APPLICATION OF FULL FACTORIAL DESIGN IN THE EVALUATION OF BIODIESEL PRODUCTION FROM MIXTURES OF CASTOR AND SOYBEAN OILS

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ABSTRACT

Blends of varying ratios of castor and soybean oils were used to produce biodiesel by alkaline transesterification using the methylic route. To increase the yield of esters, an experimental study using a full 2^4 factorial design was performed to evaluate the influence of methanol:oil molar ratio, KOH concentration, temperature, and reaction time on the biodiesel produced from each blend. Yields exceeding 95% were obtained, and the highest conversion was 99.05% at 25°C with a reaction time of 20 min using 2% KOH as a catalyst and a methanol:oil molar ratio of 12:1. To reduce process costs based on the amount of methanol used without limiting the conversion to esters, a second set of process conditions was identified, in which a 98.59% conversion to esters was obtained using the same temperature, reaction time, and catalyst concentration but a different methanol:oil molar ratio (6:1).

KEYWORDS

biodiesel; castor oil; soybean oil; transesterification; full factorial design

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doi:10.5419/bjpg2015-0008

1. INTRODUCTION

Given the global demand for energy sources with reduced environmental impacts, biomass has increasingly been used as a raw material for fuel production (Carvalho et al., 2010). In this context, biodiesel produced from vegetable oils represents an alternative fuel and has advantages over other sources: it is renewable, has low toxicity, and is biodegradable (Conceição et al., 2007; Wu et al., 2014). Additional characteristics, such as lubricity and burning efficiency, also contribute to the potential use of biodiesel as a fuel in diesel engines (Lôbo et al., 2009). Biodiesel is an oxygenated fuel produced from the catalytic transesterification of triglycerides, found in vegetable oils and animal fats, with an alcohol. The biodiesel produced in this reaction has to be purified by secondary processes, such as washing and drying. In addition to the type of catalyst used, this process is influenced by other factors, such as the choice of lipid source, the alcohol:oil ratio, the catalyst concentration, the temperature, and the reaction time (Mendonça et al., 2011; Carvalho et al., 2013).

Soybean oil (*Glycinemax L.*) is an important raw material for biodiesel production. In Brazil, more than 80% of the biodiesel is produced from soybean oil (Alberici et al., 2010). Although the biodiesel produced from soybeans is of adequate quality, the inclusion of other raw materials from different crops is important to reduce the dependence on a single oilseed, to improve product characteristics, to encourage the development of family farms, and to explore regional diversity of each producing country (Teixeira et al., 2009; Cesar and Batalha, 2010). In northeastern region of Brazil, particularly, the cultivation of castor (*Ricinus communis*) has increased because this crop is well adapted to semiarid regions, leading to large yields (Serra et al., 2011). Moreover, castor oil has specific characteristics that are attractive for biodiesel production.

Castor oil contains approximately 90% triglycerides of ricinoleic acid and has advantages over other oils in the transesterification reaction because of this oil's greater solubility in methanol and ethanol, which allows the reaction to be performed at lower temperatures and with reduced process times (Conceição et al., 2007; Serra et al., 2011). The biodiesel produced from castor oil is also oxidatively stable (Zuleta et al.,

2012). However, the use of the methyl or ethyl esters of ricinoleic acid as biodiesel is hindered by the presence of the hydroxyl group, which causes disadvantages, such as difficulty of water removal, low cetane index, high density and viscosity (Berman et al., 2011; Carvalho et al., 2013). For example, the high viscosity, which is typical even after the transesterification of the oil, can reduce the fuel atomization, causing the formation of deposits in the fuel injection system, thus, hindering the work of the fuel pumping system.

Although biodiesel blends, including mixtures of soybean and castor bean biodiesel, have been used to improve important fuel properties (Carvalho et al., 2013; Dantas et al., 2011; Gomes et al., 2010), less attention has been given to biodiesel production from vegetable oil mixtures (Meneghetti et al., 2007). An evaluation of the physicochemical characteristics of the oil to be transesterified during biodiesel production is important because the oil characteristics influence the quality of the biodiesel produced, the reaction conditions, and the yield of the reaction. High levels of acidity and moisture in vegetable oils, for example, reduce the yield of biodiesel in the reaction (Canakci & Gerpen, 2001). In addition, the specific gravity and viscosity of castor oil can affect operations in manufacturing plants due to difficulties in pumping and flow. Thus, adjusting the physicochemical parameters of the oil to obtain biodiesel within the quality specifications, as well as ensuring the use of a raw material with properties that are suitable for the operations in the production plant, is essential.

Because castor oil has high viscosity and density, studies of mixtures containing differing proportions of castor oil and other oils is a strategy that enables the production of biodiesel and allows the product to meet quality specifications (Barbosa et al., 2010). In the present study, blends containing different proportions of castor oil and soybean oil were prepared, and the suitability of their physicochemical characteristics for the production of biodiesel via transesterification was assessed. To evaluate the quality of the product, biodiesel was produced from mixtures of castor and soybean oils. After the castor oil:soybean oil ratios were defined, a full factorial design was used to evaluate the influence of the methanol:oil ratio, the catalyst concentration, the temperature, and the reaction time in the biodiesel production and reaction yield.

2. MATERIALS AND METHODS

2.1 Blend preparation and characterization

The mixtures of oils were prepared using refined, commercial, edible soybean oil and refined castor oil supplied by Bom Brasil (Salvador, Brazil). The castor and soybean oil blends were prepared in the proportions shown in Table 1. After homogenization, the physicochemical properties of these oil mixtures were analyzed using the official methods of the *American Oil Chemists Society* (AOCS) to characterize the raw material for biodiesel production (AOCS, 1998). Table 2 shows the tests used to characterize the oils.

2.2 Biodiesel production

The mixtures of castor and soybean oils (Table 1) were subjected to transesterification under reaction conditions previously established (Keera et al., 2011). All of the reactions were performed in a 2 L LabMax reactor (Mettler Toledo), equipped with mechanical agitation (350 RPM), temperature control, pH meter, automatic solution dispenser, and reflux condenser. Four hundred grams of oil were placed in the reactor, and the reaction

temperature was set at 60°C. Simultaneously, an alcohol solution of potassium hydroxide (1%) was prepared by dissolving the catalyst in methanol. The methanol:oil molar ratio used in the reaction was 6:1. The transesterification reaction was initiated with the addition of the potassium hydroxide solution to the oil under stirring. At the end of the reaction (1h), the mixture was incubated without stirring until phase separation between the biodiesel and glycerin was observed. The glycerin was separated and stored. The transesterification product that remained in the reactor was neutralized with a solution of ammonium chloride (5%) and thrice washed with distilled water for the removal of impurities. The washing process was repeated until the lower layer had a pH similar to the pH of the water, indicating that the product was free of catalyst. After washing, the biodiesel was dried at 100°C, weighed and stored for further characterization. The biodiesel was dried by heating up to 100 °C for half an hour to eliminate the remaining water after washing. The water in the biodiesel was evaporated during the drying process in the reactor. No degradation of the sample was observed in the drying process. Nine biodiesel samples were characterized to verify the suitability of the products based on the

Table 1. Composition of castor and soybean oil mixtures.

Blends	Castor oil (% v/v)	Soybean oil (% v/v)
1	0	100
2	2	98
3	5	95
4	10	90
5	20	80
6	40	60
7	60	40
8	80	20
9	100	0

Table 2. Methods employed to characterize castor and soybean oil mixtures.

Tests	Methods AOCS
Acid value / (mg KOH/ g of sample)	Cd 3d-63
Moisture and volatile matter / %	Ca 2b-38
Free fatty acids / oleic, %	Ca 5a-40
Peroxide value	Cd 8-53
Saponification value	Cd 3-25
Iodine value	Cd 1-25

Table 3. Levels and factors for the 2^4 factorial design applied to the evaluation of the transesterification reaction of the mixture containing 20% castor oil and 80% soybean oil.

Factors	Levels		
	-1	0	+1
Temperature, T / °C	25	45	65
Reaction time, t / min	20	55	90
Catalyst concentration, C / %	1	1,5	2
Methanol:oil molar ratio, M:O	6:1	9:1	12:1

Table 4. Methods employed to characterize the biodiesel samples.

Tests	Methods
Specific gravity at 20°C / kg m ⁻³	ASTM D4052
Kinematic viscosity at 40°C / mm ² s ⁻¹	ASTM D445
Water content / mg kg ⁻¹	ASTM D6304
Cold filter plugging point / °C	ASTM D6371
Acid value / (mg KOH/g oil)	ASTM D664
Corrosiveness to copper	ASTM D130
Flash point / °C	ASTM D93
Oxidative stability / h	EN 14112
Content of mono-, di- and triacylglycerols / %	ASTM D6584/NBR 15324
Ester content / %	EN 14103

specifications and to select one blend for further experiments. The selected blend was subjected, through an experimental design, to a series of transesterification reactions under various process conditions to optimize the reaction, considering the influence of the process temperature, reaction time, catalyst concentration, and molar ratio of alcohol and oil.

2.3 Experimental design of transesterification reaction

The blend composed of 20% castor oil and 80% soybean oil was selected for evaluation of the conditions for obtaining biodiesel using a full factorial design. The transesterification experiments were performed using 500 g of the oil mixture in the reactor according to the described procedure. The reaction variables studied were defined as factors (k) and included the methanol:oil molar ratio (M:O), the concentration of the KOH catalyst (C, %), the reaction time (t, min), and the system temperature (T, °C). Maximum and minimum values for each factor were chosen in accordance with previously reported data. The

response evaluated was the final yield (E, %). The experimental data were processed using the software, *Statistica*. Through a full factorial design, the influence of the four factors ($k = 4$) at two levels ($n = 2$) for the methanolysis of the mixture of soybean and castor oils via basic catalysis was evaluated. The total number of experiments required for this experimental design was $n^k = 2^4 = 16$. Additionally, four replicates were performed at the central point to evaluate the experimental error, for a total of 20 experiments. The factors used in the experiments are shown in Table 3. The 20 reactions were performed in random order.

2.4 Biodiesel characterization

The biodiesel samples obtained from the transesterification reactions of the castor and soybean oil mixture were analyzed according to the methods shown in Table 4 (ASTM, 2014; EN 14112, 2003; EN 14103, 2011). Specific gravity tests were performed using a digital densimeter (KEM, model DA-640). The kinematic viscosity was measured using an automatic viscometer (Herzog, model HVU 481). The water content of the biodiesel was

obtained using a titrator (Schott Instruments, Titroline KF Trace). The cold filter plugging point (CFPP) was obtained using an automated CFPP tester (Tanaka Scientific, model AFP-101). A thermostatic bath (Petrotest DP) was used to measure the corrosiveness to copper. The flash point was analyzed with an automated flash point tester (Tanaka, model APM-7). The oxidation of the samples was analyzed using a 743 Rancimat (Metrohm). The tests of the ester, monoacylglycerol, diacylglycerol, and triacylglycerol contents of the sample were performed using a gas chromatograph (Varian, model CP-3800 GC), equipped with a capillary injection system.

3. RESULTS AND DISCUSSIONS

The blends of castor and soybean oils were characterized for acid value, water content, free fatty acids, peroxide value, saponification, and iodine value, and the results are shown in Table 5. Table 5 shows an increase in the acid value and in the concentration of free fatty acids in the blends containing 60% or more castor oil (blends 7, 8, and 9). Free fatty acids must be present at low concentrations (less than 3%) for biodiesel production by the transesterification reaction with alkaline catalysis (Kumar et al., 2013); when hydroxides are used as catalysts, an excess of free fatty acids could lead to saponification reactions that compete with the transesterification reaction. All of the blends obtained contained less than 1% free fatty acids; thus, pre-treatment to adjust the acidity of the raw material was not necessary.

The peroxide value slightly increased with an increase in the castor oil content of the blends, as observed in Table 5. Products with peroxide values between 1 and 5 are classified as being in a low oxidation state (O'Brien, 2004). The maximum peroxide value obtained for the samples was 3.5, which did not exceed the recommended limit.

An increase in the castor oil content of the blends did not significantly influence the saponification value when the castor oil content was less than 20% (blends 1, 2, 3, 4, and 5). A large increase in saponification value was observed in blend 6, which contained 40% castor oil.

The results shown in Table 5 indicate that the moisture content increased with an increase in the percentage of castor oil. The sample with 100% castor oil (blend 9) had a higher moisture value than the other blends. Despite this castor oil concentration-dependent increase, all of the blends had a low moisture content that was within the acceptable limits for transesterification reactions (Banerjee & Chakraborty, 2009).

A decrease in the iodine value was observed when the percentage of castor oil in the blends increased (Table 5). This behavior is most likely due to the higher degree of unsaturation of the soybean oil (predominantly linoleic acid with conjugated double bonds) than that of the castor oil (ricinoleic acid with a hydroxyl group, double bonds are prevalent). Fatty acids with greater extents of unsaturation have a greater capacity for iodine absorption and, consequently, a higher iodine value (Deshpande & Kullarni, 2012).

Table 5. Results of the characterization of the castor and soybean oil blends.

Parameter	Blend								
	1	2	3	4	5	6	7	8	9
Acid value/(mg KOH/g of sample)	0.33	0.30	0.29	0.30	0.24	0.30	0.79	1.40	1.75
Moisture and volatile matter / %	0.004	0.01	0.01	0.01	0.01	0.01	0.01	0.02	0.04
Free fatty acids (oleic / %)	0.17	0.15	0.16	0.15	0.12	0.15	0.40	0.70	0.88
Peroxide value	2.2	2.2	2.3	2.1	2.4	2.5	2.6	2.5	3.5
Saponification value	103	102	103	99	102	118	119	116	121
Iodine value	125	120	116	116	126	107	111	78	79

Table 6. Results of the characterization of the biodiesel samples obtained through transesterification of castor and soybean oil blends.

Parameter	Blend									ANP
	1	2	3	4	5	6	7	8	9	Specification
Specific gravity at 20°C / kg/m ³	884	884	886	892	886	892	903	915	926	850 - 900
Kinematic viscosity at 40°C / mm ² s ⁻¹	4.5	4.5	4.3	4.4	5.3	7.4	9.8	11	16	3.0 - 6.0
Water content / mg kg ⁻¹	80	130	140	160	240	310	330	340	360	200, max.
Cold filter plugging point / °C	-3	-4	-4	-4	-5	-7	-7	-9	-13	19, max.
Corrosiveness to copper	1	1	1	1	1	1	1	1	1	1, max.
Flash point / °C	171	172	173	174	174	184	196	179	199	100, min

The biodiesel samples obtained through transesterification of the oil blends (Table 1) were characterized to verify that they met the specifications of the National Agency of Petroleum Natural Gas and Biofuels (ANP, 2014). No significant variation in the density of the biodiesel was observed with the addition of up to 40% castor oil (blends 1-6), as shown in Table 6, and the densities of the biodiesel produced were within the quality standards range (850-900 kg/m³). The kinematic viscosity of the biodiesel samples varied more significantly in blends 5-9 (containing 20% or more castor oil). However, only biodiesel samples produced with blends containing more than 40% castor oil did not comply with the ANP specifications for viscosity. An increase in the castor oil content caused an increase in the water content, but even the biodiesel produced from the blend containing 20% castor oil (blend 5) met the specifications. This fact can be explained by the higher humidity of castor oil compared to soybean oil and the presence of the hydroxyl group in the ricinoleic acid that hinders the removal of water.

Increases in the concentration of castor oil in the blends caused a decrease in the cold filter plugging point and an increase in the flash point in the biodiesel samples; however, all of the samples met the specifications for these two parameters. No variation in the corrosiveness to copper was observed in any of the samples.

The parameters evaluated in the samples containing up to 20% castor oil met the specifications. The biodiesel produced from pure castor oil and the biodiesels obtained from mixtures containing more than 20% castor oil did

not meet the specifications, particularly density and viscosity specifications. In this sense, the production of biodiesel from castor bean and soybean oils is not recommended when the ratio of the first is greater than 20%. To maximize the amount of castor oil in the mixtures while maintaining satisfactory biodiesel production, the blend with 80% soybean oil and 20% oil castor oil was chosen as the raw material to be used in the evaluation of the process conditions. This evaluation was performed with the aid of a 2⁴ full experimental design.

The matrix of the 2⁴ full factorial design and the results of the performance experiments for the 20 reactions are shown in Table 7. The highest yield of 99.05% was obtained under the following reaction conditions: temperature of 25°C, reaction time of 20 min, 2% KOH as the catalyst and a methanol:oil molar ratio of 12:1 (experiment 13). Goyal et al. (2012) optimized the conversion of high free fatty acid *Jatropha curcas* oil to biodiesel using response surface methodology and obtained yield of 98.3% with methanol/oil ratio (11:1) using NaOH as catalyst (1% w/w) in 110 min time at 55°C temperature. Jeong and Park (2009) further investigated the effect of reaction temperature, oil-to-methanol molar ratio, and catalyst quantity (KOH) on biodiesel yield from castor oil and obtained about 92% yield at 40-min reaction, 35.5 °C, with methanol/oil ratio of 8:1, and a catalyst concentration of 1.45% of KOH by weight. Cavalcante et al. (2010) studied the synthesis of fatty acid esters from castor oil using an alkaline catalyst. The highest yield (86.32%) was obtained using an ethanol:oil: molar ratio of 11:1, 1.75% KOH and a reaction time of 90 min. By comparing

Table 7. Matrix and results of the $2^4 (n^k)$ full factorial design.

Experiment	T	t	C	M:O	Yield / %
1	-1	-1	-1	-1	95.93
2	+1	-1	-1	-1	98.01
3	-1	+1	-1	-1	97.66
4	+1	+1	-1	-1	98.15
5	-1	-1	+1	-1	98.59
6	+1	-1	+1	-1	98.15
7	-1	+1	+1	-1	98.66
8	+1	+1	+1	-1	98.20
9	-1	-1	-1	+1	98.74
10	+1	-1	-1	+1	97.11
11	-1	+1	-1	+1	99.03
12	+1	+1	-1	+1	97.83
13	-1	-1	+1	+1	99.05
14	+1	-1	+1	+1	98.24
15	-1	+1	+1	+1	98.97
16	+1	+1	+1	+1	98.00
17	0	0	0	0	97.91
18	0	0	0	0	98.59
19	0	0	0	0	98.66
20	0	0	0	0	98.64

T = Temperature ($^{\circ}\text{C}$), t = Reaction time (min), C = Catalyst (%), and M/O = Methanol to oil ratio.

the present results with the literature, it is apparent that there has been an optimization of process conditions in relation to time, process temperature, and biodiesel yield.

The full factorial design allowed the verification of the significance of each studied parameter and their interactions through an analysis of variance (ANOVA) using the p value. Using the Pareto chart (Figure 1), the temperature and methanol:oil molar

Figure 1. Pareto chart used in the evaluation of temperature (T), reaction time (t), catalyst concentration (C) and methanol:oil molar ratio (M/O) effects in the transesterification reaction of the castor oil and soybean oil mixture. TxM/O, txC, TxC, CxM/O, txM/O are second-order interaction factors.

Figure 2. Response surfaces. Yield (E, %) as a function of (a) Time and temperature; (b) Catalyst concentration and temperature; (c) Oil:methanol molar ratio and temperature; (d) Catalyst concentration and time; (e) Oil:methanol molar ratio and time; (f) Oil:methanol molar ratio and catalyst concentration.

ratio interaction (TxM/O) was found to have the greatest influence on the reaction system. The combination of these parameters had a negative effect on the conversion of esters. On the other hand, catalyst concentration (C) has shown to have a significant positive effect on reaction yield. This means that raising the values of this parameter from the lowest (−1) to the highest (+1) level leads to an increase on the final yield.

The curvature analysis was not statistically significant at a 95% confidence level, suggesting the possibility of employing a linear mathematical model to represent the yield within the experimental domain studied. The response

surfaces for the linear model are shown in Figure 2. The use of response surface methodology (RSM) allowed the visualization of the influence of two variables simultaneously and the region of best ester yield. With this experimental design, the highest yield of biodiesel from castor/soybean oil (99.05%) was obtained in experiment 13. In this experiment, the following conditions were used: reduced temperature (25°C) and reaction time (20 min), a catalyst concentration of 2%, and a methanol:oil molar ratio of 12:1.

To reduce the process costs relative to the amount of methanol used without affecting the yield of the reaction, experiment 5 was identified

Table 8. Characterization of the biodiesel sample obtained from the blend of 20% castor oil and 80% soybean oil.

Tests	Results	ANP Specification
Specific gravity at 20°C / kg m ⁻³	889	850 - 900
Kinematic viscosity at 40°C / mm ² s ⁻¹	4.9	3.0 - 6.0
Water content / mg kg ⁻¹	201	200, max.
Cold filter plugging point/ °C	-5.5	19, max.
Acid value / (mg KOH/g oil)	0.34	0.50, max.
Corrosiveness to copper	1	1, max.
Flash point / °C	179	100, min.
Oxidative stability / h	7	6, min.
Ester content / %	98	96.5, min.
Monoacylglycerol content / %	0.54	0.70, max.
Diacylglycerol content / %	0.11	0.20, max.
Triacylglycerol content / %	0.12	0.20, max.

Reaction conditions: temperature, 25°C; reaction time, 20 min; catalyst concentration, 2%; methanol:oil molar ratio, 6:1.

as an alternative to experiment 13. Experiment 5 used a methanol:oil molar ratio of 6:1, with a resulting yield of 98.59%. To verify whether the methyl esters produced in these process conditions are within the specifications for biodiesel, as required by the ANP, sample 5 was characterized. According to the results shown in Table 8, the sample was within the quality standards established by ANP for the parameters evaluated.

4. CONCLUSIONS

Castor and soybean oil mixtures can be used to change the properties of raw materials to obtain oils with characteristics that are more favorable to biodiesel production. A biodiesel that met relevant specifications was obtained using mixtures containing up to 20% castor oil. The use of a full factorial design allowed the evaluation of the influence of reaction variables and the establishment of appropriate reaction conditions for a mixture composed of 20% castor oil and 80% soybean oil. The product of the transesterification reaction was analyzed and the results indicated that the obtained biodiesel was of adequate quality, demonstrating that the preparation of blends of different raw materials is an interesting approach to ensure adequate oil for the transesterification reaction and biodiesel production.

ACKNOWLEDGEMENTS

The authors are grateful to the Research Foundation of the State of Bahia (Fundação de Amparo à Pesquisa do Estado da Bahia - FAPESB) and the National Council for Scientific and Technological Development (Conselho Nacional de Desenvolvimento Científico e Tecnológico - CNPq) for financial support.

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