Optimization, characterization and properties
of biodiesel produced from waste sunflower frying oil

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Résumé - *Le biodiesel est un carburant issu de ressources renouvelables, généralement produit par la transestérification des huiles végétales ou graisses animales. Dans cette étude, nous avons optimisé les conditions de réaction de la transestérification de l'huile de tournesol résiduelle de friture. Les esters d'alkyle d'acides gras synthétisés ont été caractérisés par la spectroscopie FT-IR et la CG-SM. A cet effet, une série d'expériences a été effectuée à 25 °C et à 50 °C avec différents types d'alcools et à des temps de réaction s'étalant de 30 à 240 minutes. Le meilleur rendement des esters d'alkyle d'acides gras a été obtenu à 25°C en utilisant le méthanol comme alcool après une heure de réaction. Les propriétés du biodiesel obtenu sous les conditions optimales notamment la viscosité cinématique, la densité, le point trouble et le point éclair ont été déterminées et comparées avec les normes ASTM D6751 du biodiesel. Les résultats montrent que le biodiesel produit est conforme aux normes et qu'il n'y a pas de différence significative entre les propriétés des EEAG et celles des EMAG.*

Abstract – *Biodiesel from renewable resources generally produced with the transesterification of vegetal oils or animal fats. In this study, we have improved the reaction conditions of the transesterification of the sunflower oil outcome in from frying. Fatty acid alkyl ester synthesized were characterized by the FT-IR spectroscopy and the CG-SM. For that, a range of experiences have been made in 25 °C and in 50 °C with different types of alcohols and in reaction times going from 30 to 240 minutes. The best result of fatty acid alkyl ester have been obtained at 25°C using methanol as an alcohol after an hour of reaction. Features of the obtained biodiesel under optimal conditions in particular kinematic viscosity, density, the cloud point and the flash point were determined and compared to ASTM D6751 biodiesel standards. The results show that the produced biodiesel lines with the standards and there is no major difference between EEAG and EMAG features.*

Keywords: Biodiesel – Characterization - Fatty acid alkyl esters - FT-IR - Fuel properties
- GC-MS - Waste Sunflower frying oil - Transesterification.

1. INTRODUCTION

For some time, the depletion of crude oil reserves was central to the debate on energy and the subject is becoming even more pressing due to the rapid industrial development of emerging countries, which weighs on the demand [1-3].

The tensions induced by the energy demand, combined with the consideration of the emissions of greenhouse, led to search alternative sources of petroleum-based fuel, including diesel and gasoline fuels [4-6]. Among the alternative possible sources, the

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biodiesel known also as fatty acid alkyl esters (FAAE) seems to be an interesting solution.

Biodiesel is a renewable fuel that can be produced from a range of organic feedstock including fresh or waste vegetable oils, animal fats, and oilseed plants. It is also non-toxic, biodegradable and so more compatible with the environment [7-9].

At this time, a problem that could hinder the commercialization of biodiesel is the cost of its production due to the cost of virgin vegetable oil [5, 10]. Therefore, it is necessary to explore ways to reduce the production costs of biodiesel. On the other hand, the agri food industries and the restaurants generate large quantities of waste frying oil, which are poured in an anarchic way and may lead to environmental and health problems if they are not properly managed [11, 12].

In this sense, methods that permit to minimize the costs of the raw material are of special interest and can be reached by the use of waste frying oil to produce biodiesel [13, 14]. Thus, the use of waste frying oils seems to be a way that allows both recycling and reducing the production cost of biodiesel.

Biodiesel is traditionally produced by transesterification reaction which is a technique of choice for its production. During this reaction, the triglycerides contained in vegetable oils, react with a short-chain alcohol such as methanol or ethanol in the presence of a catalyst to obtain fatty acid methyl esters 'FAME' or fatty acid ethyl esters 'FAEE' [15, 16] according to the reaction scheme shown in figure 1.

Glycerol is the by-product of the reaction which can be used in its traditional applications (pharmaceutical, cosmetic and food industries) or in its recently developed applications (animal feed, carbon feedstock in fermentations, polymers, surfactants, intermediates, lubricants and biodiesel production) [17].
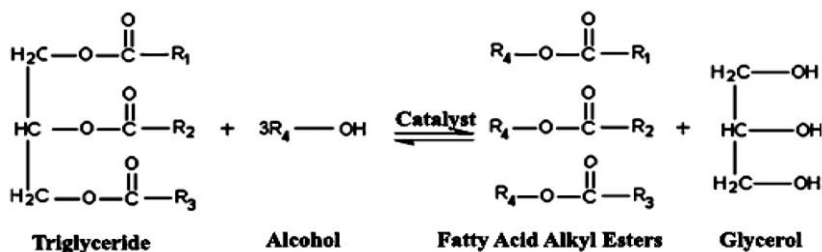

Fig. 1: Overall reaction of the transesterification of vegetable oils

Several parameters affect the transesterification reaction, such as the type and the amount of the catalyst, the alcohol to oil molar ratio, the reaction time, the reaction temperature and the amount of the free fatty acids 'FFA' [18, 19].

In general, a large excess of alcohol is used to shift the equilibrium to the right [20]. Among alcohols, methanol is the most commonly used because of its high reactivity and low price [21]. Ethanol is a good candidate to replace methanol because of its several advantages: the most important one is the fact that it can be produced from agricultural resources and is less toxic than methanol.

Also, the use of ethanol in the production of biodiesel improves the cold properties (such as cloud point, cold filter plugging point, or pour point) when compared to the use of methanol [22, 23].

Transesterification reaction can be alkali, acid or enzyme-catalyzed, the alkaline catalysts such as NaOH, KOH, and CH_3Na are the most commonly used and are preferred because they lead to high yields [24, 25]. Sodium hydroxide is widely used because of its low cost an dit can give high conversions of vegetable oils [26].

The aim of this work is the synthesis of fatty acid alkyl esters from waste sunflower frying oil and the optimization of the most important factors affecting the yield of the reaction such as reaction time, reaction temperature and the alcohol type.

The produced biodiesel produced is analyzed using Fourier Transform Infra-Red and Gas Chromatography Mass Spectroscopy and the fuel properties of the biodiesel are determined and discussed.

2. EXPERIMENTAL PART

2.1 Reagents and materials

Waste frying oil used for the experiments was sunflower oil purchased from Cevital Company, Algiers, Algeria. The waste sunflower frying oil was collected from home. Sodium hydroxides (NaOH) 99%, anhydrous methanol (MeOH) 99,7% and ethanol (EtOH) 99,8% were purchased from (Sigma-Aldrich, Germany).

The experimental setup consists of a 250 ml three-necked round bottom flask equipped with a thermometer, a dropping funnel and a condenser. The mixture in the reactor was heated and well-stirred at the same speed for all experiments by hotplate with magnetic stirrer.

2.2 Analytical methods

The free fatty acids content of waste sunflower frying oil was determined by titration according to AOCS Official Method Cd 3a-63. The waste sunflower frying oil and biodiesel densities were determined with a DMA 5000 density meter from Anton Paar, Germany according to ASTM D5002.

The kinematic viscosity for the waste sunflower frying oil and biodiesel was determined according to the ASTM D445 method. Other biodiesel properties including flash point and cloud point were obtained according to the ASTM D93 and ASTM D2500 methods.

2.2.1 GC conditions

The principal product of the reaction was analyzed by GC-MSfor the evaluation of fatty acid alkyl esters profile. GC separation was recorded with HP Model 6890 plus (Agilent technologies) gas chromatograph. A HP5-MS capillary column (30 m × 0.25 mm and film thickness 0.25 μm) was used.

The oven temperature was initially held at 100°C for 2 min, and increased to 290 °C at 4 °C per min, with a plateau of 14 minutes. The temperatures of the injector and the detector are respectively 250 °C and 300°C; Helium was used as carrier gas (flow rate 0.5 ml/min). Sample volume was 1.0 μl and the split ratio was 1/20.

2.2.2 MS conditions

Analysis by GC-MS was performed on the same chromatograph coupled to a HP MSD mass spectrometer 5973 A. The source temperature was maintained at 230 °C and that of the quadruple at 150°C. Mass spectra were acquired in the electronic impact mode (70 eV).

FAAE profile was further ascertained by comparing MS spectra of samples with those from the NIST mass spectral library of the GC-MS system while quantification was done by Agilent-Technologies data handling software (Chem. Station 6890).

2.2.3 FT-IR

FT-IR spectra were obtained by using a model Perkin-Elmer Spectrum One and the spectra were recorded using KBr discs.

2.3 Transesterification procedure

The waste sunflower frying oil must be first filtered to remove potatoes debris, and then heated above 100 °C for 15 min to evaporate the water. In the first step, a solution of sodium alkoxide was prepared from a required amount of the selected alcohol and sodium hydroxide.

This solution was introduced into a dropping funnel and immediate lyadded to the waste sunflower frying oil preheated to the desired temperature (in this study 100 g of oil was used for each of the experiments). T_0 was noted the time of beginning of the reaction.

Upon the addition of the sodium alkoxide, which should be freshly prepared to maintain its catalytic activity and prevent moisture absorption, vigorous stirring and a constant reaction temperature were maintained throughout the duration of the experiment.

When the reaction reached the preset reaction time, heating and stirring were stopped and the reaction mixture was transferred into a separating funnel. The upper layer contains fatty acid alkyl esters, residual alcohol and catalyst, whereas the lower layer contains a mixture of glycerol, excess of alcohol and catalyst.

The bottom glycerol phase was removed and the fatty acid alkyl esters layer was then washed with warm water at 50 °C repeatedly until the water became clear. Finally, the residual water was eliminated by treatment with Na_2SO_4, followed by filtration.

The experiments were carried out at two different temperatures 25 and 50 °C using methanol and ethanol at various reaction times ranging from 30 to 240 min. The catalyst type (sodium hydroxide)and its amount (1% by weight (wt.)), the alcohol/WSFO molar ratio of 6:1 and the agitation speed of 1100 rotation per minutes (rpm) were fixed as common parameters in all experiments.

3. RESULTS AND DISCUSSION

3.1 Physical properties of waste sunflower frying oil

Some properties of the WSFO are listed in **Table 1**. The amount of the free fatty acids is in the range of the limits as the FFA % obtained is less than 1 % [27].

Table 1: Some properties of waste sunflower frying oil

Properties	Unit	Waste sunflower frying oil
Density at 15°C	g/cm³	0.923
Kinematic viscosity at 40°C	mm²/s	35.84
FFA	% by weight	0.91

3.2 IR analysis

Infra-Red Fourier Transform analysis of the principal products was performed. Spectra on figures 2 and 3 show that the bands of C=O and -C-O (ester function) appear at 1739 and 1245 cm^{-1} respectively and are in agreement with literature data [28, 29].

Peaks at 1435 cm^{-1} and 1035 cm^{-1} are characteristic of the functional groups O-CH$_3$ and O-C$_2$H$_5$ of methyl and ethyl esters, it is due to the alcohol radical (in this case methanol and ethanol respectively) used in the transesterification reaction [30, 31].
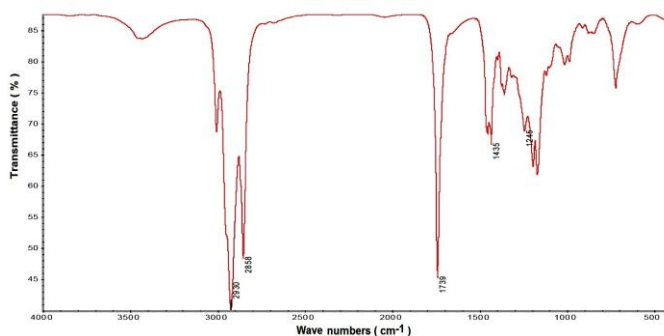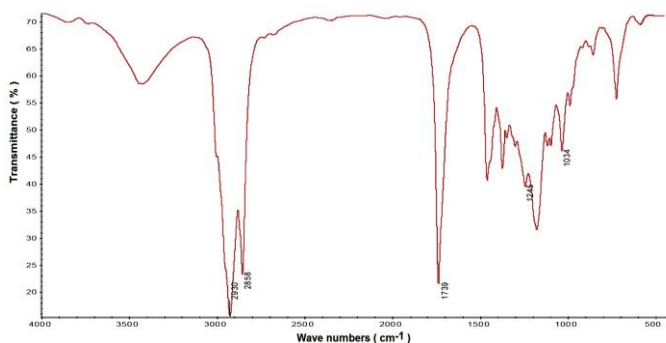
Fig. 2: FT-IR spectra of FAME

Fig. 3: FT-IR spectra of FAEE

3.3 GC-MS analysis

Analysis by mass spectroscopy (Fig. 5-8) reveals the presence of the molecular ion peaks of all compounds, as for example, the peaks at m/z 294 and m/z 284 present in the mass spectra of figures 5 and 7 which correspond to [C$_{19}$H$_{34}$O$_2$]+ and [C$_{18}$H$_{36}$O$_2$]+.

The characteristic fragments of methyl and ethyl esters which appears atm/z 276 ([M-31]+) and m/z 239 ([M-45]+) may correspond to the loss of methoxy and ethoxy groups. The peaks at m/z 74 and m/z 88 are probably due to Mac Lafferty rearrangement as shown in figure 4.

The of ions at m/z 279, 265, 251, 237, 223, 210, 197, etc. of general formula [CH$_3$OCO(CH$_2$)$_n$]+ for EMAG and m/z 269, 255, 241, 227, 213, 199, 185, etc. of general formula [CH$_3$CH$_2$OCO(CH$_2$)$_n$]+ for EEAG reveal the presence of both the linear hydrocarbon chain and the ester group.
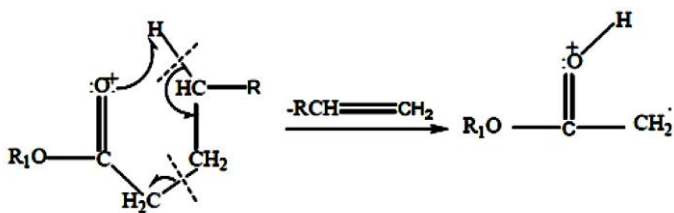
R1 = - CH₃ (m/z 74) in the case of methyl esters and
R1= (- C₂H₅) m/z 88 in the case of ethyl esters
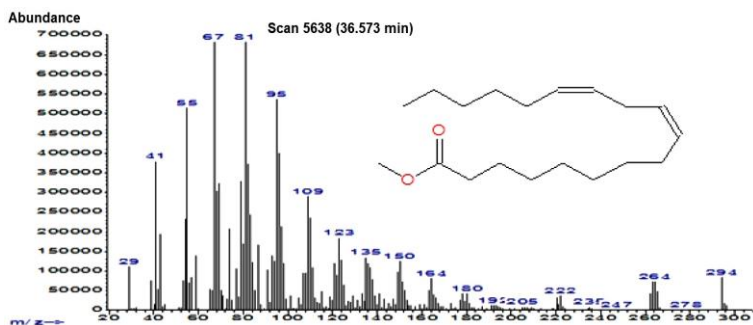
Fig. 4: Mac Lafferty rearrangement

Fig. 5: Mass spectrum of methyl octadec-9E, 12E-dienoate
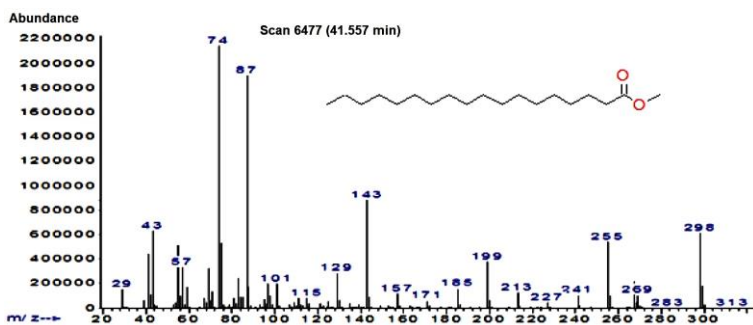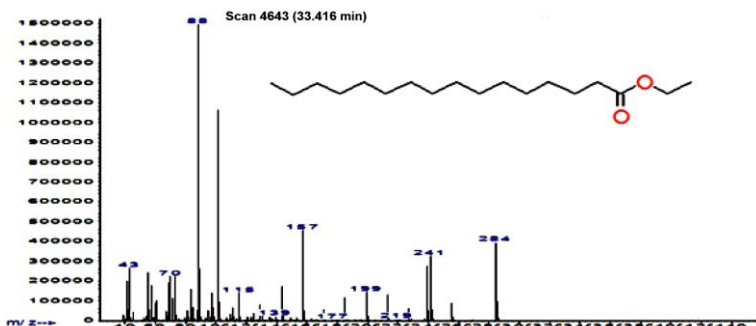

Fig. 6: Mass spectrum of methyl octa decanoate

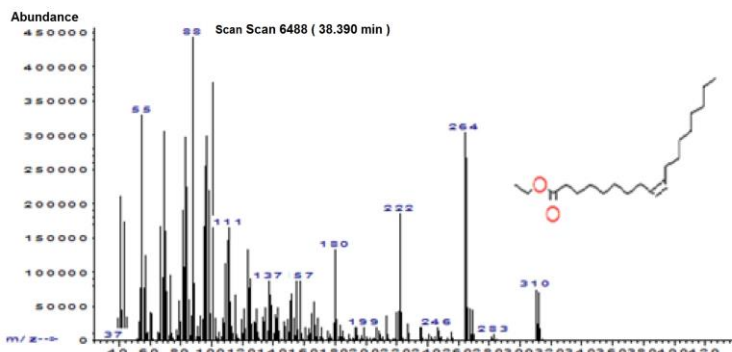Fig. 7: Mass spectrum of ethyl hexadecanoate

Fig. 8: Mass spectrum of ethyl octadec-9Z-enoate

The transesterification of WSFO was conducted to determine the optimum conditions for the production process. The effects of the main reaction parameters, such as reaction time, reaction temperature and alcohol type on FAAE yield are examined.

3.4 Effect of reaction temperature

Transesterification reaction can occur at different temperatures, depending on the oil properties [32]. It could be temperatures close to the boiling temperature of alcohol [33, 34], or ambient temperature [35] However, temperatures above boiling point of alcohol are avoided as at high temperature it accelerates the saponification of glycerides by the basic catalyst before completion of the transesterification reaction [36].

To study the influence of reaction temperature with time, some alkaline transesterification experiments using methanol and ethanol are conducted at two different temperatures 25 and 50 °C. The results represented in figures 9 and 10 show that it is better to work at 25°C.
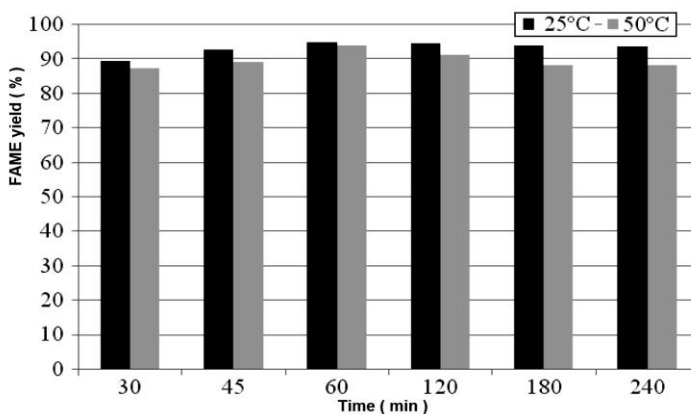

Fig. 9: Effect of reaction temperature on the yield of FAME
Reaction conditions: methanol/WSFO molar ratio 6:1,
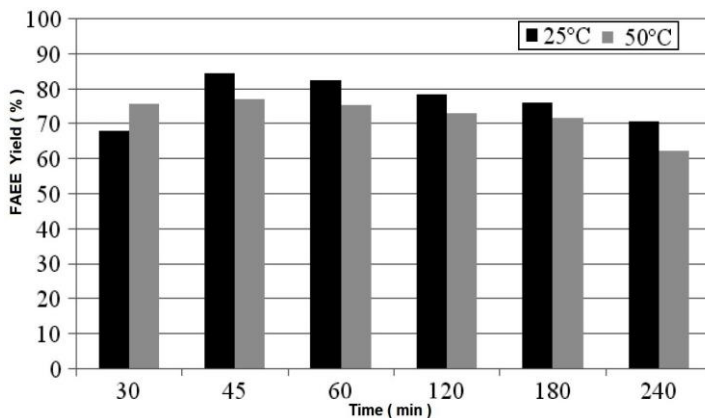NaOH amount 1 wt. % and agitation speed 1100 rpm

Fig. 10: Effect of reaction temperature on the yield FAEE
Reaction conditions: ethanol/WSFO molar ratio 6:1
NaOH amount 1 wt. % and agitation speed of 1100 rpm

3.5 Effect of reaction time

Figures 11 and 12 show the effect of reaction time on the FAAE yields. Transesterification experiments of waste sunflower frying oil are carried out at optimal temperature using methanol and ethanol between 30 min and 240 min.

Fig. 11: Effect of reaction time on the yield of FAME
Reaction conditions: Reaction temperature 25°C, NaOH amount 1 wt. %,
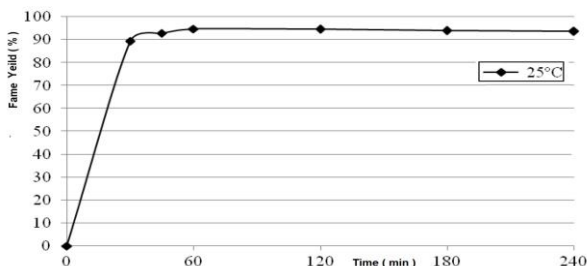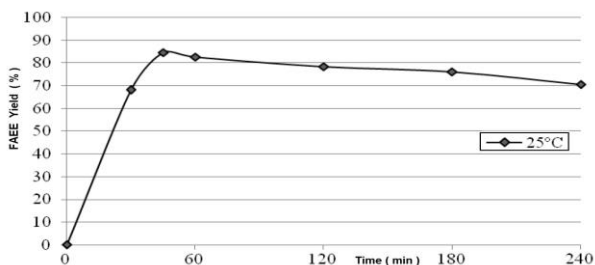methanol/WSFO molar ratio 6:1, and agitation speed of 1100 rpm

Fig. 12: Effect of reaction time on the yield of FAEE
Reaction conditions: Reaction temperature 25°C, NaOH amount 1 wt. %,
ethanol/WSFO molar ratio 6:1, and agitation speed of 1100 rpm.

The results obtained from the experiments (figures 11 and 12) reveal that fatty acid alkyl esters yields increase with reaction time at the beginning to achieve the maximum

yield at 60 min when methanol is used and 45 min when ethanol is used. Then the yield decreases slightly with increasing reaction time.

This is in agreement with literature data which show that longer reaction time will lead to a reduction in the yield due to the backward reaction of transesterification (hydrolysis), which tends to produce more fatty acids to form soap [35, 37].

As the maximum of biodiesel yields are reached at 60 min and 45 min, they have been selected as the optimal reaction times for waste sunflower frying oil transesterification using methanol and ethanol respectively.

3.6 Effect of type of alcohol

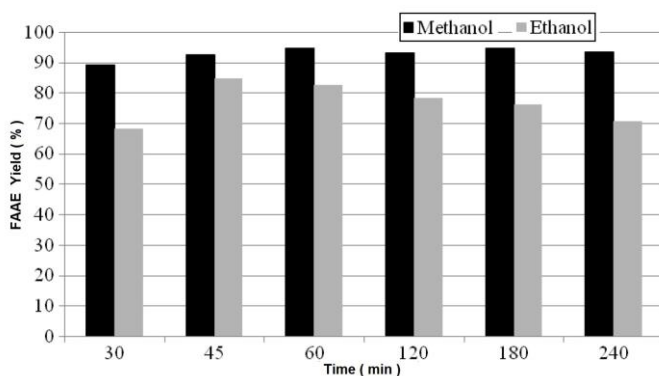Figure 13 illustrates the effect of alcohol type on FAAE versus time at optimal temperature.

Fig. 13: Effect of alcohol type on the yield of FAAE
Reaction conditions: Reaction temperature 25°C, NaOH amount 1 wt. %, alcohol/WSFO, molar ratio 6:1 and agitation speed of 1100rpm.

The obtained results show that the yields of the transesterification reaction with methanol are higher than those obtained with ethanol. These results are in agreement with the works of [38], which indicate that the production of biodiesel using ethanol is more complex than using methanol.

It should be noted that by using ethanol instead of methanol, the separation processis more difficult and takes a long time. This can be explained by the fact that excess of ethanol is contained mainly in the phase rich in glycerol, even if it is well known that it has strong affinities with the phase rich in ethyl esters, playing thus arole in stabilizing the phases which become difficult to separate[39].

The result of this affinity is the diminution of the reaction yield because the glycerol may drive some quantities of FAEE [40].

4. BIODIESEL PROPERTIES

Fuel properties of the biodiesel produced under optimum conditions including density, kinematic viscosity, flash point and poor point are studied and summarized in **Table 2**.

Table 2: Fuel properties of FAME and FAEE produced from WSFO under optimal conditions compared with the ASTM D6751 standards of biodiesel

Properties	Unit	FAME	FAEE	ASTM D6751 biodiesel
Density at 15°C	g/cm³	0.884	0.889	0.870 – 0.900
Kinematic viscosity at 40°C	mm²/s	4.62	4.79	1.9 – 6.0
Flash point	°C	> 190	> 190	> 130
Cloud point	°C	5	4	Not specified

4.1 Kinematic viscosity at 40 °C

The kinematic viscosity is an important property regarding the fuel injection in Diesel engine. For the use of biodiesel in diesel engines, the kinematic viscosity must be between1.6 and 6.0 mm²/s. The obtained results show that the kinematic viscosities of the samples obtained at the optimal conditions are respectively 4.62 mm²/s for EMAG and 4.79 mm²/s for EEAG. These values are consistent and in the range of the standard viscosity of the biodiesel.

4.2 Density at 15 °C

Density is another important property characterizing fuels and affects the performance of Diesel engines, because it affects the amount of fuel injected [41]. According to the standard of biodiesel, the value of the biodiesel density varies between 0.860 and 0.900 g/cm³. In our case, for the biodiesel obtained from waste sunflower frying oil, the values are in the range of the biodiesel standards (**Table 2**).

4.3 Flash point

Biodiesel standards specify that flash point should be higher than 130°C. According toour results, both of methyl and ethyl esters values respect this standard. The obtained results represent a benefit which reduces the risk of auto-ignition during storage and transport at high temperatures [42, 43].

4.4 Cloud point

Biodiesel standards have not set precise values of cloud point due to climatic variations depending on the location of each country [22]. The cloud point of biodiesel (EMAG and EAAG) is relatively higher than those of other works [36, 44], and so indicates that the biodiesel is less suitable in cold conditions.

6. CONCLUSION

In this study, alkaline catalyzed transesterification of waste sunflower frying oil is investigated, and the physical properties show that this oil is efficient for biodiesel production. The optimization results show that reaction time of one hour, a temperature of 25 °C and methanol are the optimal conditions for the transesterification of waste sunflower frying oil and give the highest yield of biodiesel.

Furthermore, the analysis of the fatty acid alkyl esters by IR and GC/MS show that the spectra obtained are very clear and show no peak and no parasite band. This illustrates the fact that the process of production and purification of FAAE was realized successfully: the use of biofuel seems to be possible as the biggest problem is to avoid the deterioration of engines of different gears by impurities which may remain after the synthesis and purification of Biodiesel.

The results obtained confirm the structure of the produced biodiesel and that there are no significant differences between the properties of FAME and those of FAEE. The density, kinematic viscosity and flash point values obtained are in agreement with biodiesel standards. The cloud point is slightly higher, but for a country which is situated in North Africa such as Algeria the value is suitable because of the hot climate.

Globally, the properties of biodiesel produced are well compared to those of the biodiesel standards, and it can be concluded that the transesterification of waste frying oil is a promising way for producing cheap alternative fuels (biodiesel), which could reduce pollution and protect the environment.

NOMENCLATURE

WSFO	Waste Sunflower Frying Oil
ASTM	the American Society for Testing and Material
FAAE	Fatty Acid Alkyl Esters
FAEE	Fatty Acid Ethyl Esters
FAME	Fatty Acid Methyl Esters
FFA	Free Fatty Acids (%)
FT-IR	Fourier Transform Ifra Red
GC-MS	Gas Chromatography-Mass Spectrometry
RPM	Rotation per Minute
Wt	Weight (g)

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