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### DIFFERENT PURIFICATION METHODS AND QUALITY OF SUNFLOWER BIODIESEL

A.L.M.T. PIGHINELLI<sup>1</sup>, R.A. FERRARI<sup>2</sup>, A.M.R.O MIGUEL<sup>2</sup>, K.J. PARK<sup>1</sup>

<sup>1</sup>A.L.M.T. PIGHINELLI, PhD student, School of Agricultural Engineering, State University of Campinas (UNICAMP), P.O. Box 6011, 13083-875 Campinas, São Paulo, Brazil, annalets@feagri.unicamp.br.

<sup>2</sup>R.A. FERRARI and A.M.R.O MIGUEL, Food Technology Institute (ITAL) Av. Brasil 2880 PO Box 139 CEP 13070-178 Campinas-SP Brazil.

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**ABSTRACT** Biodiesel is a fuel obtained from triacylglycerides and mostly produced through transesterification, a chemical reaction of vegetable oils with alcohol, methanol or ethanol. Raw material selection should take the costs into account; because 85% of production cost is related to vegetable oil. The objective of this study was to evaluate oil expression of sunflower seed (*Helianthus annuus* L.), the study of sunflower crude oil as raw material for biodiesel by transesterification reaction, in laboratory and pilot scales, and three different biodiesel purification methods. For the best conduction of experiments, an experimental design was used. The best result for oil expelling, 68.4 %, was obtained with screw speed of 114 rpm and for seeds with 25 oC and 6.9 % of moisture content. For biodiesel production at laboratory scale, the best result, 87.5 %, was obtained with molar ratio of 4.7:1 (ethanol:oil) and 4.42 % of catalyst concentration related to the quantity of oil that had to be transesterified. That experimental condition was applied in bigger scale with a batch stirred tank reactor. For purification with washing, the biodiesel yield was 84.2 %. Purification with silica had a yield of 84.6 % and distillation, 92.3 %. Distillation of biodiesel produced a biofuel with better quality in relation to the technical limits of the Brazilian standard.

**Keywords:** Crude oil, Transesterification, Ethanol, Washing, Silica, Distillation.

**INTRODUCTION** Biodiesel has been more intensively researched due to its great importance in the global scenery, as it is considered the most suitable substitute for diesel. In order to align its production to sustainability concepts, it is necessary to reduce its cost of production, since 85 % of that is owed to the main raw material: vegetable oils. According with data from Brazilian National Petroleum, Natural Gas and Biofuels, the total of biodiesel produced in Brazil between the years of 2005 and 2009 was 2.9 billion of liters (ANP, 2008). Biodiesel is chemically defined as the simple alkyl monoesters of long chain fatty acids derived from renewable feedstocks such as vegetable oils. The most widely adopted process for producing biodiesel consists of a chemical reaction in which the oil triacylglycerols are reacted with an alcohol (methanol or ethanol) using an alkaline catalyst (usually NaOH or NaOme) to produce simple alkyl monoesters (biodiesel) and glycerol. The glycerol is recovered by gravity and/or centrifugation, and

is a valuable co-product (Knothe, 2005). Ethanol is the most viable alcohol, since it is produced from renewable sources (Encinar et al., 2007) but ethylic transesterification may pose some inconveniences like: ethyl esters formation is more difficult than that of methyl ester, since it produces stable emulsions that complicate esters separation and purification (Kucek et al., 2007). After the recovered of glycerol by gravity, the biodiesel is purified, eliminating contaminants like free glycerol, soaps, metals, alcohol and catalysts (Cooke, 2007). The most applied treatment to purify biodiesel is washing with an acid solution and other purification methods are rarely found in literature (Predojevic, 2008). Response surface methodology (RSM) is a powerful tool for the optimization of chemical reactions. The main advantages of this method include an understanding of how the test variables (process variables) affect the selected process response (Barros Neto et al., 2003). The aim of this study was to evaluate the influence of temperature and presser rotation on crude oil yield, as well as to examine the reaction conditions in molar ratio (between ethanol and vegetable oils) and the catalyst concentration at the crude sunflower oil transesterification. It was also evaluate three purification methods and current Brazilian biodiesel standard was used as a reference for biodiesel quality control.

## METHODOLOGY

**Obtaining and processing the raw material** The sunflower seeds were acquired at a local business. The seeds employed were identified as the species *Helianthus annus* L. var. IAC-Iarama. The grains were analyzed as for the moisture (AOAC, 2007) and oil contents (AOCS, 2008). After the characterization, the grains were conducted to Pilot Oil Plant from Food Technology Institute (ITAL) and the oil extraction was performed. The sunflower seeds were selected, removing any foreign material or deteriorated grain that could affect the oil extraction. Five kilograms of seeds were used in each experimental run. The heating was done in a cooker built up from the drum of a concrete truck connected to a gas heater. Temperatures were measured with an infrared thermometer (BT TIP 400, resolution 0,1°C). Pressing process was performed with a stainless steel expeller press with capacity to press 40 kg of grains per hour (Figure 1).

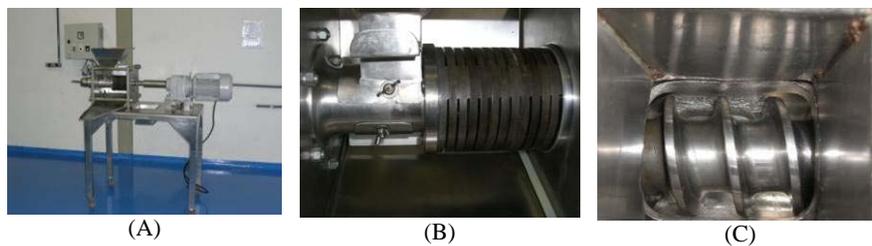


Figure 1. Expeller equipment.

A  $2^2$  factorial experimental design with four factorial points, four axial points and three central points, totalizing 11 experiments, was adopted to optimize the oil extraction. Two independent variables were evaluated: press speed (R) and grain temperature (T). The response is summarized as oil yield (OY). A second order mathematical model (Equation 1) was used to analyze the effects of the independent variables on the oil yield using the software Statistica 5.0. Table 1 presents the real values corresponding to the codified levels of the experimental design. The levels chosen for each process variables were based on preliminary studies carried out in our laboratory with other vegetable oils (Pighinelli et al., 2009; Pighinelli et al., 2008; Pighinelli et al, 2007). All the eleven oil

samples were filtrated, homogenized and characterized. The oil sample was analyzed as for: (a) Acid number, AOCS Ca 5a - 40 (2008), (b) Fatty acid composition, AOCS, Ce 1e - 91; Ce 1F - 96; Ce 1-62 e Ce 2-66 (2008) and AOAC 996.06 (2007), (c)Moisture content and volatile matter, AOAC (2007).

$$OY = b_0 + b_1R + b_2T + b_{11}R^2 + b_{22}T^2 + b_{12}RT \quad (1)$$

b: regression coefficient

1: linear effect of the rotation

2: linear effect of the temperature

11: quadratic effect of the rotation

22: quadratic effect of the temperature

12: interaction effect between R and T

Table 1. Experimental design for oil expelling – real level values.

Variables	Codified levels				
	-1.41	-1,0	0	1.0	1.41
Rotation (rpm)	85	90	102	114	119
Temperature (°C)	25	30	42.5	55	60

**Laboratory transesterification** Transesterification reactions were done with anhydrous ethanol and a sodium methylate solution (30%) as a catalyst. The same experimental design used for oil extraction was used for transesterification reaction at laboratory. Two independent variables were evaluated: molar ratio ethanol:oil (MR) and catalyst concentration related to the quantity of oil that had to be transesterified (C). The response is summarized as unpurified biodiesel (BY). Table 2 presents the real values corresponding to the codified levels of the experimental design. The ethanol mass used in each experimental point was calculated according to the oil mean molecular mass, and the mass ranged between 4.1844 and 20.9220g. Initially, 25 g of sunflower crude oil was added to the flask, which was heated to 45°C. In another flask, a specified amount of sodium methylate was added to a predetermined amount of anhydrous ethanol. The resulting solution was added to the pre-heated oil and stirred for 30 min. Afterwards, the glycerin phase was recovered immediately after phase separation. Analysis of the ethyl ester phase was carried out by chromatograph liquid Waters 600E with refraction index detector Waters 410 and equipped with two columns in series (Jordi Gel DVB 300 mm x 7.8 mm of 500 Å and 100 Å), 50 minutes running time, 40°C injector and column temperatures, 0.5 ml flow rate by minute and 20µL injected sample volume. Quantification was based on external calibration using standard solutions of triacylglycerols, diacylglycerols, monoacylglycerols, free fatty acids and commercial biodiesel. Biodiesel samples were prepared in THF (tetrahydrofuran) to a final concentration of 1 mg/mL.

Table 2. Experimental design for transesterification – real level values.

Variables	Codified levels				
	-1.41	-1,0	0	1.0	1.41
Molar ratio	3.0	4.7	9.0	13.3	15.0
Catalyst conc. (wt %)	1.00	1.58	3.00	4.42	5.00

**Reactor transesterification** The best experimental condition obtained in laboratory scale was applied in a batch stirred tank reactor (Figure 2). The reactor was built with financing of The State of Sao Paulo Research Foundation (FAPESP 2006/59907-1). The reactor's structure was made with stainless steel plates, with a conical bottom, 25 liters of capacity and a heating system with indirect steam, as presented in Figure 1A. The mixture was stirred with a system connected to a gearmotor SEW and a frequency inverter to adjust the rotation. Temperature was adjusted by a thermometer, as shown in Figure 1B. It was

used 15.0 kg of sunflower crude oil, 3.9 kg of anhydrous ethanol and 0.6 kg of sodium methylate solution (30%). Chemical reactions were carried out at 45°C for 30 min. Initially, the mechanical stirrer was set at 580 rpm, until the oil has reached the temperature. After the addition of catalytic solution, the rotation was decreased to 415 rpm and this rotation remained until the conversion in biodiesel occurred. For the final stage of the reaction, a 580 rpm was applied. The adjustment of rotation took into account the mix intensity that allowed to chemical reagents and oil an adequate blend, favoring the mass transfer during the transesterification reaction and avoiding the excessive incorporation of air during the process. Later, the mixture was lead to decant for 6 hours to allow the separation of glycerol and unpurified biodiesel. Any excess ethanol in biodiesel layer was evaporated under reduced pressure at temperature < 60°C. The biodiesel was purified and characterized.

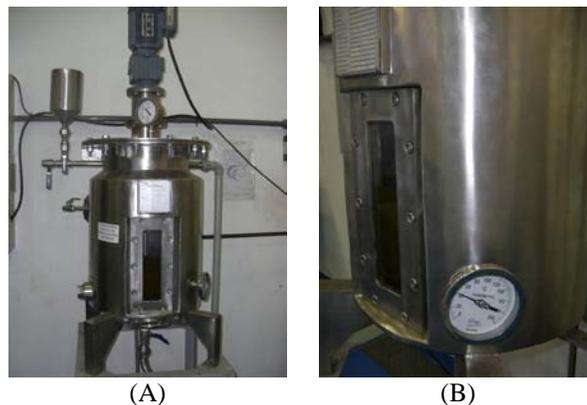


Figure 2. Batch stirred tank reactor (A), Thermometer (B).

**Purification studies** Three purification methods were studied to evaluate their efficiency concerning to biodiesel quality. It was calculated the yield of each kind of purification, comparing the volume of unpurified biodiesel and the volume of biodiesel produced. The biodiesel characterization was carried out in relation to the technical limits of the Brazilian standard (Resolution 7 of the national Petroleum, Natural Gas and Biofuels, ANP, dated March, 2008), using the following methods: ABNT NBR 7148 for specific gravity at 20°C (kg/m<sup>3</sup>), ASTM D 6304 for water content (mg/kg), ABNT NBR 14598 for flash point (°C), ABNT NBR 14359 for copper corrosion for 3 h at 50 °C, ASTM D 6584 for total glycerin (wt%), free glycerin (wt%) and acylglycerides (wt%), ASTM D 6371 for cold filter plugging point (°C), ASTM D 4530 for carbon residue (wt%), EN ISO12662 for total contamination (wt%), EN 14103 for total ester content (wt%), EN 14110 for ethanol content (wt%), ASTM D 1298/4052 for kinematic viscosity at 40°C (mm<sup>2</sup>/s), ASTM D874 for sulfated ash (wt%), ASTM D4294/5453 for total sulfur content (wt%), ASTM D664 for acid number (mg KOH/g), visual aspect, EN 14538 for calcium, magnesium, sodium and potassium content, EN 14107 for phosphorous content, EN 14111 for iodine number and EN 14112 for oxidation stability (BRASIL, 2008b).

The first purification method consisted in washing the biodiesel with 3 liters of 5% w/w solution of hydrochloric acid (37%), heated at 90°C (FERRARI et al., 2005). The second purification method was a technique named “dry washing”, using 2 wt% of silica (GRACE DAVISON TRISYL® 300B 97%) and 1.6 wt% of auxiliary filter (Celite™), in relation to unpurified biodiesel mass. Purification was conducted at 90°C, under mechanical stirrer and reduced pressure, for 30 min. Distillation was the last purification

method evaluated. A flask was adapted to a bath and a reflux condenser, under pressure of 680 mmHg.

## RESULTS AND DISCUSSION

**Raw material processing** Moisture content in the grain was 7.5% and lipids content was 46.8%. The oil mass obtained in the different trials varied from 51.70% to 68.38%, as shown in Table 3. The best result was associated with test number 3, in which the press speed was set at 114 rpm and the temperature at 30.0 °C. In contrast, the lowest oil mass results corresponded to the test 5, the speed rotation set at 85 rpm and the temperature at 42.5 °C. These results indicated a clear tendency to increase the oil mass with reduction in the temperature and increase in the pressing speed. To verify the effect of each variable on the evaluated response, the oil extraction was optimized using the software Statistica 5.0 and the data presented in Table 3. The adjustment coefficients of the experimental data to the mathematical model represented by Eq. (1) are presented in Table 4.

Table 3. Average oil yield corresponding to the pressing of sunflower seed based on a 2<sup>2</sup> factorial experimental design, with four axial and factorial points and three central points.

Test	Variables		Oil yield (wt%)
	Press speed (rpm)	Temperature (°C)	
1	90 (-1)	30.0 (-1)	54.77
2	90 (-1)	55.0 (+1)	61.49
3	114 (+1)	30.0 (-1)	68.38
4	114 (+1)	55.0 (+1)	64.38
5	85 (-1.41)	42.5 (0)	51.70
6	119 (+1.41)	42.5 (0)	62.72
7	102 (0)	25.0 (-1.41)	61.11
8	102 (0)	60.0 (+1.41)	64.85
9	102 (0)	42.5 (0)	68.04
10	102 (0)	42.5 (0)	66.13

The results shown in Table 4 demonstrate that the coefficients  $b_0$ ,  $b_1$  and  $b_{11}$  were considered statistically significant at a 90% confidence level. This implies that amongst the variables considered in this study, only the pressing rotation showed a statistically significant effect on the oil mass in the interval considered. The positive value of the  $b_1$  coefficient indicates that the oil mass showed a tendency to increase as the rotation increased, independent of the grain temperature. Based on these results, the codified mathematical model representing the oil extraction (Eq. (1)), can be reduced to a quadratic model containing three parameters Eq. (2).

Table 4. Regression coefficients associated with the codified mathematical models for oil extraction.

Regr. coefficient	Effect	Standard deviation	p
$b_0$	<i>67.0720</i>	<i>0.9574</i>	<i>0.0091</i>
$b_1$	<i>8.0366</i>	<i>0.9589</i>	<i>0.0756</i>
$b_{11}$	<i>-8.8110</i>	<i>1.2714</i>	<i>0.09123</i>
$b_2$	2.0068	0.9589	0.2838
$b_{22}$	-3.0106	1.2714	0.2544
$b_{12}$	-5.3617	1.3540	0.1675

Values marked in italics are statistically significant coefficients at a level of 90%.

$$OY = 65.3410 + 7.9824R - 7.3836R^2 \quad (2)$$

The analysis of variance was performed to verify the validity of the models represented by Eq. (2) and the results are presented in Table 5. The results of the analysis of variance associated with the oil expelling indicate that the F value between the regression and the residues is almost 4 times higher than the F value shown in the table. The results also show that the calculated F value was about 12 times less than the values displayed in the tables between the lack of adjustment and pure error. The association of these results to the high correlation factor ( $R^2 = 0.78$ ) indicates that the model proposed represented the experimental data well. The mean absolute deviation related to the oil expression obtained from Eq. (2), related to the experimental data was 7.30%. By considering the analysis of variance valid associated with the high correlation values as well as the low absolute average deviation values, the mathematical models generated to explain the oil expression can be considered valid for the intervals of pressing rotation and grain temperature studied. In this way, Eq. (2) was employed to generate the response surface related to the oil expression, as presented in Figure 3.

Table 5. Analysis of variance associated with the reduced codified mathematical models corresponding to the oil expression, evaluated at 90% of confidence.

Source of variation	df	SSq	MS	F <sub>calc</sub>	F <sub>tab</sub>	R <sup>2</sup>
Regression	2	207.0	104.0	12.20	3.26	0.78
Residues	7	59.5	8.5			
Lack of adjustment	6	57.6	9.6	5.24	58.20	
Pure error	1	1.8	1.8			
Total	9	267.0				

From Figure 3, it can be seen that the pressing speed had an effect on the oil yield in the range studied. This result indicates that a increase in pressing rotation results in a better oil yield. As can be seen from Figure 3, the rotation between 105 and 110 rpm was that presenting a better oil yield, independent of the grain temperature used.

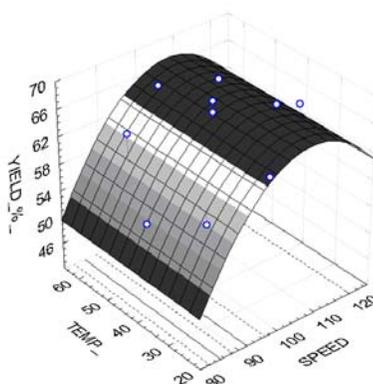


Figure 3. Response surface obtained from the reduced codified model corresponding to the oil expression.

The acid number of sunflower crude oil was 2.8 mg KOH/g and moisture content and volatile matter was 0.04%. Triacylglycerides (TAGs) found in this oil contained 3.6% of palmitic acid (16:0), 3.2% of stearic acid (18:0), 78.8% of oleic acid (18:1), 12.5% of linoleic acid (18:2), 0.1% of linolenic acid (18:3), 0.3% of arachidic acid (20:0), 0.9% of behenic acid (22:0) and 0.4% of lignoceric acid (24:0) as their main fatty acid

components. Sunflower seeds used were high oleic type, as can see by fatty acid composition.

**Laboratory transesterification** The unpurified biodiesel mass obtained in the different trials varied from 58.60% to 87.45% (Table 6). The best result was associated with test number 2, in which the molar ratio was 4.7:1 (ethanol:oil) and the catalyst concentration of 4.42%. It was possible to notice a tendency to increase the biodiesel mass with reduction in the molar ratio. Optimization data is shown in Table 6. The adjustment coefficients of the experimental data to the mathematical model represented by Eq. (1) are presented in Table 7.

Table 6. Average unpurified biodiesel yield corresponding to the laboratory transesterification based on a 2<sup>2</sup> factorial experimental design, with four axial and factorial points and three central points.

Test	Variables		Unpurified biodiesel (wt%)
	Molar ratio	Catalyst conc. (wt%)	
1	4.7 (-1)	1.58 (-1)	72.54
2	4.7 (-1)	4.42 (+1)	87.45
3	13.3 (+1)	1.58 (-1)	60.17
4	13.3 (+1)	4.42 (+1)	58.60
5	3.0 (-1.41)	3.00 (0)	83.69
6	15.0 (+1.41)	3.00 (0)	60.65
7	9.0 (0)	1.00 (-1.41)	64.24
8	9.0 (0)	5.00 (+1.41)	72.61
9	9.0 (0)	3.00 (0)	73.68
10	9.0 (0)	3.00 (0)	73.29
11	9.0 (0)	3.00 (0)	74.19

The results shown in Table 7 demonstrate that all the coefficients were considered statistically significant at a 90% confidence level. The negative of both coefficients indicates that the unpurified biodiesel mass showed a tendency to increase as the molar ratio and catalyst concentration decrease. Based on these results, the codified mathematical model representing the biodiesel production at laboratory scale (Eq. (1)), can be reduced to a quadratic model containing six parameters Eq. (3).

$$BY = 73.7211 - 18.4809MR - 1.8544MR^2 + 6.3040C - 5.6230C^2 - 8.2415MRC \quad (3)$$

Table 7. Regression coefficients associated with the codified mathematical models for biodiesel transesterification.

Regr. coefficient	Effect	Standard deviation	p
<i>b<sub>0</sub></i>	<i>73.7211</i>	<i>0.2598</i>	<i>0.0000</i>
<i>b<sub>1</sub></i>	<i>-18.4809</i>	<i>0.3187</i>	<i>0.0003</i>
<i>b<sub>11</sub></i>	<i>-1.8544</i>	<i>0.3803</i>	<i>0.0396</i>
<i>b<sub>2</sub></i>	<i>6.3040</i>	<i>0.3187</i>	<i>0.0025</i>
<i>b<sub>22</sub></i>	<i>-5.6230</i>	<i>0.3803</i>	<i>0.0045</i>
<i>b<sub>12</sub></i>	<i>-8.2415</i>	<i>0.4500</i>	<i>0.0030</i>

Values marked in italics are statistically significant coefficients at a level of 90%.

b: regression coefficient

12: interaction effect between MR and C

1: linear effect of the molar ratio

2: linear effect of the catalyst concentration

11: quadratic effect of the molar ratio

22: quadratic effect of the catalyst concentration

Table 8. Analysis of variance associated with the reduced codified mathematical models corresponding to the biodiesel production, evaluated at 90% of confidence.

Source of variation	df	SSq	MS	F <sub>calc</sub>	F <sub>tab</sub>	R <sup>2</sup>
Regression	5	873.0	175.0	66.70	6.26	0.99
Residues	4	10.5	2.62			
Lack of adjustment	3	10.1	3.35	8.28	215.71	
Pure error	1	0.4	0.41			
Total	9	883.0				

The analysis of variance was performed to verify the validity of the models represented by Eq. (3) and the results are presented in Table 8. The results of the analysis of variance associated with the biodiesel production indicate that the F value between the regression and the residues is almost 10 times higher than the F value shown in the table. The results also show that the calculated F value was about 25 times less than the values displayed in the tables between the lack of adjustment and pure error. The association of these results to the high correlation factor ( $R^2 = 0.99$ ) indicates that the model proposed represented the experimental data well. The mean absolute deviation related to the biodiesel production obtained from Eq. (3), related to the experimental data was 11.70%. By considering the analysis of variance valid associated with the high correlation values as well as the low absolute average deviation values, the mathematical models generated to explain the biodiesel production can be considered valid for the intervals of molar ratio and catalyst concentration studied. In this way, Eq. (3) was employed to generate the response surface related to the biodiesel production at laboratory, as presented in Figure 4. From Figure 4, it can be seen that the molar ratio had an effect on the unpurified biodiesel yield in the range studied. This result indicates that a decrease in molar ratio results in a better biodiesel. Catalyst concentration between 2.5 to 5.5 wt% indicated a higher biodiesel yield.

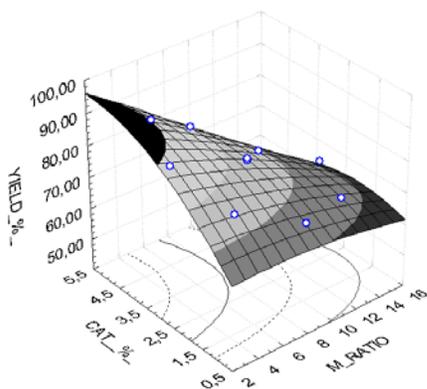


Figure 4. Response surface obtained from the reduced codified model corresponding to the biodiesel production.

**Reactor transesterification** Best experimental condition obtained at laboratory scale was applied at batch reactor: molar ratio of 4.7:1 (ethanol:oil) and 4.42 wt% of catalyst concentration related to the quantity of oil used. After all ethanol was evaporated from unpurified biodiesel, it was observed great amount of soaps, differently that was observed during the reactions conducted at laboratory scale. Soap formation is inevitable when this kind of catalyst is used for alcoholysis. This compound acts as strong emulsifiers and their accumulation usually leads to lower reaction yields and greater technical difficulties

in obtaining a good phase separation. Therefore, that alkali catalyst concentration should be reduced as much as possible, as well as the amount of water present in the chemical reactants (Domingos et al., 2008).

**Purification** The biodiesel samples were conducted to laboratory and the purification techniques were applied. Biodiesel washing was really affected by the presence of soaps in unpurified biodiesel. A lot of acid water was necessary to neutralize the sample and remove the contaminants. It was generated 3.1 liters of liquid effluent and the yield of this purification technique was 84.2%. The yield of “dry washing” was almost the same that was measured for biodiesel washing, 84.6%. Silica has the advantage of not generating liquid effluents. Distillation had the higher yield in purified biodiesel: 92.3%. During the purification process, it was possible to see a very efficient removal of soaps and contaminants.

**Biodiesel characterization** As presented in Fig. 5, all the biodiesel samples had an aspect limpid and with no impurities. According to Table 9, some parameters were in non-conformity with Resolution 7 of ANP.

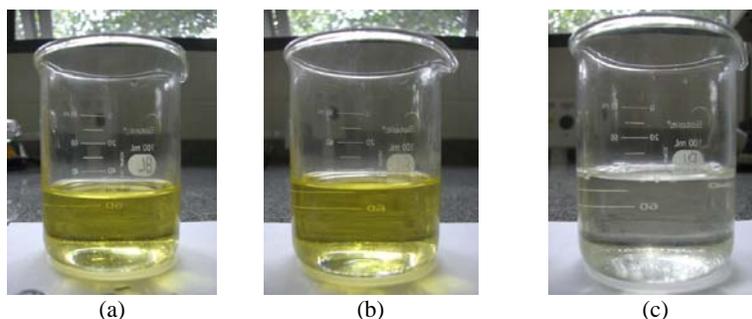


Figure 5. Sunflower crude oil ethyl esters purified (a) Washing (b) Silica (c) Distillation.

The higher water content in all samples can be explained by the absorption of water during transesterification reaction. As sodium methylate is a really hygroscopic matter the water could have contaminated the biodiesel. After purification with water, biodiesel should be dried under reduced pressure to remove all excessive water. Silica chemical structure not allows water removing but as the process was conducted under reduced pressure, some water could be removed. Distillation process was carried on under reduced pressure and temperature above 150 °C, these experimental conditions provided higher water evaporation. In relation to acid number, it was not possible to confirm if the higher acid number observed in all biodiesel samples was caused by raw material initial acid number (2.8 mg KOH/g) or if it was caused by residues accumulated during the purification processes (Wyatt et al., 2005). Probably, this higher value is caused by free fatty acids formation that is miscible with esters, contributing to increase its acid number. Oxidation stability was in non-conformity with ANP resolution. It was already expected because sunflower oil is a non-stable raw material. This problem can be solved by employing antioxidants, like BHT. European limits for triacylglycerols are: 0.8 % for monoacylglycerides, 0.2 % for diacylglycerides and triacylglycerides.

Table 9. Specification parameters of sunflower crude oil ethyl esters.

Parameter	Water	Silica	Distillation	Limit
Density at 20 °C (kg/m <sup>3</sup> )	873.5	873	870	850-900
Kinematic viscosity at 40 °C (mm <sup>2</sup> /s)	5.3	5.3	5.0	3.0-6.0
Water content (ppm)	1260.3	788.3	566.2	500
Total contamination (mg/kg)	0.8	4	6.8	24
Flash point (°C) minimum	165.6	101.6	102.7	100.0
Yield of esters (%) minimum	97.2	98.3	99.3	96.5
Carbon residue (% w/w)	0.02	0.05	0.01	0.05
Ash content (% w/w)	0.02	n.d.	0.01	0.02
Sulphur (mg/kg)	3.1	3.1	3.8	50
Sodium + Potassium (mg/kg)	2.8	5.0	1.7	5
Calcium + Magnesium (mg/kg)	1.8	n.d.	n.d.	5
Phosphorus (mg/kg)	n.d.	n.d.	n.d.	10
Copper corrosion, 3 h at 50 °C	1	1	1	1
Cold filter plugging point (°C)	-9	-2	-11	19
Acid value (mg KOH/g)	3.10	0.93	0.30	0.50
Free glycerol (% w/w)	n.d.	n.d.	0.02	0.02
Total glycerol (% w/w)	0.70	0.71	0.16	0.25
Monoacylglycerides (% w/w)	2.53	2.52	0.40	Report
Diacylglycerides (% w/w)	0.27	0.30	0.14	Report
Triacylglycerides (% w/w)	0.04	0.04	0.12	Report
Ethanol (% w/w)	0.02	0.02	0.07	0.20
Iodine value (g/100 g)	89.3	88.8	96.6	Report
Oxidation stability at 110°C (h)	1.3	2.4	<1	6

n.d.: not detected.

**CONCLUSION** Our data recommend the following procedure for the sunflower oil expelling: 114 rpm for expeller rotation and 25 °C for grain temperature. For transesterification at laboratory scale, the following procedure was recommended: molar ratio of 4.7:1 and catalyst concentration of 4.2 wt%. Besides these experimental conditions were adequate for laboratory scale, when the batch reactor was used, the biodiesel production was not satisfactory. Distillation was the best purification method and the final biodiesel product was able to meet most of the technical requirements of Brazilian specifications.

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