

Biodiesel from Rapeseed Oil of Turkish Origin as an Alternative Fuel

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ABSTRACT

The crude rapeseed oil was transesterified using methanol and using sodium hydroxide as a catalyst, and the varieties affecting the monoester yield were investigated. The methyl ester fuel called Biodiesel, produced under the determined optimum reaction conditions, was tested according to the standard methods for its fuel properties. Biodiesel fuel properties were found to be very close to those of Grade No. 2-D diesel fuel.

Index Entries: Rapeseed oil; *Brassica napus* L.; transesterification; methyl ester; alternative diesel fuel; biodiesel; biofuel; CI engine fuel.

INTRODUCTION

Vegetable oils are the most important biomass origin for alternative motor fuels. The use of vegetable oils as source of energy has long been known. History records that the first studies concerning the use of vegetable oil started in the year 1928 and that R. Diesel, the inventor of the engine that bears his name, used vegetable oils as a fuel in his engines (1,2). For many years, the ready availability of inexpensive petroleum middle distillate fuels provided little incentive for experimenting with alternative, renewable fuels for diesel engine. The energy crisis of the 1970s sparked a renewed interest in the use of vegetable oils as fuels and today the subject continues to attract attention because of the successful results obtained in the applications (3–13).

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Short-term engine tests indicate good potential for the whole of vegetable oil fuels. Long-term endurance tests show that there are serious problems in injector coking, ring sticking, gum formation, and lubricating oil thickening and gelation. These problems are related to the high viscosity and nonvolatility of the oils, which result in inadequate fuel atomization and incomplete combustion (14). Reduction in viscosity greatly reduces engine operation problems. The most prevalent solution to the problem of viscosity is to use the transesterification (also called alcoholysis) method. Transesterification is a kind of process that occurs when a triglyceride, such as a vegetable oil, reacts with an monohydric alcohol (methanol, ethanol, and so on) in the presence of a catalyst (acidic or alkali catalyst) to produce glycerol and fatty acid esters. This ester mixture is referred to as Diesel-Bi, Biodiesel, Di-Ester, Green Ester. In the production of Biodiesel, rapeseed, soybean, and sunflower are the oils most preferred, but monohydric alcohol methanol is preferred to ethanol. The rate of the reaction is dependent on the composition of the vegetable oil, temperature, duration, and especially on the oil-alcohol molar ratio and the amount of catalyst. The literature provides a considerable amount of work about the refinement of the ester product, the necessary conditions for storage and transportation, fuel properties, motor tests, environmental effects, and economical analysis (1–43). A general evaluation of the studies concerning the use of Biodiesel shows that it can be used in diesel engines without modifications, it has properties equivalent or superior to diesel fuel, it is an environmentally friendly product, and it is the best diesel fuel alternative with proven applicability. In Turkey, studies concerning the use of Biodiesel have been carried out and some of them are still continuing. However, the industrial application of these laboratory-scale studies are still not performed (44–49). Since Turkey's economy is dependent mainly on agriculture and the country provides more than 90% of its crude oil demand through imports, the evaluation of biomass-originated alternative engine fuels is of great importance. In Turkey, a wide variety of oilseeds can be cultivated and processed. Sunflower, olive, corn, cotton, and soybean are major oil sources. Nowadays, these major oil crops, (0)- and (00)-kind rape cultivation are also encouraged, especially in Thrace, and the Aegean and Mediterranean regions. An estimated increase of 73% in the country's oil crop production (including rapeseed) is expected in the near future with the completion of a major irrigation project in the Southeast Anatolia region. Actually, the annual production of rapeseed is about 1500 tons and the seeds are sold at the same price as that of sunflower. This study is an investigation and evaluation of the possibilities as a diesel fuel alternative of the oil extracted from rapeseed cultivated in Turkey within the scope of the Thrace Leader Farmer project. The experimental work covers the following steps: characterization of the rapeseeds and of rapeseed oil; determination of the optimum conditions for the transesterification reaction (rapeseed oil-methanol, catalyst: NaOH); and determination of the fuel properties of Biodiesel.

Table 1
The Seed Characteristics of Rape Seed

Characteristics	Rape seed
Seed color	Black
Seed shape	Spherical
Average diameter, 10^{-3}m	2.20
Average weight, 10^{-6}m	3.82
Moisture content, wt %	6.61
Oil content, dry basis, wt %	41.08
Protein content of the meal, wt %	29.64

MATERIALS AND METHODS

(00)-kind rapeseed (*Brassica napus* L. Oleifare, winter rapeseed), cultivated during the 1992–1993 season in the Thrace region (Tekirdağ) of Turkey, was used in the experimental study. For the characterization of the rapeseeds, the dimensions, weight, moisture, and oil content of the seeds and the protein content of the meal have been determined. The results are shown in Table 1. The rape oil was obtained from finely ground seeds by mechanical cold-press extraction method.

The grinding process was done in a blender labeled Braun, the cold-press extraction was carried out with a laboratory-type Carver hydraulic press (Fred S. Carver, Wabash, IN) at room temperature under the pressure $69 \times 10^{-6} \text{ Nm}^{-2}$. The technological characteristics of the crude rapeseed oil were determined according to the standard methods of oil and fat analysis (50–52). Composition of the rapeseed oil was investigated by TLC/FID Iatroscan TH-10 MK IV analyser with SIII rods (Iatron Lab., Tokyo). The working conditions were chosen following the results obtained in the analyses of standard mixtures (27,53,54):

Developing system:

Sample volume	1 μL
Sample composition	0.1-g Sample/10 mL chloroform
Developing solvent mixture	Petroleum ether:Diethyl ether:Acetic acid (70:30:2)
Developing time	20 min
Dryer system	5 min in air temperature, 5 min in air oven at 110°C

Flame ionization detector:

Air flow rate	160 mL/min
Hydrogen flow rate	2000 mL/min
Scan speed	30 s/scan
Recorder	Iatrocorder TC-11
Calculation method	Absolute calculation

Table 2
The Technological Characteristics of Crude Rape Seed Oil

Characteristics	Crude rape seed oil
Density, 15°C, kg/m ³	909.5
Refractive index, 20°C	1.4726
Acid value, mg KOH/g	2.7
Saponification value, mg KOH/g	185.78
Iodine value (Kaufman method, gL/100 g)	98.80
Fat composition wt %	
Triglycerides	96.95
Free fatty acids	1.35
Monoglycerides	1.70
Calculated mean molecular weight of the oil, kg/mol	858
Fatty acid composition, wt %	
Palmitic	4.44
Stearic	2.72
Oleic	74.15
Linoleic	13.23
Calculated mean molecular weight of the fatty acids, kg/mol	279

Fatty acid composition of the rapeseed oil was subjected to capillary gas chromatographic analysis with the apparatus of Hewlett Packard 5890 series II fitted with a flame ionization detector (FID). Fatty acid methyl esters were prepared for chromatographic analysis (GLC) using BF₃-methanol reagent (55). The working conditions were as follows:

Column	Ultra 2 (25 m × 0.32 mm × 0.52 μ film thickness of 5% diphenyl, 95% dimethyl polysiloxane)
Gases	
N ₂ , carrier gas	2 mL/min
Air	374 mL/min
H ₂	27 mL/min
Injection temperature	200°C
Flame ionization detector (FID) temperature	250°C
Oven temperature program	30°C (5 min), 30–170°C (5°C/min), 170°C (5 min), 0–200°C (3°C/min), 200°C (30 min)
Detection response factor	1.0

The technological characteristics of the crude rapeseed oil are shown in Table 2.

Transesterification Reaction Procedures and Refinement of the Esterification Product

Transesterification reactions of the crude rapeseed oil were carried out using methanol as the alcohol and sodium hydroxide as the catalyst. Methanol and sodium hydroxide were obtained from Merck. A 250 mL three-necked flask equipped with condenser, thermometer, and contact thermometer was used for reactions. The neck for the thermometer was used for taking samples from the reaction mixtures for chromatographic analysis at certain time intervals. Reaction mixtures were heated on a magnetic heater and stirrer to the boiling point of methanol, and catalyst was added to the mixture at that moment. Reaction temperatures were controlled within a range of $\pm 1^\circ\text{C}$. Conversion of the crude rapeseed oil to its monoester and other reaction products was analyzed with Iatroscan TH-10 MK IV (TLC/FID). The working conditions are given in the previous section. One-milliliter samples for TLC/FID analysis were taken from the reaction mixtures at certain time intervals and then they were quenched in test tubes that included 1 mL distilled water and all the test tubes were settled down in a vessel filled with ice to stop the reaction. The method of washing with petroleum ether and distilled water, which was used in our previous studies and by other researchers, was performed in the refinement step (20,27,44–48). In the refinement step of the transesterification reaction, the mixtures were first allowed to cool to room temperature, after which the ester and glycerol layers were separated in a separator funnel. Excess methanol in the ester phase was removed in a rotary evaporator under reduced pressure. The ester phase was then taken again to the separator funnel. After the addition of petroleum ether (equal to the ester phase volume, bp 40–60°C) and distilled water (at 20°C and equal to twice the ester phase volume), the pH of the solution was adjusted to 7.0 by adding acetic acid. Following the washing process repeated three times with distilled water, the ester phase was dried over anhydrous sodium sulfate (25% by the weight of the ester phase volume), and the petroleum ether was removed in the rotary evaporator under vacuum, hence revealing the refined methyl ester product (Biodiesel) with a yield of 99.01%. The refinement agents were obtained from Merck.

Biodiesel, produced under the determined optimum conditions of transesterification reaction, was tested for its diesel fuel properties according to the ASTM standard test methods and analytical methods.

RESULTS AND DISCUSSIONS

For the transesterification reaction, the results of the study of Freedman et al. and of our previous work were taken into account and the optimum conditions were determined (19,48). In the case of alkali catalyst usage in transesterification reaction, the maximum amount of free fatty acids is

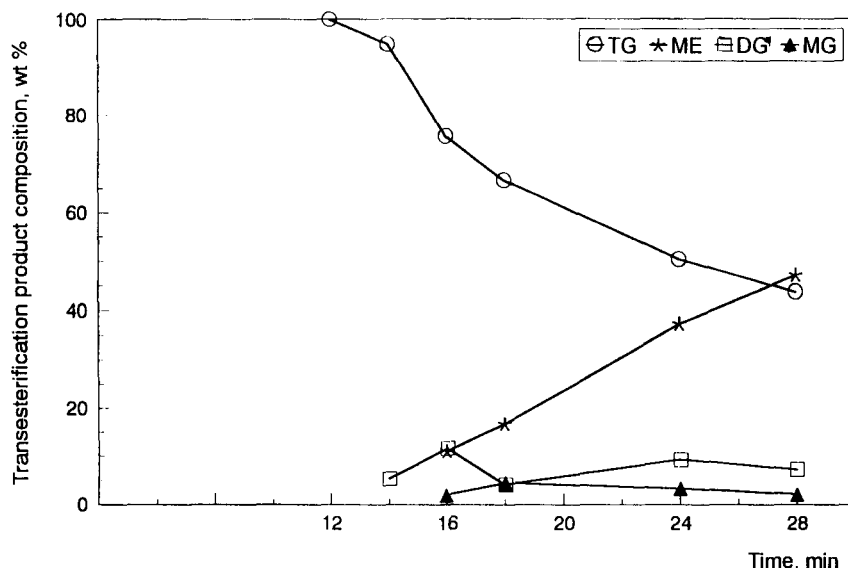


Fig. 1. Change in transesterification product composition with time (Oil/alcohol molar ratio, 1:6; temperature, $65 \pm 1^\circ\text{C}$; catalyst, 1.2% NaOH by the weight of the oil). TG, triglyceride; DG, diglyceride; MG, monoglyceride; ME, methyl rapeseed esters.

proposed to be 0.5% wt (acid value < 1) in order to obtain high methyl ester yield. For higher acid values, the activation effect of the catalyst is diminished because of the saponification. As can be seen from Table 2, the free fatty acid content of the rapeseed oil was found to be 1.35%, which is slightly higher than the proposed value. This fact was considered in the determination of the alkali catalyst amount and studies having long duration by the use of acidic catalyst were not preferred. Considering these findings, the beginning reaction conditions were chosen as follows: oil/alcohol molar ratio, 1:6; temperature, $65 \pm 1^\circ\text{C}$; catalyst, 1.2% NaOH by the weight of oil; time, 30 min. Figure 1 shows the variation in the product composition under these conditions as a function of reaction time. The conversion did not take place in the first 14 min; it began at 16 min. After 28 min, a methyl ester percentage of 47.09% was present in the reaction product mixture. These results have been taken into consideration when determining the conditions that will favor higher ester yields. The variables affecting the methyl ester yield of transesterification reaction, such as molar ratio of crude rapeseed oil to methanol, amount of sodium hydroxide as catalyst, and reaction temperature, were investigated.

Effect of the Molar Ratio

Among the variables affecting the ester yield in the transesterification reaction, the most important one is the molar ratio of the reactants. Since the reaction is reversible, an increase in the amount of one of the reactants will result in higher ester yields. In the experiments done to investigate

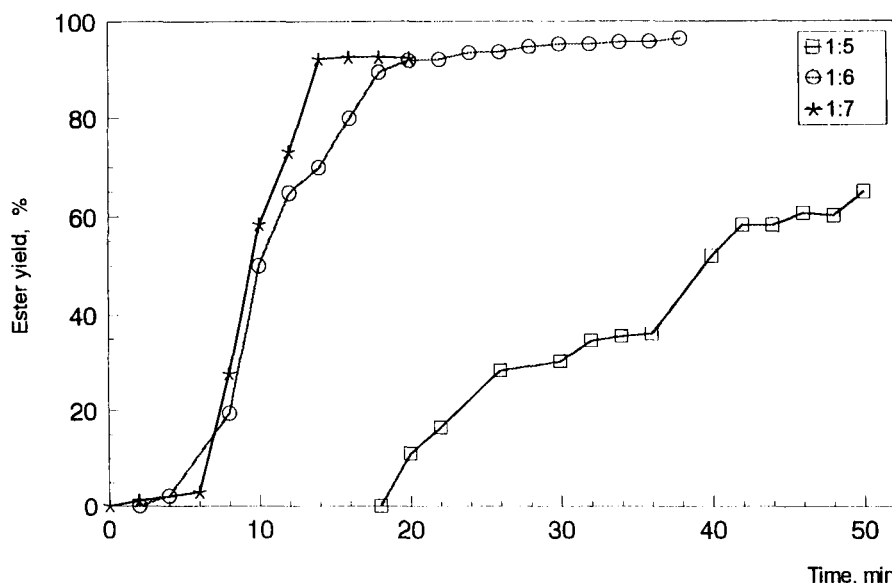


Fig. 2. Effect of the Oil/alcohol molar ratio on ester yield. (temperature, $65 \pm 1^\circ\text{C}$; catalyst, 1.7% NaOH by the weight of the oil).

this effect, the reaction conditions were chosen as follows: temperature, $65 \pm 1^\circ\text{C}$; catalyst, 1.7% of NaOH by the weight of the oil; time, 38 min. The variation in ester yields that were obtained by reactions conducted at 1:5, 1:6, and 1:7 oil/alcohol molar ratios can be seen in Fig. 2. As expected, a higher yield of ester was obtained in a shorter period of time for 1:7 molar ratio compared to 1:6. While an ester yield of 58.38% was observed in the reaction with 1:7 oil/alcohol molar ratio in the first 10 min, this value was 50.03% for 1:6 molar ratio in the same period. After 20 min, these values were respectively 92.17 and 91.78%. The yield obtained in the reaction with the molar ratio of 1:7 could not have been surpassed and in the reaction product mixture more partial glycerides were found to be left compared with the reaction of 1:6 molar ratio. For the reaction of 1:6 molar ratio, an ester yield of 96.36% was reached after 38 min. Despite the long duration and the high amount of energy consumption, when the product compositions and ester yields were compared in the light of the reasons indicated in the determination of catalyst ratios, it was concluded that 1:6 molar ratio was more suitable than 1:7.

Effect of the Amount of Catalyst

In experiments in which the effect of the variation in the catalyst amount was investigated, the reaction conditions were as follows: oil/alcohol molar ratio, 1:6; temperature, $65 \pm 1^\circ\text{C}$; catalyst, 1.7 and 2.0% of NaOH by the weight of the oil. The results obtained in these reactions are shown with the results of 1.2% NaOH in Fig. 3. This figure shows the

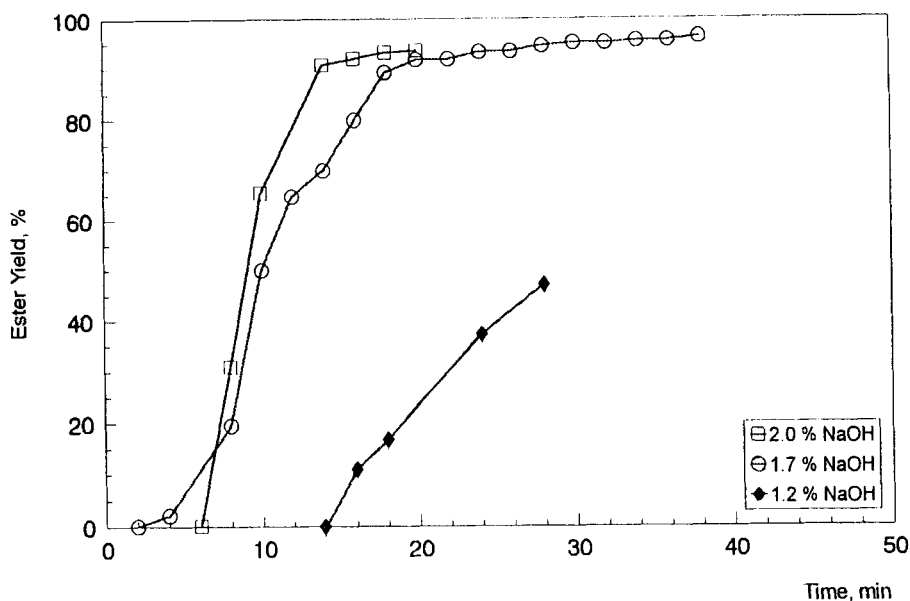


Fig. 3. Effect of the catalyst amount on ester yield (temperature: $65 \pm 1^\circ\text{C}$).

variation in ester yield (%) with respect to time. As the amount of the catalyst was increased from 1.2 to 2.0%, the yield increase occurred in a shorter period of time, and a comparison of these results with those of 1.7 and 2% NaOH shows that a higher ester yield was obtained in a longer period of time. Thus, less partial glycerides were left in the product mixture. This is an advantage prior to the refinement of the methyl ester. Since the aim of the study was to produce an ester fuel in higher yields with optimum conditions and as a result of the evaluation of this section, the amount of the catalyst suitable for the transesterification reaction was chosen to be 1.7% by the weight of the oil.

Effect of the Temperature

Although transesterification with an alkali catalyst is normally conducted near the boiling point of the alcohol, it is known that reactions can also be carried out at room temperature. The reactions were carried out with the aim of observing the effects of temperature decrease on the ester yield and the temperatures chosen were less than 65°C , namely 50°C and 40°C , aiming a reduction in the energy necessity. A comparison between the ester yields obtained at these temperatures and the yield obtained at 65°C is given in Fig. 4. In the experiment conducted at 50°C , the conversion began at 20 min and after 56 min an ester yield of 62.94% was obtained. The ester conversion in the reaction conducted at 40°C began at 40 min and at 80 min, the yield reached value of 62.86%. As shown in Fig. 5, the temperature increase influenced the reaction in a positive manner, and in the transesterification reaction carried out at 65°C a higher yield

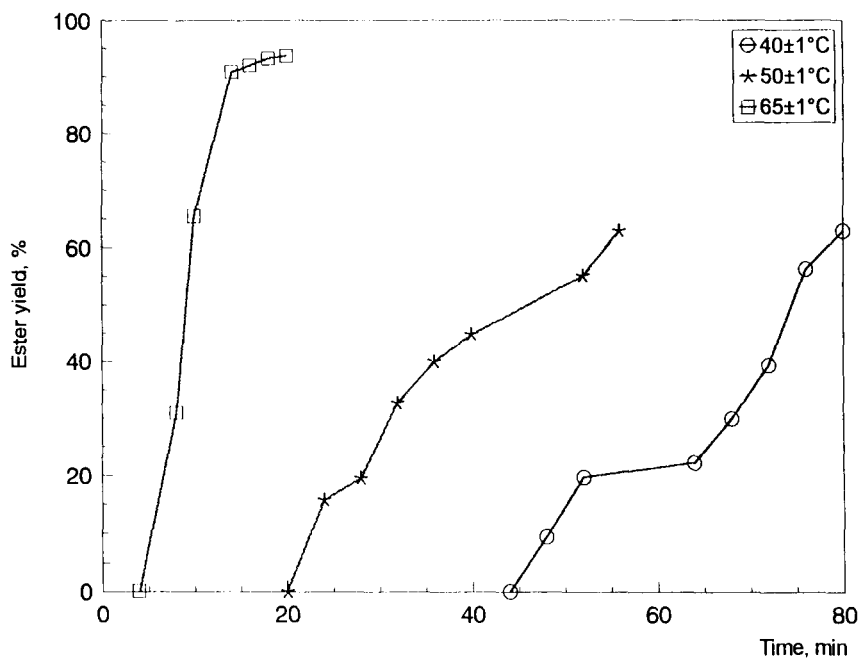


Fig. 4. Effect of the temperature on ester yield (Oil/alcohol molar ratio, 1:6, catalyst, 1.7% NaOH by the weight of the oil).

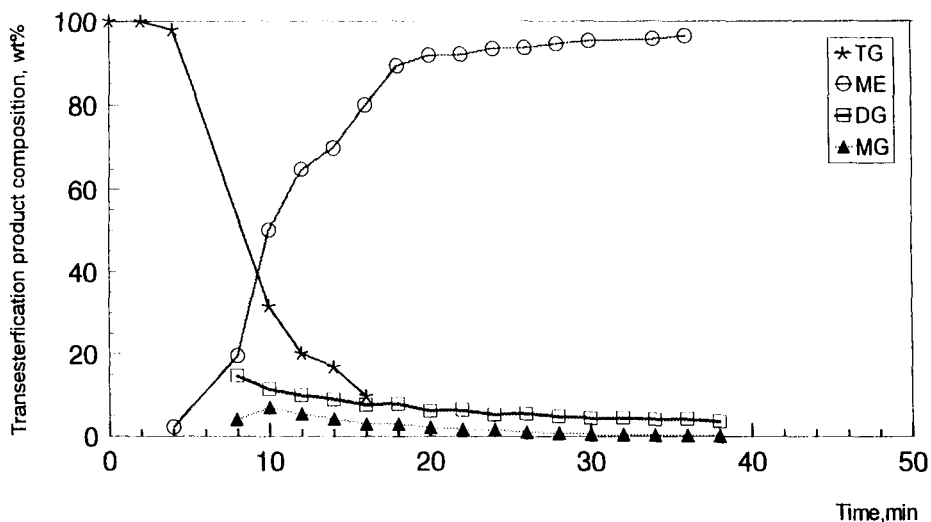


Fig. 5. Change in transesterification product composition with time. (Oil/alcohol molar ratio, 1:6; temperature, $65 \pm 1^\circ\text{C}$; catalyst, 1.2% NaOH by the weight of the oil). TG, triglyceride; DG, diglyceride; MG, monoglyceride; ME, methyl rapeseed esters.

value was obtained. The reaction duration and temperature were chosen respectively 38 min and $65 \pm 1^\circ\text{C}$ after the observation of the comparison made between low temperature-long period of time and high temperature-short period of time.

After all the experiments the optimum reaction conditions were chosen as follows: oil/alcohol molar ratio: 1:6; temperature: $65 \pm 1^\circ\text{C}$; duration: 38 min; catalyst: 1.7% of NaOH by the weight of the oil. Under these conditions, the variation with time of the reaction product composition is given in Fig. 5. As can be seen from these curves, reaction began at 4 min, and at this moment a methyl ester conversion of 2.16% was observed. The diglyceride and monoglycerides that began to appear at 8 min showed a regular decrease during the reaction. At 18 min the triglyceride (TG) disappeared and methyl ester (ME) conversion reached 79.89%. The diglycerides (DG) and monoglycerides (MG) were converted to methyl ester and the methyl ester conversion reached 96.36% at 38 min. Between 28 and 38 min the amount of methyl esters varied between 94 and 97% and the DG amount decreased to 3.54% while MG decreased to 0.1%.

Fuel Properties of the Biodiesel

The fuel properties of the Biodiesel and reference Grade No.2-D diesel fuel are given in Table 3. In this table, the standards formed in Austria (ONORM C1190 Austrian Official Standard for Rapeseed Oil Methyl Ester), where the applications of Biodiesel were prevalent and popular, are also given (56). As can be seen, Biodiesel contains an ME value of 99.91%. This value is the most important measure for the production quality of ME. The absence of partial glycerides and the very low amount of the free fatty acids are positive factors for fuel composition. None of the methanol, petroleum ether, glycerin, or sodium hydroxide were observed in the fuel. But the high flash point tells us that methanol and petroleum ether do not exist or they are present in minor quantities. In the refinement step, it was thought that the amount of the glycerin and NaOH were reduced to a negligible level after continuous washings. Considering those evaluations, the Biodiesel composition was found to be of a very high quality, according to the European minimum standards (12,56). Table 3 shows us that Biodiesel properties are in good accordance with diesel fuel and ONORM C1190 and are also suitable according to ASTM D 975-90 (the Standard Specification for Diesel Fuel Oils). The positive properties of Biodiesel compared to diesel fuel are the high flash point, the increase in cetane index, and the low sulfur content. The high flash point is an advantage that ensures security during storage, transportation, and usage. Since Biodiesel contains practically no sulfur, its use will significantly reduce pollution originating from SO_2 . The high cetane index value is another useful CI motor fuel property.

Table 3
Fuel Properties of Biodiesel and Grade No. 2-D Diesel Fuel and Austrian Standard Values of Biodiesel

Properties	Fuels			Grade No. 2-D diesel fuel (48)	Austrian Standard ONORM C1190 (Austrian official standard for rape seed oil methyl ester)
	Method	Biodiesel	Method		
Density, 15°C, kg/m ³	ASTM D 1298	875	DIN 51 757	816	860-900
Refractive index, 15°C	ASTM D 1218	1.4457		1.4814	
Composition, wt %	TLC/FID				
Methyl esters		99.91			
Free fatty acids		0.09			
Composition of methyl esters, wt%	GLC				
Methyl palmitat		4.83			
Methyl stearat	1.83	1.83			
Methyl oleat		75.65			
Methyl linoleat		17.69			

(continued)

Table 3 (Continued)

Mean mol wt, kg/mol	Calculation	295			
Acid value, mg KOH/g	(50)	0.19		ÖNORM C1146	Max. 1.0
Saponification value, mg KOH/g	(50)	194.26			
Iodine value	(51)	93.19			
(Kaufmann method, gL/100 g)					
Water content, wt %	ASTM D 1744	0.0002			
Sulphur content, wt %	ASTM D 4294	0.0001	0.26	ÖNORM EN 41	Max. 0.02
Cetane index	(57)	49.6	-		
Cetane number			42.9	ISO 5165 ISO 3104 ISO 3105	Min. 48
Viscosity, cSt	ASTM D 495				
20°C					6.5-9.0
37.8°C		4.1	2.94		
Flash point, °C	ASTM D 92	171	52	ÖNORM C1122 or ISO 2718	Min. 55
Pour point, °C	ASTM D 97	-13	-18		
Cold filter plugging point, °C	P 309/83	-18	-	ÖNORM EN 116	Max. -6
Gross heating value, MJ/kg	ASTM D 240	41.0	45.3		
Copper corrosion (3 h, 100°C)	ASTM D 130	No. 1a	No. 1a		

CONCLUSIONS

In this study, the possibility of crude rapeseed oil as Biodiesel was investigated. This study is the first laboratory scale investigation made in Turkey concerning the evaluation of rapeseed as an alternative diesel fuel.

Biodiesel was produced through the rapeseed oil-methanol transesterification reaction. The reaction conditions were determined as follows:

Oil/alcohol molar ratio	1:6
Catalyst (NaOH)	1.7% of the oil weight
Temperature	$65 \pm 1^{\circ}\text{C}$
Duration	38 min

The ester yield of the reaction was 96.36%. From the product mixture, the methyl ester (Biodiesel) was obtained with a purity of 99.01% through the refinement method of washing the mixture with petroleum ether and water. The fuel properties of the Biodiesel were determined with the help of the standard tests and it was found that the Biodiesel properties were very close to those of Grade No. 2-D diesel fuel.

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