Chapter 9 Biofuel Production from Sunflower Oil and Determination of Fuel Properties

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Abstract The depletion of crude oil is central to the debate on energy, and the subject becomes even more pressing because of the rapid industrial development of emerging countries that hangs over demand. Growing emissions of combustion generated pollutants, and their increasing costs will make biomass sources more attractive. Currently, biodiesel is becoming popular as a more environment-friendly fuel, because it is a renewable, domestic resource with an environment-friendly emission profile, readily biodegradable and nontoxic.

The objective of our work is to produce biodiesel from a renewable and sustainable energy resource which is sunflower oil through transesterification process using alkaline catalyst and methanol, to optimize some parameters in the aim to obtain the best reaction yield, and to study some biodiesel's properties such as kinematic viscosity, density, cloud, and flash points; the biodiesel was analyzed by infrared spectroscopy and gas chromatography–mass spectrometry. The results of the analysis confirm that the synthesized biodiesel is a mixture of fatty acid methyl esters, a comparative study of biodiesel has been conducted versus standard biodiesel ASTM D6751, and the results obtained show good properties when compared to those of biodiesel's standard.

Keywords Biodiesel • FT-IR • Fuel properties • GC-MS • Transesterification • Sunflower oil

Nomenclature

 T Temperature, $^{\circ}C$

Wt. Weight, g

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Subscripts

9.1 Introduction

For some time, the depletion of crude oil reserves is 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 [[4–7\]](#page-6-0). Among the alternative possible sources, the 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 nontoxic and biodegradable, more compatible with the environment $[8-10]$.

Biodiesel is traditionally produced by transesterification 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) [\[11](#page-6-0), [12](#page-6-0)] according to the reaction scheme shown in Fig. 9.1.

Several parameters affect the transesterification reaction, such as the type and the amount of the catalyst, the alcohol to oil ratio, the reaction's time, the reaction's temperature, and the amount of the free fatty acids [[13,](#page-6-0) [14\]](#page-6-0).

The aim of this work is the synthesis of fatty acid methyl esters from sunflower oil (SFO). The optimization of the most important factors affecting the yield of the reaction such as time and temperature of the reaction is established. The biodiesel

Fig. 9.1 Overall reaction of the transesterification of vegetable oils [\[11\]](#page-6-0)

produced is analyzed using Fourier transform infrared and gas chromatography– mass spectroscopy, and the fuel properties of the biodiesel have been determined and discussed.

9.2 Experimental Part

In the first step, a solution of sodium methoxide was prepared from a required amount of methanol and sodium hydroxide. This solution was introduced into a dropping funnel and immediately added to the sunflower oil preheated to the desired temperature; vigorous stirring and a constant temperature were maintained throughout the duration of the experiment. When the reaction reached the preset reaction time, the reaction mixture was transferred into a separating funnel. The upper layer contains fatty acid methyl 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 methyl esters layer was then purified.

The study was carried out at two different temperatures using methanol at various reaction times ranging from 30 to 360 min. The other factors such as molar ration and type and amount of catalyst were fixed as common parameters in all experiments.

9.3 Results and Discussion

9.3.1 Effect of Reaction Temperature

The experiments were carried out at two temperatures such as 25 and 50 \degree C with 1 wt% of NaOH, methanol/SFO molar ratio 6:1, and agitation speed 1,100 rpm. The FAME yield versus time at the different temperature is shown in Fig. [9.2.](#page-3-0) It was observed that it is better to work at 25° C than at 50° C.

9.3.2 Effect of Reaction Time

Figure [9.3](#page-3-0) shows the effect of reaction time on the FAME yields. Transesterification experiments of sunflower oil are carried out at optimal temperature (25 $^{\circ}$ C) between 30 and 360 min.

Results obtained from the experiments reveal that fatty acid methyl esters yield increase with reaction time at the beginning to achieve the maximum yield at 60 min. Then the yield decreases slightly with increasing reaction time. This is in

Fig. 9.2 Effect of reaction temperature on the yield of FAME

Fig. 9.3 Effect of time reaction on the yield of FAME

agreement with literature data, which shows that longer reaction time will lead to a reduction in the yield product due to the backward reaction of transesterification (hydrolysis), which tends to produce more fatty acids to form soap [\[15](#page-6-0)]. Due to reaching the maximum biodiesel yield at 60 min, it has been selected as the optimal reaction time for sunflower oil transesterification using methanol.

Fig. 9.4 FT-IR spectrum of FAME

9.3.3 IR Analysis

Infrared Fourier transform analysis of the principal product was performed. Spectrum in Fig. 9.4 shows that the bands of $C=O$ and $-C-O$ (ester function) appear at 1,743 cm⁻¹ and 1,245 cm⁻¹, respectively, and that of O–CH₃ appears at $1,437$ cm⁻¹; those results are in agreement with literature [\[16](#page-6-0), [17\]](#page-6-0).

9.3.4 GC-MS Analysis

Analysis by mass spectroscopy reveals the presence of the molecular ion peaks of all compounds. For example, the peak at m/z 270 present in the mass spectrum (Fig. [9.5\)](#page-5-0) which correspond to $[C_{17}H_{34}O_2]^+$, $[C_{16}H_{31}O]^+$ fragment appears at m/z
239 ([M.31]⁺) and represents the loss of methoxy group. The peak at m/z 74 is 239 ($[M-31]^+$) and represents the loss of methoxy group. The peak at m/z 74 is
probably due to Mac I afferty rearrangement probably due to Mac Lafferty rearrangement.

9.4 Biodiesel Properties

Fuel properties of the biodiesel produced under optimum conditions including density, kinematic viscosity, flash point, and cloud point are studied and compared with the ASTM D6751 standards of biodiesel (Table [9.1\)](#page-5-0) [\[18](#page-6-0)].

Fig. 9.5 Mass spectrum of methyl hexadecanoate

Table 9.1 Fuel properties of FAME produced from SFO under optimal conditions compared with the ASTM D6751 standards of biodiesel

Property	Unit	FAME	ASTM D6751 biodiesel
Density at 15° C	g/cm^3	0.886	$0.870 - 0.900$
Kinematic viscosity at 40 \degree C	mm^2/s	4.51	$1.9 - 6.0$
Flash point	\circ \sim	>190	>130
Cloud point	$^{\circ}C$		Not specified

The obtained results show that the kinematic viscosities and the density of the samples obtained at the optimal conditions are consistent and in the range of the standard of the biodiesel. We note also that the value of the flash point is higher than 190 \degree C; this result represents a benefit that reduces the risk of autoignition during storage and transport at high temperatures [[19\]](#page-6-0).

9.5 Conclusion

The results of the present study demonstrated that 1 h of reaction and a temperature of 25 C (which can be considered as an ambient temperature and therefore does not require energy expenditure) present the optimum conditions for the production of biodiesel from sunflower oil and give a high yield (95.28 %). The results of analysis (IR and GC-MS) confirm that the structure of the produced biodiesel and the fuel properties are in good agreement with the standards of biodiesel. The transesterification of vegetable oil is an alternative for producing biodiesel, which could reduce pollution and protect the environment.

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