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Optimization of the Transesterification Reaction in Biodiesel Production and Determination of Density and Viscosity of Biodiesel/Diesel Blends at Several Temperatures

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ABSTRACT: Waste frying oil has been used to optimize the production of biodiesel. Biodiesel was prepared through sodium ethoxide catalyzed methanolysis from the transesterification of recycled waste frying oil. Optimization of the transesterification reaction for biodiesel production was carried out by means of statistical analyses using ANOVA. The optimum conditions for reaction were the following: a oil—methanol mole ratio of 1:9, temperature of 50 °C, catalyst mass fraction of 0.9 %, and reaction time of 40 min, which enabled a yield of 98.7 % determined by gas chromatography/mass spectrometry (GC/MS) analysis. The density and viscosity of biodiesel/diesel blends have been determined as a function of composition at several temperatures.

INTRODUCTION

Air pollution is one of the most serious environmental problems worldwide. Since diesel engines exhaust a huge amount of pollutants, a clean alternative fuel becomes highly necessary, and many efforts to develop clean fuels have been under way in many countries. Among many possible sources, biodiesel fuel derived from vegetable and animal fat has attracted attention as a possible substitute for petrodiesel fuels.[1−9] Biodiesel is defined by the Brazilian Biodiesel Program as "a fuel obtained from mixtures, at different proportions, of fossil diesel and alkyl esters derived from vegetable oil or animal fats". Technically speaking, biodiesel is the alkyl ester of fatty acids, made by a chemical reaction called transesterification of vegetable oil or animal fats, with short-chain alcohols (normally methanol or ethanol).[6] The continuous use of petroleum increases the amount of carbon dioxide released into the atmosphere. However, if pure or blend biodiesel is used as fuel, the amount of released carbon dioxide can be reduced.

In this scenario, Brazil appears as an emerging power in the production of biodiesel, especially due to the following reasons. First, Brazil has climatic conditions to grow different kinds of crops. In Brazil, soybean oil is already used for biodiesel production, and other sources, such as sunflower, peanut, cotton, palm, coconut, babassu, and castor oil, may be used in the near future.[6] Second, Brazil is the world's leader in ethanol production from sugar cane, and the production of biodiesel using ethanol may become economically viable.

The purpose of this research is to study the optimization of biodiesel production from the transesterification of recycled waste frying oil as well as the effect of temperature on the density and viscosity of biodiesel/diesel blends. Mixtures were prepared at a volume fraction from B2 (indicates 2 % of biodiesel with 98 % diesel) to B100 (100 % biodiesel). The density and viscosity are known to significantly affect the atomization process, that is, the initial stage of combustion, in diesel engines.

Optimization of the transesterification reaction in biodiesel production has been studied by many authors. Salamatinia et al.[10] studied the effects of ultrasonic irradiation on production conditions of palm oil transesterification. Experimental data were used to optimize process conditions by means of response surface methodology (RSM). Ferella et al.[11] used RSM to determine the best conditions for the first reaction of the two-stage transesterification industrial process developed by Fox Petroli SpA. Kafuku and Mbarawa[12] produced biodiesel from *Croton megalocarpus* oil. Factors affecting the biodiesel production process such as reaction time, oil—alcohol mole ratio, temperature, agitation, and catalyst concentration were studied to determine the optimal process conditions for conversion of fatty acids to methyl esthers. Tan et al.[13] used the RSM to optimize the biodiesel production process by using the noncatalytic supercritical methyl acetate technology (SCMA), and various parameters on the yield of biodiesel were optimized. The optimum conditions were 399 °C for the reaction temperature, 30 $\text{mol}\cdot\text{mol}^{-1}$ of methyl acetate-to-oil mole ratio, and a reaction time of 59 min to achieve 97.6 % biodiesel yield. Bautista et al.[14] studied the biodiesel production from waste cooking oil. The process was optimized by using factorial design and RSM. The variable chosen was the fatty acid concentration in the waste cooking oil, while the responses were biodiesel purity and yield. Patil and Deng[15] proposed an optimization study on biodiesel production for edible (canola and corn) and nonedible (*Jatropha curcas* and *karanja*) vegetable oil. The process gave yields of about (90 to 95) % for *J. curcas*, (80 to 85) % for *Pinus glabra*, (80 to 95) % for canola, and (85 to 96) % for corn using potassium hydroxide (KOH) as a catalyst. Sinha et al.[16] studied the transesterification process for biodiesel production from rice bran oil. The various process variables like catalyst concentration, amount of methanol, reaction time, and temperature were optimized to produce biodiesel with maximum yield. Chen et al.[17] optimized the reaction conditions for biodiesel production

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in two-phase aqueous-oil systems with soluble lipase NS81006 and NS81020, produced by submerged fermentation of genetically modified *Aspergiluus oryzae/Aspergillus niger* microorganism. The central composite design (CCD) of the RSM was used to assess the effect of temperature, methanol:oil mole ratio, stirring rate, and enzyme concentration. Boauid et al.[18] studied the biodiesel production process from different vegetable oils. The authors developed and optimized the process by using factorial design and RSM. The effect of catalyst concentration, ethanol/oil mole ratio, and temperature were assessed. Catalyst concentration was found to be the most important parameter on conversion. Ghadge and Raheman[19] used the central composite rotatable design (CCRD) and RSM to study the effect of alcohol quantity, acid concentration, and reaction time on the reduction of free fatty acids content of muhua oil during its pretreatment for biodiesel production. The optimum conditions of these parameters reduced the acid level of mahua oil to less than 1 %. Antolín et al.[20] studied the biodiesel production by sunflower oil transesterification. The author used Taguchi's methodology for the optimization of temperature, reactant proportion, and methods of purification.

Studies on the density and viscosity of biodiesels have also been encountered in the literature. Baroutian et al.[21] determined the dynamic viscosity and density of binary and ternary blends of palm oil, biodiesel, and diesel fuel as a function of temperature. The viscosity and density data decreased nonlinearly and linearly with temperature, respectively. Albuquerque et al.[22] determined the density, viscosity, and index iodine of biodiesel oils obtained from different biomass sources and their binary blends prepared at different concentrations. It was observed that biodiesel obtained from castor oil presented problems regarding density and viscosity specification. Alptekin and Canakci[23] prepared blends using two different diesel fuels with biodiesels from six different vegetable oils. The density and viscosity of the blends were measured in accordance with ASTM test methods. The results showed that the viscosity and density values increased with the increasing biodiesel concentration in the fuel blends. Generalized equations were used to predict the viscosity and density of the blends. Excellent agreement was found between the measured and the estimated values of the properties. Dzida and Prusakiewicz[24] studied the effect of temperature and pressure on density, speed of sound, and specific heat capacities of petroleum diesel oil and biodiesel fuel. The temperature range studied was from (0 to 90) °C and pressure from (0.1 to 101) MPa. Benjumea et al.[25] determined basic properties of several palm oil biodiesel—diesel fuel blends according to the corresponding ASTM standards. Mixing rules were used as a function of volume fraction of biodiesel to predict the properties. Tate et al.[26,27] determined density and kinematic viscosity of three biodiesel fuels at temperatures up to 300 °C. The densities and viscosities of three biodiesel fuels decreased with the temperature. Kerschbaum and Rinke[28] studied the temperature effect on the viscosity of biodiesel fuel at temperatures below 0 °C. Samples of biodiesel were examined in the temperature range from (−15 to 30) °C, and empirical equations were used to correlate the viscosity.

■ EXPERIMENTAL SECTION

Materials. Waste frying oil was collected from the campus restaurants of the Centro Universitário da FEI. Methanol (99.85 % purity, Commercial Alcohols Inc.; Brampton, ON, Canada), sodium ethoxide (CH_3CH_2ONa, reagent grade, ACP Chemicals Inc.; Montreal, QC, Canada), sodium hydroxide (99 % purity, Merck), and potassium hydroxide (90 % purity, Merck) were used without further purification. Commercially available diesel fuel from PETROBRAS was purchased to prepare the blends.

Table 1. Yield, *Y*, Obtained with Different Catalysts

catalyst	% Y
sodium hydroxide	88.02 ± 0.55
potassium hydroxide	89.88 ± 0.72
sodium ethoxide	94.3 ± 0.58

Biodiesel Production. A pretreatment was performed with sodium chloride (NaCl) to neutralize the free fatty acids in the waste frying oil. The amount of NaCl needed for 300 g oil was 30 g. The sample was heated at 70 °C for 2 h. The treated oil sample presented an acid value of 0.96 mg of KOH/g. Afterward NaCl was separated from the oil, and the transesterification reaction was prepared. The best catalyst and its concentration and the best oil—methanol mole ratio were determined. The transesterification reaction was carried out at $t = 50$ °C, 30 min, and oil—methanol mole ratio of 1:6, and the catalysts tested were sodium hydroxide, potassium hydroxide, and sodium ethoxide. Sodium ethoxide was shown to provide the best conversion (Table 1). Conversion was determined by gas chromatography (GC) analysis, and tests were performed in duplicate. After having determined the best catalyst, catalyst concentration, mole ratio, temperature, and time were varied. The results were used to determine the best conditions for biodiesel production. The transesterification reactions were carried out with 100 g of oil and different amounts of sodium ethoxide, that is, (0.5, 1.0, and 1.5) g. Oil-to-methanol mole ratios tested were of 1:6, 1:8, and 1:12. Reactions took place at (40, 50, and 60) °C at different reaction times, namely, (15, 30, and 60) min. The reactions were maintained under agitation at a stirring frequency of 360 rpm. Reaction conditions were varied to determine the best condition for the production of biodiesel.

Chromatography. Biodiesel composition and free glycerol were determined by GC (Varian Star model 3600). The chromatograph was equipped with a flame ionization detector (FID) and a 60 m × 0.25 mm DBWAX column. The injector and detector temperature were set at 250 °C. The column temperature was initially maintained at 40 °C for 1 min, raised to 250 at 7 $°C\cdot min^{-1}$, and finally maintained at this temperature for 20 min. Ethyl ester was identified by comparison of its retention times with that of a standard solution.

GC-MS Analysis. Analyses were conducted in a Shimadzu GC-MS automated chromatograph model GC-17A/QP-5050A coupled to an AOC20i autosampler (Shimadzu, Kyoto, Japan). Chromatographic separation was performed in a DB-5MS nonpolar fused-silica capillary column (30 m × 0.25 mm i.d. × 0.25 μm film thickness; J&W Scientific, Folsom, CA, USA). All analyses were obtained using EI/MS (in positive mode) with scan acquisition mode (40 to 500 m/z). The oven temperature was initially at 60 °C, then increased at a rate of 3 $°C\cdot min^{-1}$ to 240 °C, and kept at this temperature for 10 min, resulting in total analysis time of 70 min. The carrier gas was helium at a constant flow rate of 1 $mL\cdot min^{-1}$, and sample aliquots (1 μL) were injected in split mode (1:20). Injector and detector temperatures were maintained at (220 and 240) °C, respectively.

Density. Density was determined by using a vibrating-tube densimeter (Anton Paar, model DMA 4500) according to ASTM D1298.

Viscosity. Viscosity was measured by using a viscometer Stabinger (Anton Paar, model SVM 3000) according to ASTM Standard D 445.

Table 2. Test Conditions and % Yield Obtained of the Full Factorial Design for Time, Temperature, Catalyst Mass Fraction, and Oil−Methanol Mole Ratio

t/min	θ/°C	% catalyst mass fraction	oil−methanol mole ratio	% yield	% yield	mean
15	40	1.5	1:8	88.45	88.67	88.56
15	40	0.5	1:6	89.32	89.83	89.58
60	50	0.5	1:6	90.53	90.94	90.74
30	60	1.5	1:8	87.28	88.43	87.86
15	60	1.0	1:6	91.71	92.58	92.15
15	50	0.5	1:12	90.48	92.3	91.39
60	60	1.0	1:8	88.78	89.3	89.04
15	60	1.5	1:12	86.41	87.29	86.85
60	60	1.5	1:6	85.43	86.36	85.90
30	60	0.5	1:6	88.31	88.34	88.33
15	60	0.5	1:8	89.12	88.79	88.96
60	40	1.0	1:6	88.92	87.54	88.23
60	50	1.5	1:8	92.3	91.98	92.14
30	40	1.0	1:8	81.45	83.53	82.49
60	60	0.5	1:12	88.65	88.21	88.43
30	50	1.0	1:6	90.55	90.87	90.71
15	50	1.0	1:8	95.1	93.24	94.17
60	40	1.5	1:12	89.98	89.76	89.87
60	40	0.5	1:8	89.96	89.21	89.59
30	40	1.5	1:6	88.98	88.76	88.87
30	60	1.0	1:12	88.41	88.56	88.49
30	40	0.5	1:12	87.88	88.02	87.95
30	50	0.5	1:8	90.41	90.34	90.38
30	50	1.5	1:12	89.44	89.91	89.68
15	40	1.0	1:12	89.68	89.74	89.71
60	50	1.0	1:12	89.98	91.44	90.71
15	50	1.5	1:6	89.07	87.19	88.13

Copper Corrosion. Copper corrosion was determined by using Herzog GmbH (model D130) according to ASTM D130.

NMR Analysis. ^{1}H and ^{13}C spectra were recorded in $CDCl_3$ with a Varian INOVA 500 spectrometer. Chemical shift values were recorded relative to the δ_H (7.24 ppm) and δ_C (77.0 ppm) signal of the predominantly deuterated solvent.

Heating Values. Heating values were determined using an IKA bomb calorimeter (model C-2000) according to ASTM D240.

RESULTS AND DISCUSSION

Optimization of the Transesterification Reaction. The best catalyst concentration (C), the oil−methanol mole ratio of (R), the best time (t), and temperature (θ) were carried out according to a full 3^4 factorial design. The amounts of sodium ethoxide used were (0.5, 1.0, and 1.5) g, and the oil-to-methanol mole ratio were 1:6, 1:8, and 1:12. Reaction times were (15, 30, and 60) min and temperatures (40, 50, and 60) °C.

Each test was replicated twice, and the response selected to test the yield of the transesterification reaction was monoglyceride concentration. Experimental results were worked out using analysis of variance (ANOVA), which allows evaluating whether the effect and the interaction among the investigated factors are significant with respect to the experimental error. The significance of the main factors and their interactions were assessed by the F-test method with a confidence level of 95 %.[29,30] RSM, a mathematical−statistical tool, was used for modeling monoglyceride concentration.

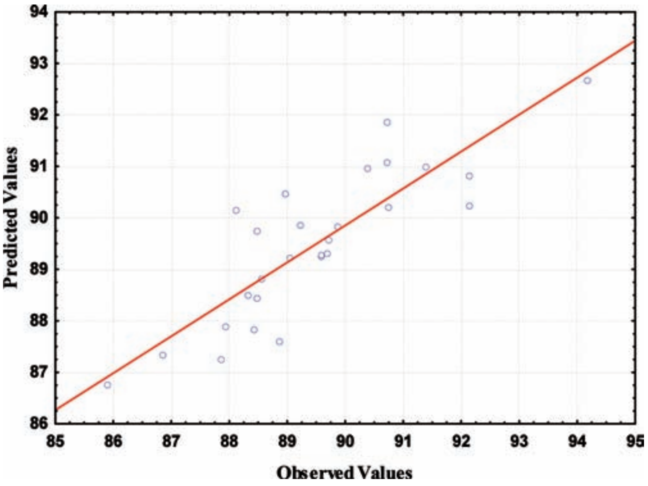

Figure 1. A comparative plot between experimental yield and predicted yield for catalyst mass fraction, oil−methanol mole ratio, temperature, and time.

Table 2 lists the conditions of all the tests developed by the full factorial design, in terms of both coded and noncoded variables. The best condition for biodiesel production was determined using ANOVA analysis. Each result is expressed as an arithmetic mean of two replications, and the conversion was determined by GC analysis.

Figure 1 shows the experimental values versus the predicted values using the model equation. Effects with a statistical significance lower than 95 % were not reported, according to the F-test utilized. Equations 1A, 1B, 1C, 1D, 1E, and 1F were used to predict the yield of the transesterification reaction, as a function of: time and temperature, time and catalyst mass fraction, time and oil−methanol mole ratio, temperature and catalyst mass fraction, temperature and oil−methanol mole ratio, and catalyst mass fraction and oil−methanol mole ratio, respectively.

$$\%\text{yield} = 38.0034 - 0.0428T + 2.1654\theta + 0.0018T^2 - 0.0021T\cdot\theta - 0.0212\theta^2 \tag{1A}$$

$$\%\text{yield} = 89.6648 - 0.1818T + 7.8322C + 0.0018T^2 + 0.0342T\cdot C - 4.9311C^2 \tag{1B}$$

$$\%\text{yield} = 87.7297 - 0.2003T + 1.2109R + 0.0018T^2 + 0.0061T\cdot R - 0.0797R^2 \tag{1C}$$

$$\%\text{yield} = 31.7202 + 2.1802\theta + 13.4383C - 0.0212\theta^2 - 0.0882\theta\cdot C - 4.9311C^2 \tag{1D}$$

$$\%\text{yield} = 30.2396 + 2.1581\theta + 1.8053R - 0.0212\theta^2 - 0.0076\theta\cdot R - 0.0797R^2 \tag{1E}$$

$$\%\text{yield} = 82.0463 + 7.3256C + 1.2271R - 4.9311C^2 + 0.1967C\cdot R - 0.0797R^2 \tag{1F}$$

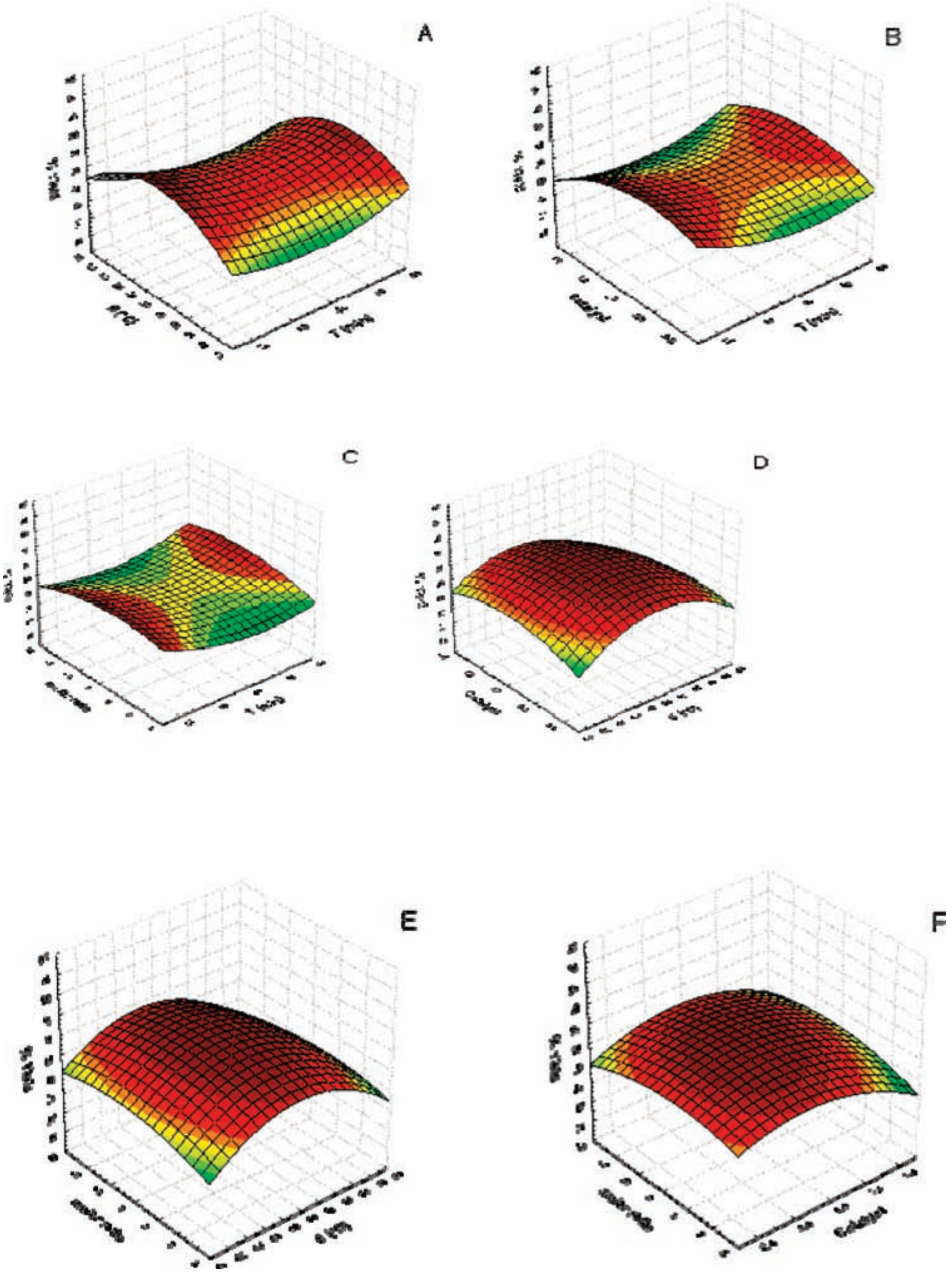

Figure 2. Surface response plots showing the effect of catalyst mass fraction, oil—methanol mole ratio, time and temperature on yield [yield as a function of: time and temperature (A); time and mass fraction (B); time and mole ratio (C); temperature and mass fraction (D); temperature and mole ratio (E); and mass fraction and mole ratio (F)].

where T, θ, C, and R are time, temperature, catalyst mass fraction, and oil—methanol volume fraction, respectively.

The interactions between parameters were also found to have a significant effect on the yield. Increasing the temperature decreases the reaction time, but temperatures above 60 °C cause a reduction in the yield because of parallel reactions. However, an increase in mole ratio causes an increase in yield, which does not occur with increasing of catalyst concentration due also to parallel reactions.

Having determined the best condition, the reaction of biodiesel production was realized under the following conditions: time, 40 min; temperature, 50 °C; oil—alcohol mole ratio, 1:9, and catalyst concentration, 0.9 %. The response surfaces obtained are shown in Figure 2.

The expected product was identified based on molecular weights using GC-MS. The results obtained showed that the supernatant of the reaction mixture consisted of biodiesel, and the major compound in the biodiesel was methyl linoleic esther (50.36 %) with a retention time of 64.745 min. The other esthers were methyl estearic esther (4.52 %), methyl palmitic esther (13.18 %), methyl oleic esther (29.44 %), and methyl linolenic esther (0.19 %) with retention time of (66.581, 57.157, 65.209, and 73.777) min, respectively (Figure 3).

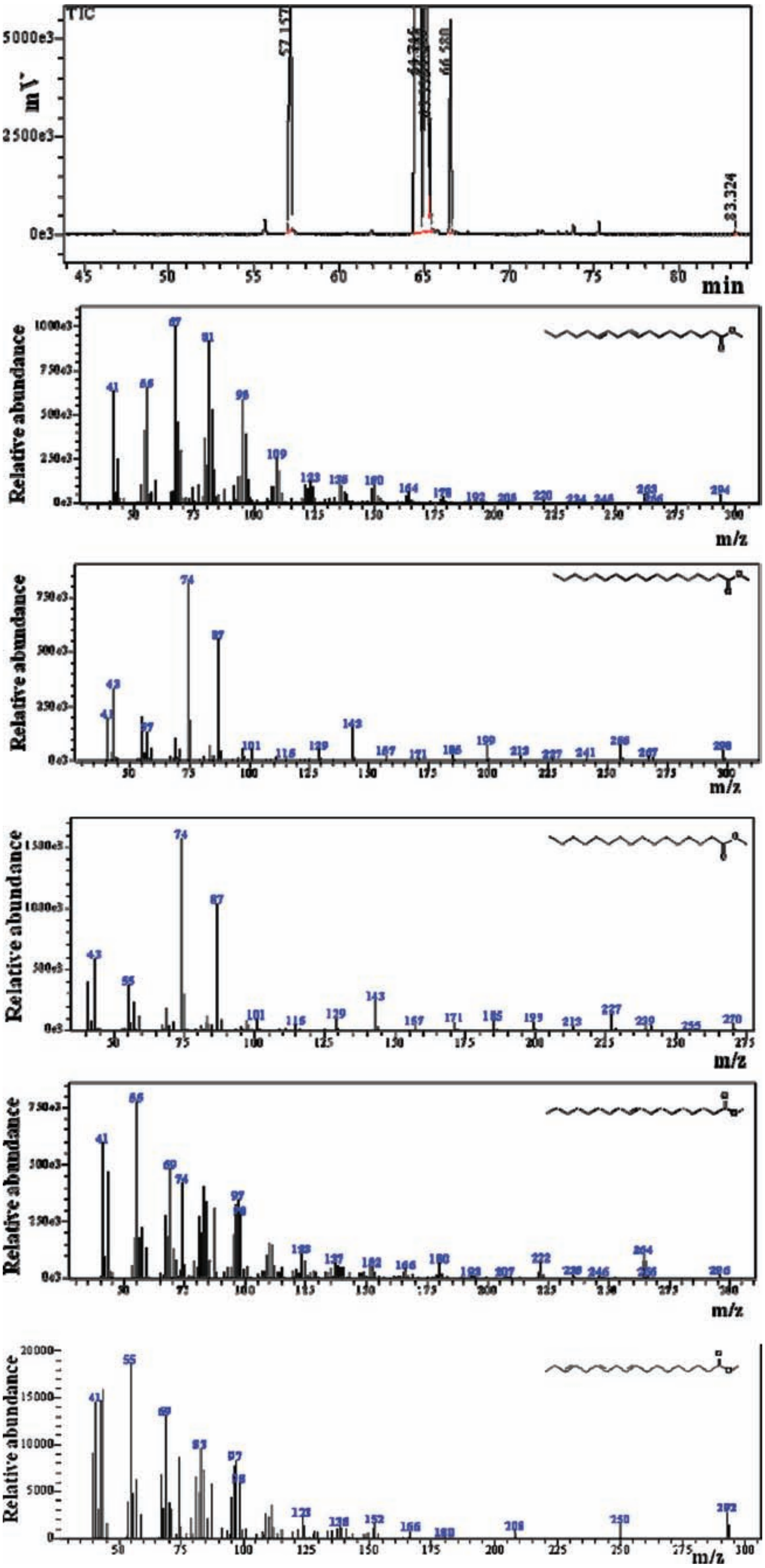

Figure 3. Chromatogram of biodiesel (min *X* mV) and mass spectra of methyl esthers [methyl linoleic esther (A), methyl stearic esther (B), methyl palmitic esther (C), methyl oleic esther (D), and methyl linolenic esther (E), respectively] (*m*/*z X* relative abundance).

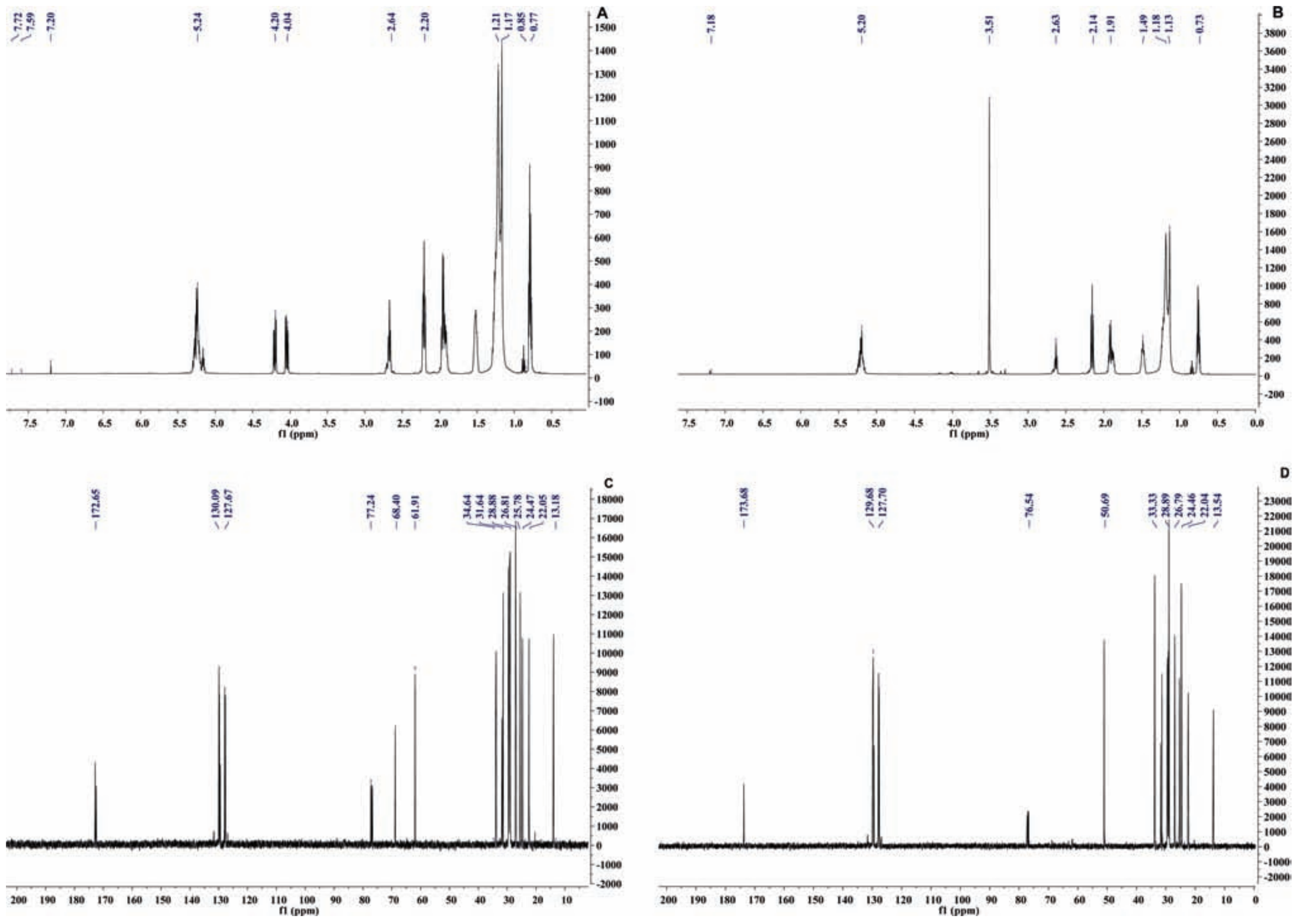

Figure 4. ^{1}H (A and B) and ^{13}C (C and D) NMR spectra of frying oil and biodiesel, respectively.

Table 3. Analysis of Biodiesel Produced at the Best Condition

properties	unit	ASTM standard	biodiesel
density at 20 °C	$g\cdot cm^{-3}$	D4052	0.88697
kinematic viscosity at 40 °C	$mm^2\cdot s^{-1}$	D7042	5.1480
heating value	$MJ\cdot kg^{-1}$	D240	40.456
copper-strip corrosion		D130	1A
conversion by GC-MS	%		98.70

The NMR spectra confirmed the conversion of frying oil to biodiesel. The ^{1}H spectra of biodiesel revealed a signal at δ 3.511 attributed to the hydrogens of the methoxyl group, and the disappearance of signals at δ 4.00 and 4.20 attributed to the hydrogens of glycerin groups, which were present in the ^{1}H spectra of the frying oil, hence confirming the conversion. NMR ^{13}C spectra revealed the signal at δ 50.999 attributed to the carbon of methoxyl groups (Figure 4).

Table 3 presents the characteristic of the biodiesel obtained under the optimized conditions.

Effect of Temperature on Density and Viscosity. *Density Measurements.* To study the effects of temperature on density and viscosity, blends were prepared at volume fractions from B2 (indicates 2 % of biodiesel with 98 % diesel) to B100 (100 % biodiesel).

Table 4. Experimental Blend Densities at Different Volume Fractions for All Investigated Temperatures

blend	$\rho/g\cdot cm^{-3}$				
	10 °C	20 °C	30 °C	40 °C	50 °C
B0	0.85409	0.84707	0.84006	0.83305	0.82659
B2	0.85529	0.84865	0.84158	0.83439	0.82727
B5	0.85679	0.85018	0.84309	0.83608	0.82889
B10	0.85889	0.85217	0.84508	0.83787	0.83079
B20	0.86327	0.85669	0.84947	0.84228	0.83509
B30	0.86568	0.85909	0.85187	0.84479	0.83767
B40	0.86959	0.86298	0.85568	0.84849	0.84128
B50	0.87348	0.86687	0.85959	0.85237	0.84519
B60	0.87717	0.87049	0.86328	0.85598	0.84878
B70	0.88109	0.87438	0.86719	0.85998	0.85279
B80	0.88497	0.87838	0.87109	0.86387	0.85659
B90	0.88887	0.88229	0.87497	0.86768	0.86049
B100	0.89389	0.88719	0.87987	0.87249	0.86518

The blend density was determined by using a densimeter manufactured by Anton Paar (model 4500). The densimeter was calibrated with air and water, and the densities were measured over the range of (10 to 50) °C with 10 °C intervals. The measured densities for the blends at several temperatures are presented in Table 4.

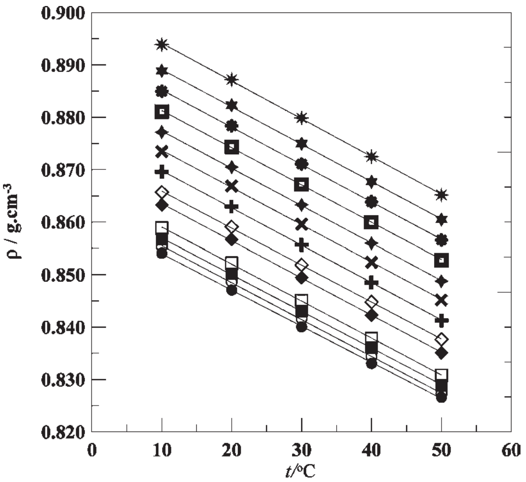

Figure 5. Density of (biodiesel + diesel) blend as a function of temperatures and at different volume fractions: ●, B0; ○, B2; ■, B5; □, B10; ♦, B20; ◇, B30; +, B40; ×, B50; four-pointed star, B60; ◘, B70; ∗, B80; ★, B90; eight-pointed star, B100. Solid lines are from eq 2.

Table 5. Linear Regression Parameters and Standard Deviations for Blend Densities According to Equation 2

	linear regression: $\rho/\text{g}\cdot\text{cm}^{-3} = a + bt/°\text{C}$			
	a	b		σ
blend	$\text{g}\cdot\text{cm}^{-3}$	$\text{g}\cdot\text{cm}^{-3}\cdot°\text{C}^{-1}$	R^2	$\text{g}\cdot\text{cm}^{-3}$
B0	0.860878	−0.0006902	0.999741	0.00018
B2	0.862526	−0.0007030	0.999780	0.00016
B5	0.863976	−0.0006990	0.999791	0.00016
B10	0.866110	−0.0007050	0.999848	0.00014
B20	0.870591	−0.0007077	0.999702	0.00019
B30	0.872916	−0.0007032	0.999781	0.00016
B40	0.876937	−0.0007111	0.999719	0.00019
B50	0.880824	−0.0007108	0.999719	0.00019
B60	0.884527	−0.0007129	0.999741	0.00018
B70	0.888386	−0.0007100	0.999809	0.00016
B80	0.892361	−0.0007127	0.999655	0.00021
B90	0.896271	−0.0007137	0.999649	0.00021
B100	0.901360	−0.0007212	0.999768	0.00020

Figure 5 shows the variation in densities of binary blends of produced biodiesel + diesel fuel as a function of temperature at different volume fractions. The density of produced biodiesel is higher than that of diesel. The results indicate that blend density decreases linearly with increasing temperature. The experimental data were correlated with temperature at different volume fractions by means of the following empirical linear equation:

$$\rho/\text{g}\cdot\text{cm}^{-3} = a + bt/°\text{C} \tag{2}$$

where ρ is density, a and b are coefficients, and t is temperature. An excellent agreement could be observed between the measured and estimated values. The constants a and b, the regression coefficients, R^2, and standard deviation, σ, are presented in Table 5.

Viscosity Measurements. Blend viscosity was determined by using a viscosimeter Stabinger manufactured by Anton Paar (model SVM 3000), and the viscosities were measured over the range of (10 to 50) °C with 10 °C intervals. The measured blend viscosities at several temperatures are presented in Table 6.

Table 6. Experimental Blend Viscosities at Different Volume Fractions for All Investigated Temperatures

	$\eta/\text{mPa}\cdot\text{s}$				
blend	10 °C	20 °C	30 °C	40 °C	50 °C
B0	5.0239	3.7805	2.9473	2.3609	1.9328
B2	5.1086	3.8363	2.9783	2.3839	1.9562
B5	5.241	3.9382	3.0601	2.4447	2.0077
B10	5.4395	4.0729	3.1631	2.5258	2.0719
B20	5.9088	4.413	3.4173	2.7234	2.2267
B30	6.2249	4.6417	3.5733	2.8506	2.3329
B40	6.6455	4.9452	3.8167	3.0359	2.4701
B50	7.1267	5.2885	4.0736	3.2324	2.6295
B60	7.6257	5.6427	4.3376	3.4335	2.7853
B70	8.2318	6.0656	4.6488	3.6671	2.9678
B80	8.8346	6.4869	4.9576	3.9154	3.1623
B90	9.5179	6.9341	5.2877	4.161	3.3585
B100	10.464	7.5343	5.7267	4.4917	3.6205

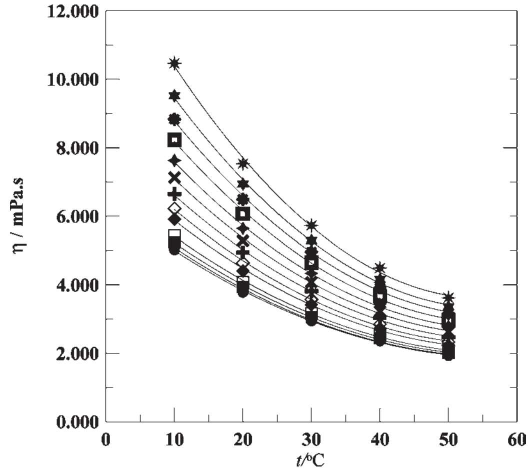

Figure 6. Viscosity of (biodiesel + diesel) blend as a function of temperatures and at different volume fractions: ●, B0; ○, B2; ■, B5; □, B10; ♦, B20; ◇, B30; +, B40; ×, B50; four-pointed star, B60; ◘, B70; ∗, B80; ★, B90; eight-pointed star, B100. Solid lines are from eq 3.

Figure 6 shows the variation in viscosity of the binary blends of produced biodiesel + diesel fuel as a function of temperature at different volume fractions. The viscosity of the produced biodiesel is higher than that of diesel. The results indicate that blend viscosity decreases nonlinearly with increasing temperature. The experimental data were correlated with temperature at different volume fractions by means of the following empirical second-degree equation:

$$\eta/\text{mPa}\cdot\text{s} = a + bt/°\text{C} + c(t/°\text{C})^2 \tag{3}$$

where η is viscosity, a, b, and c are coefficients, and t is temperature. An excellent agreement could be observed between the measured and the estimated values. The constants a, b, and c, the regression coefficients, R^2, and standard deviation, σ, are presented in Table 7.

Table 7. Second-Degree Equation Parameters and Standard Deviations for Blend Viscosities According to Equation 3

	polynomial equation: $\eta/\mathrm{mP{\cdot}s} = a + bt/{}^\circ\mathrm{C} + c(t/{}^\circ\mathrm{C})^2$				
	a	b	c		σ
blend	mPa·s	mPa·s·°C^{-1}	mPa·s·°C^{-1}	R^2	mPa·s
B0	6.42832	−0.1564780000	0.001341000000	0.998936	0.04007
B2	6.55622	−0.1612634286	0.001394857143	0.99902	0.03928
B5	6.72352	−0.165071000	0.001424500000	0.999072	0.03921
B10	6.98828	−0.1727330000	0.00149850000	0.998941	0.04360
B20	7.60398	−0.1891094286	0.001642857143	0.998917	0.04821
B30	8.03556	−0.2018952857	0.001769071429	0.999001	0.04901
B40	8.56906	−0.2147452857	0.001869071429	0.998835	0.05667
B50	9.20744	−0.2324035714	0.002031642857	0.998828	0.06125
B60	9.86726	−0.2504971429	0.002193284571	0.998782	0.06718
B70	10.67862	−0.273646286	0.002406357143	0.998736	0.07446
B80	11.48434	−0.2967167143	0.002625928571	0.998611	0.08406
B90	12.42056	−0.3258747143	0.002915928571	0.998404	0.09786
B100	13.73112	−0.3682788571	0.003349714286	0.998003	0.12161

■ CONCLUSION

A study on the optimization of the transesterification reaction for biodiesel production was carried out by means of statistical analyses using ANOVA. Biodiesel in this study was prepared through sodium ethoxide catalyzed methanolysis from the transesterification of recycled waste frying oil. Variance analysis showed the following optimum conditions for the reaction: a oil−methanol mole ratio of 1:9, temperature of 50 °C, catalyst mass fraction of 0.9 %, and reaction time of 40 min, which enabled a yield of 98.7 % by GC-MS analysis.

In addition, an investigation was performed on the effect of temperature on density and viscosity when diesel and biodiesel were blended. Results showed that the blend density increases with increasing biodiesel concentration at all investigated temperatures. A similar behavior was observed for viscosity. Densities decreased linearly with temperature, whereas viscosity decreased nonlinearly. Experimental data of the densities and viscosities were correlated with an empirical linear and polynomial equation, respectively, and the results presented excellent agreement between the measured and the estimated values.

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