Optimization of an Ultrasonic-Assisted Biodiesel Production Process from One Genotype of Rapeseed (TERI (OE) R-983) as a Novel Feedstock Using Response Surface Methodology

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Abstract: In recent years, due to the favorable climate conditions of Iran, the cultivation of rapeseed has increased significantly. The aim of this study was to investigate the possibility of biodiesel production from one genotype of rapeseed (TERI (OE) R-983). An ultrasonic approach was used in order to intensify the reaction. Response surface methodology (RSM) was applied to identify the optimum conditions of the process. The results of this research showed that the conversion of biodiesel was found to be 87.175% under the optimized conditions of a 4.63:1 molar ratio (methanol to oil), 56.50% amplitude, and 0.4 s pulses for a reaction time of 5.22 min. Increasing the operating conditions, such as the molar ratio from 4:1 to 5.5:1, amplitude from 50% to 72.5%, reaction time from 3 min to 7 min, and pulse from 0.4 s to 1 s, increased the FAME (fatty acid methyl esters) yield by approximately 4.5%, 2.3%, 1.2%, and 0.5%, respectively. The properties of the TERI (OE) R-983 methyl ester met the requirements of the biodiesel standard (ASTM D6751), indicating the potential of the produced biodiesel as an alternative fuel.

Keywords: TERI (OE) R-983; biodiesel; ultrasonic; RSM; renewable energy; novel feedstock

1. Introduction

Diesel engines have acquired significant consideration by automobile manufacturing industries and factories in recent years due to their high thermal efficiency, extremely clean exhaust emission, and better fuel economy [1,2]. The traditional fuel for diesel engines is supplied from nonrenewable resources such as fossil fuels. The use of such types of fuel is associated with many risks, such as being prone to depletion and contributing to global warming through greenhouse gas emissions. Hence, many restrictions have been imposed on the use of such fuels by environmental authorities and international communities in recent years [3,4]. In order to implement the restrictive regulations on the use of fossil fuels, an alternative option must be considered. According to the conducted studies, renewable energies are suitable substitutes for fossil resources [5–7]. One of the attractive alternatives to diesel fuel is biodiesel. Chemistry of biodiesel production is as follow:

After the oil has been prepared, its free fatty acids (FFAs) have to be examined. The presence of free fatty acids (FFAs) in vegetable oils leads to formation of soap and reduces the efficiency of methyl ester

conversion as a result. Researchers have found that FFA>1% is undesirable since free fatty acids (FFAs) cause soap formation in the process of base-catalyzed transesterification; thus, an increase in the free fatty acids (FFAs) hinders the removal of biodiesel, glycerin and water in biodiesel production process. The esterification reaction is used to reduce the free fatty acids (FFAs). Esterification is a reaction between the oil and the acid catalyst and causes the free fatty acids (FFAs) to decrease [8]. Sulfuric acid (as an acid catalyst) and methanol are usually used in the esterification process. An acid number of less than unity shows that the free fatty acids (FFAs) content is rather low and the oil can be used as a feedstock with high certainty. After the oil has been acknowledged in terms of the free fatty acids (FFAs) content, the transesterification reaction is performed to improve the mechanical and chemical properties of the oil [9]. Transesterification is a reaction between alcohol and vegetable oil in presence of an acidic, alkali and enzyme catalyst. Moreover, mostly methanol is used in the transesterification reaction to break the triglyceride bonds. As a result, fatty acid methyl esters (FAME) are produced upon the completion of the transesterification reaction [10].

Due to its low contamination during combustion, biodiesel has been utilized considerably in recent years [6,11–14]. Various methods have been investigated in order to produce biodiesel [15]. One of the shortcomings of the conventional method of biodiesel production is its high energy consumption [16]. Using ultrasonic systems is one of the strategies adopted to reduce the energy demand for biodiesel production, which has been studied by various researchers [17–19]. Some of the conducted studies are discussed below.

Teixeira et al. [20] used two methods of biodiesel production—the conventional method and the one involving use of an ultrasonic system—to produce biodiesel from beef tallow. In this study, using ultrasound irradiation (400 W, 24 kHz), transesterification of beef tallow was performed with methanol and in the presence of potassium hydroxide as a catalyst. In this study, the reaction time, the conversion percent, and the quality of the biodiesel produced using the ultrasound system were compared with the conventional method. The results showed that using ultrasound irradiation reduces the reaction time, while the conversion percent and the quality of the biodiesel were the same as those of the conventional method. Kumar et al. [21] investigated the effect of using ultrasound irradiation on decreasing the reaction time of biodiesel production from coconut oil. The results of their study indicated that using ultrasound irradiation reduces the reaction time by 15–40 min. Manh et al. [22] studied the effect of ultrasound irradiation on the transesterification yield of biodiesel produced from Tung (*Vernicia fordii*) and biodiesel produced from different blended oils. In this study, the properties of the produced biodiesel, including the acid value (AV), iodine value (IV), kinematic viscosity (KV), density, and cold filter plugging point (CFPP), were evaluated. Results showed that during the use of ultrasound irradiation, the transesterification yields of biodiesel obtained from Tung and biodiesel produced from blended oils were 91.15% and 94.03%, respectively. Also, the results showed that the time required to produce high-quality biodiesel using ultrasound irradiation is 30 min, which is a very short time compared to the time needed in conventional biodiesel production. Tamilarasan and Sahadevan [23] used an ultrasonic system to study the optimum conditions for biodiesel production from algal oil from the marine macroalgae *Caulerpa peltata*. Results showed that using the ultrasonic system, the reaction time considerably reduces with respect to the conventional method. Also, the results indicated that optimum conditions are achievable when: the concentration of sulfuric acid (H_2SO_4) is 2 wt %, the methanol-to-oil molar ratio is 15:1, the reaction time is 30 min, the temperature is at 65 °C, and the stirrer speed is equal to 500 rpm. Maghami et al. [24] compared biodiesel fuel production from waste fish oil (WFO) using conventional transesterification and assisted transesterification with ultrasound. Results of this study revealed that the optimum conditions for biodiesel production were a 6:1 alcohol-to-oil molar ratio, 55 °C, and 1% KOH. In this condition, biodiesel yields from conventional and ultrasonic methods were 78% and 79.6%, respectively. In addition, results showed that during the use of the ultrasonic method, the reaction time decreased by 30 min. Samani et al. [18] produced biodiesel from *Pistacia atlantica Desf* oilseeds with the help of an ultrasonic system. Results obtained from this study showed that increasing amplitude and pulse leads to methyl

ester content enhancement. Results also indicated that by increasing the reaction time and molar ratio, the methyl ester content increases. In addition to the above, various research works that explored the effect of biodiesel production by ultrasound irradiation on the reduction of reaction time and the energy needed for biodiesel production all revealed that using ultrasound irradiation decreases the reaction time notably [25–35]. In Table A1 (Table A1), recent research in biodiesel production technology is described. Also in Table A1, the biodiesel yield and biodiesel properties of the different feedstocks are described (Appendix A).

Despite the large number of studies that have investigated ultrasonic-assisted biodiesel production, it appears that there has been no thorough study conducted on the use of ultrasound for improving biodiesel production from rapeseed oil.

Rapeseeds are divided into two groups. These categories are based on the percentage of erucic acid: (a) inedible rapeseed with an erucic acid content of over 40%; because of its high erucic acid content, it is toxic, thus is not used as a food, but is used in industry as a fuel and lubricant. For example, the cultivars HNS9801, Crambe, Millennium, and NPN01, among others, are used for industrial use in some countries, such as the USA, Europe, Belgium, and France; (b) edible rapeseed (canola) with an erucic acid of over 2%; this type of rapeseed is not toxic, but is not suitable as a food. Different researchers, via genetic modification and the use of additives, have reduced the content of erucic acid of rapeseed to below 2%. As a result, it was converted into edible rapeseed (canola).

The potential of rapeseed oil in Iran as a biodiesel resource is as follows:

- In terms of canola cultivation, Iran is divided into four climates (the warm climates of the north and south of Iran, fairly cold, and cold). In cold climates, the winter cultivars of rapeseed are cultivated, and in the two warm climates, the spring cultivars are cultivated. So, all provinces of Iran have potential for cultivating rapeseed;
- Rapeseed requires low quantities of water and is appropriate for the climate of Iran;
- Due to its rotation with wheat, barley, and rice, rapeseed is called an economic plant;
- Due to the problems of drought and water scarcity and increasing desertification in Iran, according to the instructions of the Ministry of Agriculture in Iran, farmers must move towards strategic cultivation. One of the strategic products is the cultivation of rapeseed.

Rapeseed cultivation in Iran over the years is shown in Figure 1. Iran has a very high potential for rapeseed cultivation. Therefore, the area under cultivation of rapeseed can be increased to produce biodiesel. Rapeseed can be cultivated in deserted areas that are useless. In this way, biodiesel can be used despite desertification. As a result, environmental pollutants are reduced and the health of humans, animals, and the environment is protected.
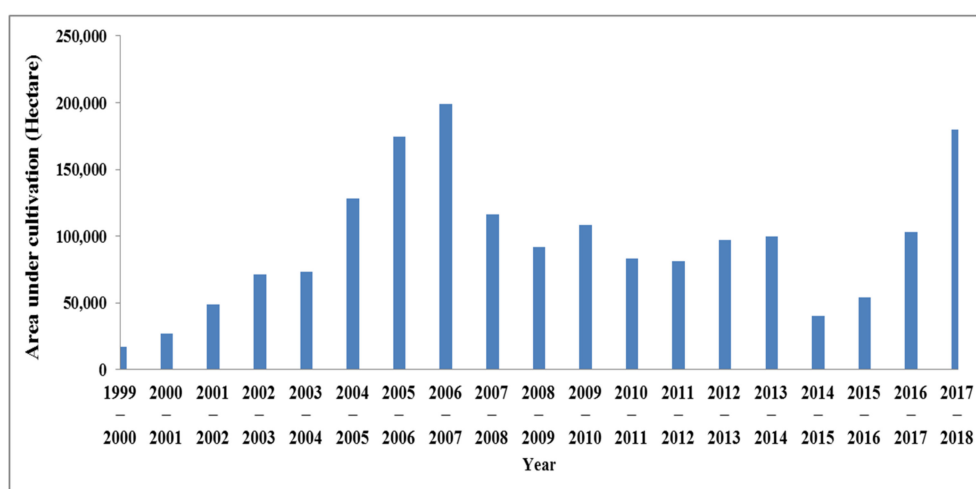

Figure 1. Rapeseed cultivation in Iran over the years.

Considering the fact that Iran has great potential for rapeseed cultivation, this study was thus designed to study the possibility of biodiesel production by ultrasound irradiation to identify the optimal conditions for biodiesel production using response surface methodology (RSM).

2. Material and Method

2.1. Equipment and Materials

In this study, one genotype of rapeseed (TERI (OE) R-983) oil was used to produce biodiesel. The TERI (OE) R-983 genotype contains 2–3% of erucic acid. Thereby, the TERI (OE) R-983 genotype of rapeseed does not have a standard for edibility. Oil was obtained from Jouybar Industrial Park located in Mazandaran province. High-purity KOH and methanol (99%) were used to prepare the biodiesel (these materials were sourced from Mina Tajhiz Aria Co., Tehran, Iran). All other chemicals used in this study were obtained from Merck Chemical Industries. Fatty acids and methyl esters (FAMEs) were analyzed by Gas chromatography (GC).

An ultrasonic system (Hielscher Model UP400S), that was provided by Farasout Sanat Engineering Co., Tehran, Iran, was used to do the transesterification process. The ultrasonic system used in this study is shown in Figure 2. The system consisted of a processor, a sonotrode, and a Personal computer (PC) controller. The ultrasonic probe is made of a titanium tip with a diameter of 7 mm and a length of approximately 100 mm. The applied operating parameters of the used ultrasonic system were 400 W and 24 kHz.
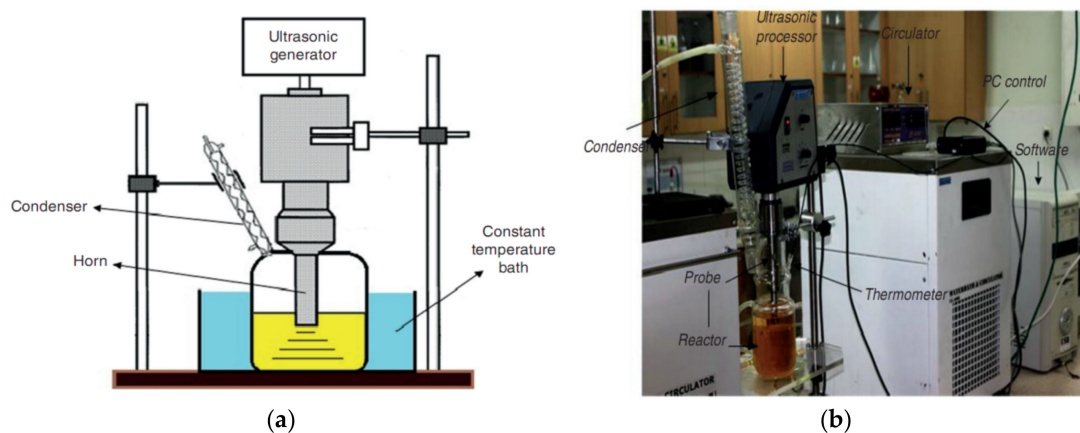

(**a**)   (**b**)

Figure 2. The biodiesel production set-up: (**a**) schematic; (**b**) device [36].

2.2. Esterification of TERI (OE) R-983 Oil

Esterification was applied to reduce the acid value content of TERI (OE) R-983 oil. Sulfuric acid (1 wt.%) was used as a catalyst for the esterification reaction. An appropriate methanol-to-oil molar ratio (MR) was applied to reduce the FFA content (MR = 9). Sulfuric acid was added to methanol and the mixture was heated to 50 °C using a heater. Later, the oil was poured to a jacketed glass reactor and was then heated to 60 °C. Thereupon, the acid and methanol mixture was added to the oil to drive the esterification reaction. After that, the mixture was transferred to a separator and after 24 h, the mixture separated into two phases: the top layer was unused methanol and acid, and the bottom layer was the oil. After separating the oil from the unused methanol and acid, the oil's acid number was measured and was found to be equal to 0.45 mg KOH/g oil, which is an appropriate amount for the transesterification reaction [37].

2.3. Analysis of Fatty Acids Structure

The Metcalfe method [38] was applied to identify the structure of the oil. The steps of the process were as follows:

About 50 mg of oil was added to a 15 mL test tube. Five milliliters of a methanol/soda mixture (NaOH solution (2%) in methanol) was prepared. The solution was sited in boiling water (100 °C) for 10 min. After the solution cooled down, 2.175 mL of boron trifluoride (BF$_3$) 20% was added and it then was placed again in the boiling water for 3 min. As soon as the solution was cooled down, 1 mL of n-heptane was added to it, and next, the solution was mixed on an orbital shaker. After that, to separate the organic phase containing methyl esters of fatty acids, 1 mL of saturated NaCl salt acid was added to the solution, and the solution was mixed again on the shaker. Then, the fatty acid phase separated from the solution was transferred into a new microtube. Finally, gas chromatography with a Clarus 580 GC (Perkin Elmer Co., Waltham, United States of America) was used to determine the fatty acid structure. After methylation of oil, 1 µL of the solution was injected into the Clarus 580. High-purity helium (99.99%) was used as the carrier gas. The temperatures of the injection port and the detector were set to be 250 °C and 300 °C, respectively.

The following temperature program for the column of the Clarus 580 was employed: 170 °C for 4 min, held at 180 °C for 0.2 min after ramping at 3 °C/min, and held at 210 °C for 25 min after ramping at 1 °C/min. Flame ionization detection (FID) was used as a detector. The total run time for a single sample was about 40 min [38]. Table 1 shows the fatty acid profile of TERI (OE) R-983 oil.

Table 1. Fatty acid profile of TERI (OE) R-983 oil.

Properties	Unit	Amount
Palmitic (C16:0)	wt.%	4.30
Palmitoleic (C16:1)	wt.%	0.19
Stearic (C18:0)	wt.%	2.21
Oleic (C18:1)	wt.%	65.94
Linoleic (C18:2)	wt.%	17.32
Linolenic (C18:3)	wt.%	6.05
Other fatty acids	wt.%	3.99
Mean molecular weight of used oil	gr/mol	882.92
Acid value	mg KOH/g oil	0.57

2.4. Transesterification

To do transesterification, methanol and potassium hydroxide were mixed for 5 min using a magnetic stirrer. The formed methoxide was then transferred to a conical flask and mixed with TERI (OE) R-983 oil. Then, the prepared mixture was transferred into the ultrasonic system for treatment. Ultrasound parameters, such as reaction time, amplitude, and pulse, were controlled by a PC controller. During the experiments, the reaction temperature and catalyst concentration were maintained at 55 °C and 1 wt.%, respectively.

2.5. FAME Yield Calculation and Statistical Analysis

After analyzing the fatty acid methyl esters (FAMEs) by GC, the FAME yield of each of the transesterification process' steps were calculated by applying Equations (1)–(3):

$$\text{FAME content}(\%) = \frac{[\sum A] - A_i}{A_i} \times \frac{m_i}{M} \times 100; \tag{1}$$

$$\text{Amount of FAME}(g) = \text{FAME content}(\%) \times \text{weight of product}(g); \tag{2}$$

$$\text{Biodiesel yield}(\%) = 100 \times \frac{\text{amount of FAME}(g)}{\text{amount of none} - \text{edible canola}(g)}; \tag{3}$$

where FAME = produced methyl esters weight; A_i = peak area of internal standard; M = weight of biodiesel phase in the reaction mixture; $\sum A$ total peaks area of chromatogram; m_i = weight of biodiesel for GC analysis.

One of the goals of this study was to determine the optimum conditions for biodiesel production. To achieve this purpose, the RSM method was applied using Design Expert software. In this study, four independent variables—molar ratio, amplitude, time, and pulse—were investigated. The selected independent variables and their levels are shown in Table 2.

Table 2. Selected independent variables in the response surface method.

Independent Variables	Units	Symbols	Level of Each Factor		
			Coded Values		
			−1	0	1
Molar Ratio	-	X1	4	5	6
Reaction Time	min	X2	3	5	7
Pulse	s	X3	0.4	0.7	1
Amplitude	%	X4	50	65	80

Design Expert software was used to design a table of randomized runs as illustrated in Table 3.

Table 3. Design experiments.

Random	Run	Molar Ratio	Reaction Time	Pulse	Amplitude	Reposes (Yield)
16	1	5:1	7	1	65	84.47
17	2	4:1	5	0.4	65	84.77
26	3	5:1	5	0.7	65	85.5
3	4	4:1	7	0.7	65	86.04
6	5	5:1	5	1	50	88.62
22	6	5:1	7	0.7	50	87
15	7	5:1	3	1	65	86.69
27	8	5:1	5	0.7	65	88.34
18	9	6:1	5	0.4	65	86.6
4	10	6:1	7	0.7	65	88.49
9	11	4:1	5	0.7	50	83.8
8	12	5:1	5	1	80	87.6
23	13	5:1	3	0.7	80	88.07
14	14	5:1	7	0.4	65	88.53
7	15	5:1	5	0.4	80	88.19
12	16	6:1	5	0.7	80	86.51
10	17	6:1	5	0.7	50	86.21
29	18	5:1	5	0.7	65	88.33
19	19	4:1	5	1	65	86.42
11	20	4:1	5	0.7	80	84.46
21	21	5:1	3	0.7	50	87.51
20	22	6:1	5	1	65	87.12
1	23	4:1	3	0.7	65	84.5
28	24	5:1	5	0.7	65	87.42
24	25	5:1	7	70	80	87.24
13	26	5:1	3	0.4	65	87.67
25	27	5:1	5	0.7	65	87.7
2	28	6:1	3	0.7	65	86.29
5	29	5:1	5	0.4	50	87.92

3. Result and Discussion

3.1. Statistical Analysis

Design Expert(R) software proposed the polynomial quadratic model. The statistical analysis was performed on this model. The suggested model is dependent on analysis of variance (ANOVA) and is based on the coded values of the experimental factors (Table 3), as offered by Equation (4).

$$\text{Conversion (\%)} = 88.06 + 1.00 \times A + 0.42 \times B + 0.10 \times C + 0.14 \times D + 0.17 \times A \times B - 0.28 \times A \times C - 0.27 \times A \times D + 0.23 \times B \times C - 0.080 \times B \times D - 0.32 \times C \times D - 2.05 \times A^2 - 0.050 \times B^2 + 0.21 \times C^2 - 0.56 \times D^2,$$

(4)

where A, B, C, D are molar ratio (methanol to oil), reaction time (min), the pulse(s), and amplitude (%) respectively. Table 5 shows the suggested model by RSM. The results of the variance examination of the RSM model are shown predicated on the ANOVA examination in Table 4. As shown in Table 5, the possibility value (p value) for the model is significantly less than 0.01. Therefore, ANOVA implies that the model is statistically significant. Corresponding to Table 5, it is obvious that the effects of molar ratio (methanol to oil) and time have a substantial influence on biodiesel production. The discussion of the relationship between the molar ratio (methanol to oil) and reaction time is significant. Other factors do not have a significant effect on biodiesel yield.

Table 4. Quadratic model statistics.

Statistical Model	Std. Dev.	CV *%	R^2	Adjusted R^2	Predicted R^2	PRESS	Result of Model
Linear	3.81	4.63	0.6836	0.6309	0.5501	495.97	
2FI **	3.73	4.53	0.7732	0.6472	0.4557	600.00	
Quadratic	2.67	3.24	0.9096	0.8692	0.5040	546.77	Suggested
Cubic	1.72	2.09	0.9839	0.9251	−0.4745	1625.41	Aliased

* Coefficient of variation, ** two factor interaction.

Table 5. ANOVA table of model.

Source	Sum of Squares	df	Mean Square	F Value	p Value Prob > F
Model	46.55496	14	3.325354	4.7904466	0.0030
Molar ratio (A)	11.90818	1	11.90818	17.154709	0.0010
Reaction time (B)	2.1168	1	2.1168	3.0494248	0.1027
Pulse (C)	0.128133	1	0.128133	0.1845866	0.6740
Amplitude (D)	0.250563	1	0.250563	0.3609566	0.5576
A–B interaction effects	0.1089	1	0.1089	0.1568794	0.6980
A–C interaction effects	0.319225	1	0.319225	0.4598699	0.5087
A–D interaction effects	0.293764	1	0.293764	0.4231913	0.5259
B–C interaction effects	0.2116	1	0.2116	0.3048272	0.5896
B–D interaction effects	0.0256	1	0.0256	0.0368789	0.8505
C–D interaction effects	0.416025	1	0.416025	0.5993183	0.4517
A^2	27.29715	1	27.29715	39.323791	<0.0001
B^2	0.016001	1	0.016001	0.0230504	0.8815
C^2	0.293825	1	0.293825	0.4232792	0.5258
D^2	2.044467	1	2.044467	2.9452233	0.1082
Residual	9.718292	14	0.694164		
Lack of Fit	8.834212	10	0.883421	3.9970191	0.0970
Pure Error	0.88408	4	0.22102		
Cor Total	56.27325	28			

3.2. Effects of Molar Ratio and Reaction Time on FAME Yield

Figure 3 shows the effect of molar ratio and reaction time on FAME yields. It can be seen from Figure 3 that with increasing the molar ratio up to 5.5, FAME yields increase. However, increasing the molar ratio over this level leads to a reduction in FAME yields. The main reason for this is

the increasing amount of methanol in the mixture, which leads to more dissolution of glycerin and alcohol in biodiesel [39]. Also, Figure 3 shows that by increasing the reaction time, FAME yields increase. Because the required methanol is available throughout the reaction time, by increasing the reaction time at the different methanol molar ratios, the FAME yield increases. Also, by increasing the molar ratio, higher amounts of alcohol are available to react with the triglycerides, and therefore, the FAME yield will have an increasing trend. At high molar ratios and lower times, due to the lack of sufficient time, the reaction is not completed, thus, the FAME yield decreased.
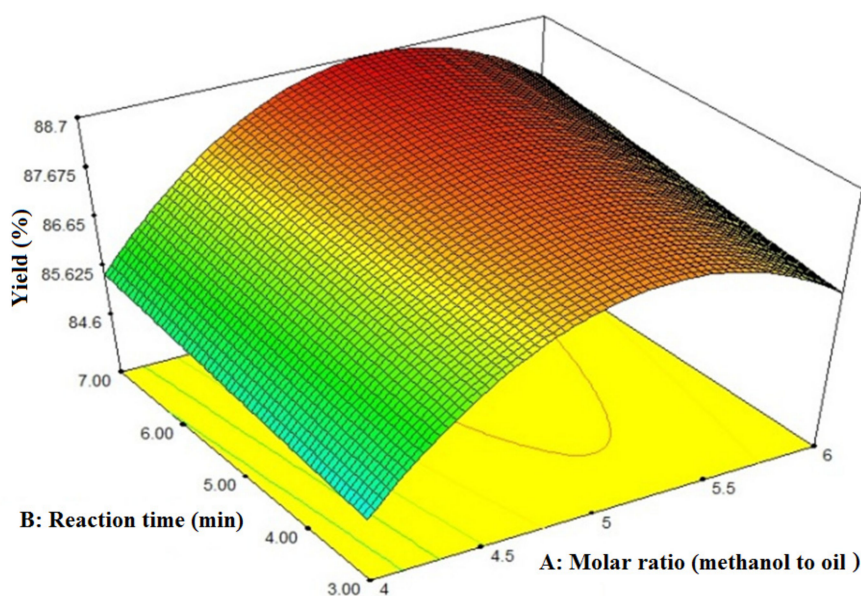
Figure 3. Effect of reaction time and molar ratio on yield (methyl ester content) at a pulse of 0.4 s.

3.3. Interaction Effects of Pulse and Molar Ratio (Methanol-to-Oil Ratio) on FAME Yield

The effect of pulse on FAME yield is shown in Figure 4. By increasing the pulse, the FAME yield increases slightly. A higher pulse increases the mixture rate, so mass transfer is highly performed, and therefore the FAME yield increases. It should be noted that if the pulse exceeds a certain amount, the FAME yield increases with a lower slope. It has been proven that if the ultrasonic pulse is less than 1, mass transfer occurs better and less energy is consumed; however, if the pulse value is less than 0.7, the ultrasonic macrostirring effect is low and cannot mix the immiscible reactants together, and therefore, the yield of biodiesel decreases. Also in Figure 4, the effect of molar ratio on FAME yield is shown. By increasing the molar ratio (methanol-to-oil ratio) to 5.5:1 in pulse about 1 s, the yield of FAME increased. However, by increasing the molar ratio (methanol-to-oil ratio) over 5.5:1, the yield of FAME decreased. According to Figure 4, when the molar ratio (methanol-to-oil ratio) is about 5.5:1 and the pulse is about 1 s, the yield of FAME is highest.
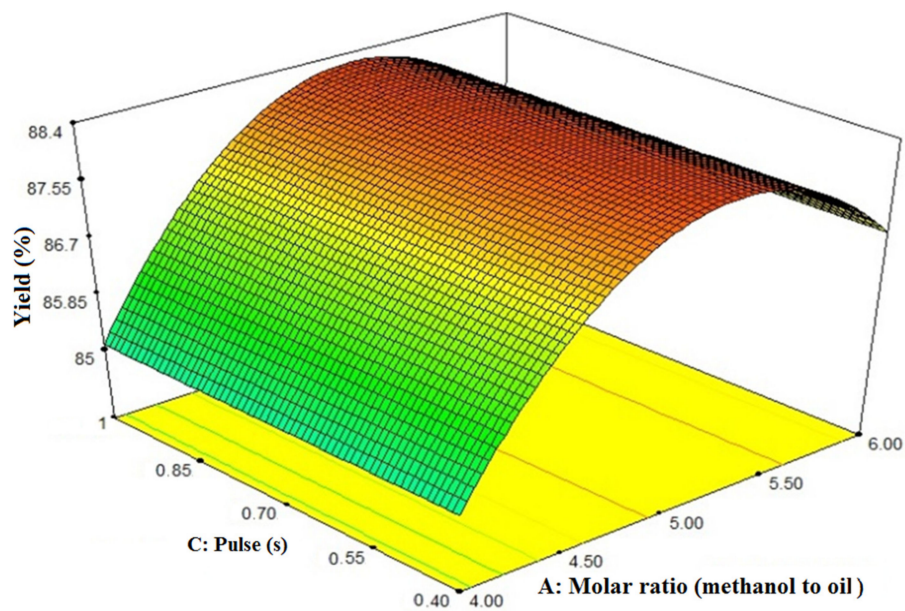
Figure 4. Effect of pulse on yield (methyl ester content) at the reaction time of 5.22 min.

3.4. Interaction Effects of Amplitude and Molar Ratio (Methanol-to-Oil Ratio) on FAME Yield

The impact of ultrasonic amplitude on FAME yield is depicted in Figure 5. By increasing the amplitude of acoustic waves, the droplets get smaller in size and the number of circulating bubbles leads to increased mixing of oil and alcohol, thus a better emulsion is generated [38]. As shown in Figure 5, by increasing the amplitude to about 72.5%, the biodiesel conversion rate increases, and when the amplitude reaches over 72.5%, the amount of FAME yield decreases. The high amplitude increases the cracking; thus, the fatty acid methyl esters oxidized to aldehydes, ketones, and lower-chained organic fractions [40]. Also in Figure 5, the effect of molar ratio on FAME yield is shown. By increasing the molar ratio (methanol-to-oil ratio) to 5.5:1, the yield of FAME increases. However, by increasing the molar ratio (methanol-to-oil ratio) over 5.5:1, the yield of FAME decreased. According to Figure 5, when the molar ratio (methanol-to-oil ratio) is about 5.5:1 and the amplitude is about 74%, the yield of FAME is highest.
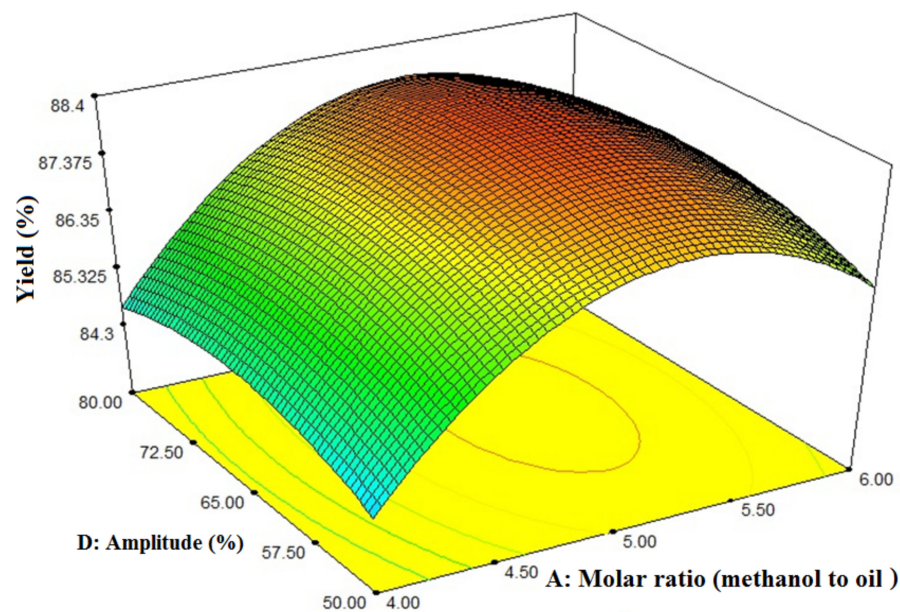

Figure 5. Effect of amplitude on yield (methyl ester content) at a pulse of 0.4 s.

3.5. Optimization of the Production Process Using Desirability Function

The optimal conditions for biodiesel production were predicted using the multifactor desirability function. After the creation of biodiesel and separation of glycerin from the methyl ester, the methyl ester was examined using GC-FID and the most optimal yield was identified. The perfect value for each and every one of the analyzed variables was expected by Equation (4) to be able to get the highest rate of biodiesel alteration. Equation (4) is dependent on the regression polynomial formula, which comes from the response surface technique (RSM) model. The perfect value for each of the factors are as follows:

(a)   Molar ratio (methanol to oil) = 4.63:1;
(b)   Reaction time = 5.22 min;
(c)   Pulse = 0.4 s;
(d)   Amplitude = 56.50%.

The regression polynomial formula estimates that if the factors acquire these values, the peak of biodiesel yield (87.175%) is obtained. To be able to validate the predicted value, the optimized parameters were experimentally analyzed and the biodiesel yield was assessed to be 87.9%. Experimental results confirmed that the worthiness expected by the response surface strategy (RSM) has an excellent agreement with the experimental results, given that the reaction time in this research is short. Thus, the FAME yield is under 95% in optimized conditions.

3.6. Properties of the Produced Biodiesel

Table 6 presents the properties of the biodiesel produced in this study. The properties of the produced methyl ester, including flash point, pour point, kinematic viscosity at 40 °C, density, acid value, and oxidative stability, were measured using the ASTM standard. All the measured properties were then compared with a biodiesel standard (EN 14214). Kinematic viscosity is one of the most important characteristics of a fuel and has a significant impact on the erosion of the engine parts. The main purpose of the esterification or transesterification process is to reduce the viscosity of vegetable oils. As shown in Table 6, the viscosity is 4.65 mm^2/s, thus, the viscosity of biodiesel produced from TERI (OE) R-983 oil is acceptable. Research results of Hoekman et al. [41] showed that in twelve types of biodiesel, the range of kinematic viscosity was from 4 to 5 mm^2/s.

Table 6. Physicochemical characterization of TERI (OE) R-983 biodiesel.

Properties	Test Method	Limits	Units	Measured Properties
Water and sediment	ASTM D2709	Max 0.05	% volume	<0.004
Density at 15 °C	ASTM D4052	0.86–0.90	kg/cm^3	0.88
Kinematic viscosity at 40 °C	ASTM D445	1.9–6.0	mm^2/s	4.65
Oxidation stability	EN 14112	Min 3	h	3.2
Flash point, closed cup	ASTM D93	Min 130	°C	213
Pour point	ASTM D97	−15 to 10	°C	−9
Acid number	ASTM D664	Max 0.50	mg KOH/g	0.29

According to Table 7, it is clear that the flash point, pour point, acid value, and oxidative stability are consistent with reports from other researchers [21,22,42]. It is important to note here that the properties of the produced biodiesel are in the range of the standard-defined properties (ASTM D6751 and EN 14112).

4. Comparison with the Literature

In Table 7, the properties of TERI (OE) R-983 biodiesel are compared with those of recently published research studies. The properties of TERI (OE) R-983 biodiesel are similar to the properties of other biodiesels. Density is an important parameter of biodiesel, with an impact on fuel quality. As shown in Table 7, the density of TERI (OE) R-983 biodiesel is less than some of the biodiesel

types. The specific gravity of biodiesel can have a great impact on the mixing of diesel and biodiesel. The specific gravity of biodiesel fuel should be such that the diesel and biodiesel fuel phases are not separated after mixing.

One of the deficiencies of biodiesel fuel is the high value of viscosity. High viscosity means less fuel atomization, higher resistance to fuel pumping, damage to the fuel filter, poor combustion, and increased exhaust gases [43]. The kinematic viscosity of TERI (OE) R-983 biodiesel is high than some of the biodiesel types. Also, the pour point of TERI (OE) R-983 biodiesel is low, which can be considered an advantage of TERI (OE) R-983 biodiesel compared with some other biodiesel types. Additionally, the oxidation stability of TERI (OE) R-983 biodiesel is better than Kusum biodiesel.

Table 7. Comparison of the properties of TERI (OE) R-983 biodiesel with biodiesels made from other feedstock.

Feedstocks	Density at 15 °C (kg/cm^3)	Kinematic Viscosity at 40 °C (mm^2/s)	Pour Point (°C)	Flash Point (°C)	Acid Value (mg KOH/g)	Oxidation Stability (h)
Current study (TERI (OE) R-983)	0.8822	4.6481	−9	213	0.29	3.20
Waste cooking oil [44]	0.875	3.84	−1	164	0.27	-
Kusum oil [45]	0.857	5.34	−2	152	0.42	0.86
Waste cooking oil [46]	0.888	2.35	−19	185		-
Canola oil [47]	0.884	4.51		184		-
Rapeseed oil [48]	0.882	4.62		175.5	0.41	-

5. Conclusions

The feasibility of biodiesel production from one genotype of rapeseed (TERI (OE) R-983) using ultrasound irradiation was investigated, and the optimum conditions for production were identified using RSM. The following results were obtained:

- The molar ratio of alcohol to oil has a positive effect on the transesterification reaction and, consequently, on biodiesel conversion. Increasing the molar ratio from 4:1 to 6:1 increased the FAME yield by approximately 4.5%;
- The optimal value for the FAME yield from TERI (OE) R-983 oil using ultrasound irradiation was 87.175%; the conditions that resulted in this value were: a 4.63:1 alcohol-to-oil molar ratio, 5.22 min reaction time, 0.40 s pulse, and 56.50% amplitude;
- The properties of the produced biodiesel being within the range of the standard-defined properties (ASTM D6751) highlights the potential of such a biodiesel to be used as an alternative fuel.

Appendix A

Table A1. Biodiesel production technology and properties of biodiesel.

References	[45]	[44]	[46]	[48]	[49]	[47]
Feedstock	Kusum	Waste cooking oil	Waste cooking oil	Rapeseed, soybean, coconut, and palm	Waste cooking oil	Canola
Production technology	Ultrasound	Ultrasound	Microwave	Ultrasound	Ultrasound	Ultrasound
Operating temperature (°C)	50	60	60	25	30	25
Run time (min)	80	30	5	−	30	50
Catalyst	Ba(OH)$_2$	Calcium triglyceride	NaOCH$_3$	KOH	KOH	KOH
Yield (%)	96.8%	93.5%	98.87%	Rapeseed (95.03%), soybean (94.66%), palm (93.08%), coconut (81.37%)	90%	>99%
Pour point (°C)	−2	−1	−19	−	−	−
Flash point (°C)	152	164	185	Rapeseed (175.5), soybean (169), coconut (108), palm (131.5)	150	184
Acid value (mgKOH/g)	0.42	0.27		Rapeseed (0.41), soybean (0.51), coconut (0.29), palm (0.43)	−	−
Viscosity (mm^2/s, 40 °C)	5.34	3.84	2.35	Rapeseed (4.62), soybean (4.7), coconut (3.2), palm (5.63)	4.08	4.51
Density at 15 °C (mg/m^{-3})	857	875	888	Rapeseed (882), soybean (883), coconut (874), palm (874)	891.1	884
Oxidation stability (h)	0.86	−	−	−	−	−
Water content (ppm)	−	−	−	Rapeseed (303.21), soybean (412.2), coconut (485.17), palm (463.32)	−	390

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