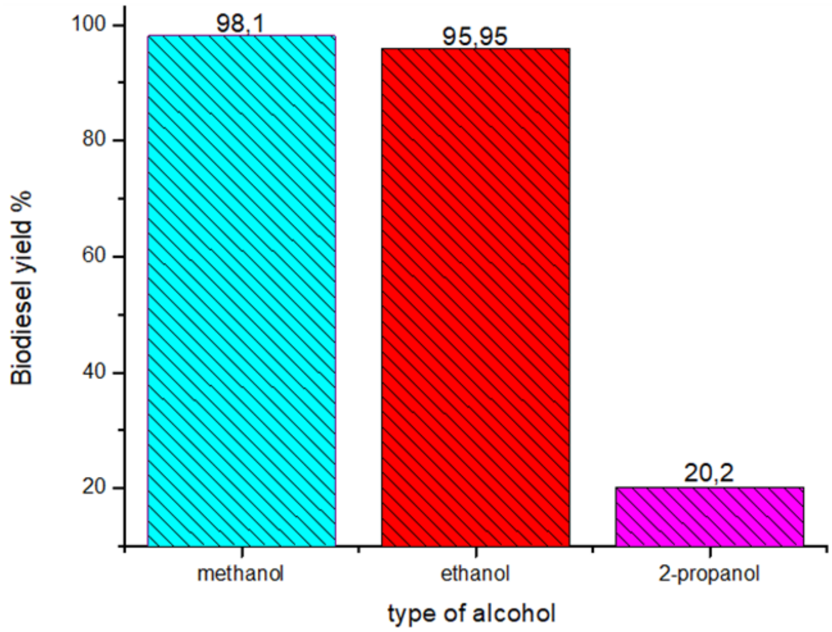

Figure 6. Evolution of biodiesel yield according to the type of alcohol (molar ratio 6: 1, 60 min, 19°C).

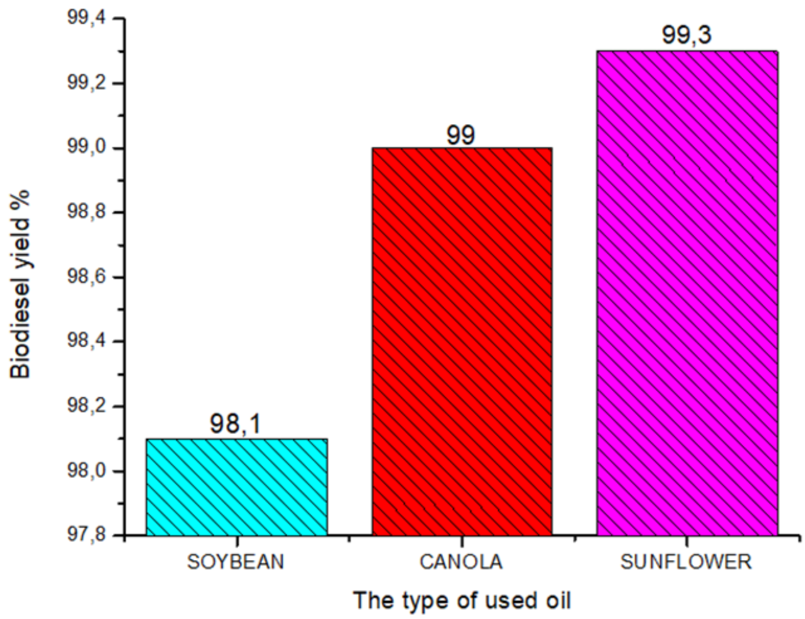

Figure 7. Evolution of the biodiesel yield according to the type of used oil (molar ratio 6: 1, 60 min, 19 °C).

Figure 6 shows that the maximum yield of biodiesel (98.1%) corresponds to methanol with a slight difference between them and ethanol, but the choice should be methanol because of its low cost.

3.1.6. Effect of the type of used oil on the yield of biodiesel.

The study of the influence of the oil type was carried out at room temperature (18 °C) for 60 min reaction time with 1 wt% by of KOH, molar ratio 6: 1 and methanol using 3 used types of oils, namely Soybean, Canola, and Sunflower each time. Figure 7 shows the evolution of biodiesel yield depending on the type of used oil.

Figure 7 shows that the maximum yield of biodiesel (99.3%) corresponds to sunflower oil with a small difference between canola and soybean, but the choice depends on the physicochemical parameters of oil, which are similar to diesel and the low cost.

The cost of sunflower and soybean is the same; on the other hand, canola's cost is 2 times greater than that of sunflower and canola.

3.2. Physicochemical parameters of new oils (NO), used frying oil (UFO), and biodiesel.

Table 1 shows the values of viscosities, densities, acid numbers, flash point and pour point of new oils (NO), used frying oil (UFO), biodiesel, and diesel used in this study are presented.

The transesterification reaction of NO and UFO was carried out under the conditions following procedures: reaction time: 60 min, ambient temperature (which makes it possible to reduce the energy loss during heating), MeOH / oil molar ratio: 6: 1, weight percentage KOH/oil: 1% with three types of used oils (canola, Sunflower, Soybean) that presents the optimal conditions to obtain a maximum conversion.

Due to their difference in the alkyl group, some physicochemical properties may be slightly different; it is noted that the ester's properties vary depending on the nature of the used oil.

Table 1. Physicochemical parameters of new oil (NO), used frying (UFO) and methyl ester (biodiesel) and diesel.

	Petro-diesel	Commercial biodiesel	Soybean			Sunflower			Canola		
			NO	**UFO**	**biodiesel**	**NO**	**UFO**	**biodiesel**	**NO**	**UFO**	**biodiesel**
Density (at 15 °C)	0.81-0.87 [48]	0.86-0.9 [51]	0.920	0.922	0.887	0.922	0.923	0.885	0.920	0.920	0.883
Viscosity (at 40 oC)(mm2/s)	1.5-5.8 [48]	3.5-5 [51]	30.84	31.61	4.60	34.27	39.97	4.32	35.04	39.75	4.31
Acidity index (mgKOH/g)	------	< 0.8 [52]	0.15	1.61	0.81	0.24	1.72	0.92	0.41	2.01	0.52
Flash point (ºC)	60 < [49]	96-190 [53]	326	320	159	308	321	186	310	314	172
Pour point (°C)	-35 -15 [50]	-15+10 [54]	-14	-13	-8	-15	-13	-11	-10	-9	-6
Color	2.5 [48]	------	0.5	2	1.5	0.5	2	1.5	0.5	2	1.5

In order to see if our synthesized diesel exhibit properties following those of international standards. Table 1 summarizes the comparative study results of biodiesel synthesized with petro-diesel and biodiesel according to international standards.

3.2.1. Viscosity.

From Table 1, it can be seen that the vegetable oil used before processing is very viscous. However, once treated by transesterification, the obtained biodiesel reveals a kinematic viscosity at 40 ° C closer to that of diesel. The viscosity decreases by of 7 to 10 order factors. This reduction is an essential advantage since it allows much better atomization by the injectors. Therefore much better combustion and a high kinematic viscosity would create problems such as deposits in the engine.

In fact, the required injection pressure will have to increase, which will lead to incomplete combustion, thus causing unburnt material that will clog on the nose of the injectors, the cylinders, and the pistons. This fact will also lead to the obstruction of the engine power supplies [55].

The viscosity of synthesized biodiesel varies from 4.31 mm^2/s to 4.6 mm^2/s; it conforms to standards and is close to diesel density.

3.2.2. The acidity index.

The acidity index of biodiesel was determined using the same operating protocol for oils. It varies according to the used oil type, but it should not go over 0.8 to 1 mg KOH/g. This can also lead to corrosion problems, noting that used oils with acidity index greater than the obtained biodiesel and new oils caused the oxidation reaction during use at high temperatures (between 160 ° C and 180 ° C), in the presence of water and oxygen, they cause the appearance of aromas and color changes in used frying oils which increases the acidity index of used oils, but when the transesterification reaction of the used oils decrease the acidity index instead, this confirms that the transesterification reaction refines the oils of these free fatty acids and also by the elimination of the glycerol which is corrosive. By comparing it with the international standard, we find that our biodiesel conforms to the standards.

3.2.3. The flashpoint.

The flashpoint of biodiesel is higher than that of diesel. This makes them less dangerous to handle than diesel.

3.2.4. The pour point.

Biodiesel has a pour point conforming to international standards close to diesel, which notably improves cold starting.

3.2.5. Density.

According to the results of Table 1, the density values are between 0.883 and 0.887. Therefore, the density of all biodiesels meets the standards, and it is close to the density of diesel. The difference recorded between the density of pure oil and waste oils may be due to the use of high temperatures (between 160 and 180 ° C) during frying. In the presence of water and oxygen, triglycerides undergo a large number of complex reactions, which can be classified into three main families: oxidation, polymerization, and hydrolysis.

It is clear that the physicochemical properties change dramatically when switching from oil to the corresponding biodiesel. The properties of diesel are then approached. Examination of Table 1 shows that our biodiesel exhibits characteristics that meet standards.

4. Conclusions

An optimization study was also carried out to optimize the transesterification reaction's conditions achieving the maximum yield of biodiesel. This study showed that the transesterification of used frying oils at room temperature (18 ° C) for 60 min of reaction in the presence of methanol with a molar ratio MeOH / UFO of 6: 1 with 1 weight percent of KOH gives an Optimal conversion of used frying oils (UFO) the yield is at 99.3% for UFO of Sunflower origin and 98.1% for UFO of Soybean origin. The obtained biodiesel has physicochemical characteristics comparable to those of commercial biodiesel and petrodiesel, according to international standards. According to these comparisons, we can say that the synthesized biodiesel has characteristics following the standards and very close to diesel in standing point of characteristics and of the released energy.

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Conflicts of Interest

The authors declare no conflict of interest.

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