

LUIZ DANIEL DA SILVA NETO¹
PAULO VICTOR FERREIRA LOZ²
JOÃO INÁCIO SOLETTI²
DAYANA DE GUSMÃO COELHO²

¹Department of Chemical Engineering, Federal University of São Carlos Rodovia Washington Luís, São Carlos - São Paulo, Brazil

²Laboratory of Separation and Process Optimization Systems, Technology Center, Federal University of Alagoas, Maceió, Alagoas, Brazil

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FACTORIAL DESIGN AND SURFACE METHOD TO OPTIMIZE ETHYLIC BIODIESEL PRODUCTION FROM CHICKEN WASTES

Article Highlights

- Ethylic biodiesel production from chicken wastes
- Production optimization
- *Physical and chemical properties of chicken wastes oil biodiesel*

Abstract

To produce biodiesel, one of the most important factors is the quality of the oil used in the production. However, other factors such as price and availability should also be taken into consideration. Chicken wastes oil shows a very promising option for this sector, since it comes from a waste. The aim of the present study was a methodology for extraction and characterization of chicken wastes oil for production of biodiesel by ethylic transesterification, besides presenting the characterization and optimization of the process variables of the production of biodiesel, the applying of RSM involving CCD. The investigation was performed following evaluation of the characterization parameters for the oil: viscosity, density, acidity index, moisture of the sample, refractive index, and saponification index. For biodiesel, viscosity, density, acidity index and ester content were evaluated. An optimum point was reached for the production of chicken biodiesel where the concentration of the catalyst is 2.1% by mass, the oil/ethanol ratio is 1:5.5 and the reaction temperature of 30 °C; at that point, a 95% conversion was achieved. At the optimum point obtained from the statistical technique for a p-value of 0.05 the results found for the physical-chemical characterization of biodiesel are found in the ANP standards.

Keywords: biodiesel; chicken wastes; ethyl transesterification; optimization.

Over the years, diesel motors became one of the main sources of power generation around the world, due to the efficiency and economy, being used in transport, agriculture, etc. However, with technological advancement and population growth, the high consumption and rapid depletion of fossil fuels compel the energy sector to adapt to this new reality [1,2]. In this way, also aiming at the significant reduction of the emission of polluting gases and, consequently, to

contribute to the minimization of the environmental impacts on the planet, several energy sources were studied and implemented over the years [3].

Biodiesel, with an energy efficiency equivalent to that of diesel oil obtained from petroleum refining, is derived from vegetable oils and animal fats. They present advantages to petroleum diesel for not emitting carbon monoxide, sulfur and hydrocarbons, have low emission of vapors, are biodegradable and non-flammable [4]. Some raw materials used for the production of biodiesel are vegetable oil [5], algae biomass [6-9], residual cooking oil [10-13], and animal fats [14-16]. Many countries use methanol as an alcoholic reagent to produce biodiesel. However, by producing a fuel with raw materials from fully renewable sources, ethanol can be used in the transesterification of biodiesel.

Correspondence: L.D. da Silva Neto, Department of Chemical Engineering, Federal University of São Carlos Rodovia Washington Luís, km 235 - SP-310 São Carlos - São Paulo, P.O. Box: 13565-905, Brazil.

E-mail: ld_neto@hotmail.com

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In Brazil, although most of the biofuel is ethanol, obtained from sugarcane, biodiesel is already part of the energy matrix. Currently, 10% of the percentage of diesel oil sold to the consumer is biodiesel and the estimated consumption was 5.3 billion L in 2018. Most of the oils used in biodiesel production in Brazil come from traditional oleaginous oils such as soybean oil. However, the inclusion of other raw materials in this matrix is important and aims to reduce confrontation with the food industry.

According to the report of the Brazilian Association of Animal Proteins (ABPA), Brazil is the second largest producer of poultry meat in the world, with a production of 13,056 million tons, behind only the 18,596 million tons produced by the United States of America [17]. Thus, the meat processing industries produce a large amount of animal fat, where part of this by-product is destined for the chemical, pharmaceutical and chemical industries [16]. However, just a part of the animal fat receives suitable management and reaches its destination, especially the ones that come from chicken slaughterhouses that do not have a processing unit to deal with them, which makes them have problems for the elimination of those residues.

The study of process variables such as the use of ethyl route from an environmental point of view is more attractive because ethanol can be obtained from renewable sources. It is important to consider that other factors influence biodiesel yield and multivariate optimization has stood out for evaluating the interaction between variables in the process response.

In this study, a Central Composite Design (CCD) to optimize the reaction parameters was used to get the best yield in the production of biodiesel from chicken wastes. The parameters considered were the oil/ethanol mass ratio, catalyst concentration and temperature on the production of ethylic biodiesel via basic catalysis.

MATERIALS AND METHODS

Raw materials

The residues used on this work, which resulted from cutting chicken meat, were provided by Cortez Supermarket, Coruripe, Brazil. From these residues we were able to extract the raw materials (chicken fatty oils).

For the realization of the analytical methodologies and reaction transesterification, ethyl alcohol, heptane, hydrogen gas and sodium hydroxide were analytical grade and purchased commercially and used without any prior purification. The materials were weighed on a precision balance (Mars, model AM220, Brazil) that had an uncertainty of $\pm 0.0001\text{g}$.

Obtaining the oil

The crude residue was obtained from chicken wastes from the supermarket ground. The cooking of the residue was carried out in a vessel with water, in a ratio of 2:1 water/fat. The oil was separated by decantation and filtered through a vacuum filter to melt the fat. The obtained fat was then filtered to remove impurities and stored in bottles kept under refrigeration. This oil was taken to a greenhouse where any residual water was removed to be used in transesterification reactions.

Gas chromatography

For the identification of the fatty acids in chicken oil, a transesterification reaction was carried out with NaOH to form ethyl esters. These samples of ethyl esters were then analyzed by gas chromatography, by adapting the method prescribed by the European standard EN 14103, where approximately 0.05 g of the sample was dissolved in 1.0 mL of heptane. CG-2010/Shimadzu was used equipped with a split/splitless injection system operating at 250 °C, split ratio of 100:1, sample volume of 1.0 μL and flame ionization detector operating at 250 °C. A 30-meter-long apolar capillary column ZB-WAXplus, with a 0.32 mm internal diameter and 0.25 micron-thick film and high purity hydrogen gas, used as drag, was used. The temperature programming was constant temperature of 200 °C (10 min). The fatty acid composition was calculated based on the identification and integration of the peak areas by normalization.

Transesterification process

Biodiesel from chicken wastes was obtained by transesterification reaction, where anhydrous ethanol was used as the alcoholic agent and sodium hydroxide was used as the catalyst. The reactions were carried out in 1500 mL reactors with a magnetic stirrer and equipped with temperature control. Initially, the catalyst was dissolved in the anhydrous alcohol and subsequently added to the chicken waste oil. The reaction time was 30 min. Subsequently, the mixture was transferred to a separating funnel and allowed to stand for 12 h. The lower layer of the glycerol was removed, and the upper phase of the biodiesel subjected to the washing process with distilled water, at room temperature. After that, the oven was dried for a total time of 24 h to remove water and alcohol present.

Optimization and data analysis

Experiment planning techniques were used to optimize the operating conditions of the production of biodiesel from fatty residues of chicken. To determine

the experimental conditions optimizing the experiment, the effect of variables and their interactions were evaluated, which influence the yield of the transesterification reaction [12,18]. In this step, the oil/ethanol ratio, amount of catalyst and temperature in the conversion of the reaction were evaluated. In literature reports, experiments were carried out on agitation speed, the type of paddles, temperature, catalyst and alcohol/molar ratio; the factors that presented a greater significance for chicken waste biodiesel were temperature, catalyst and alcohol mole ratio [19,20]. The experimental data obtained by following the above procedures were analyzed by the response surface regression procedure using the following second-order polynomial equation, Eq (1):

$$y = \beta_0 + \sum_j \beta_j x_j + \sum_{i < j} \beta_{ij} x_i x_j + \sum_j \beta_{jj} x_j^2 + e \quad (1)$$

where y is the response (percentage conversion); x_i and x_j are the uncoded independent variables and β_0 , β_i , β_{ij} and β_{jj} are intercept, linear, quadratic and interaction constant coefficients, respectively. Design Expert software package was used for regression analysis and analysis of variance (ANOVA) [21]. The software used for evaluation and validation of the data obtained was Statistica® 13.0 Statsoft, Inc.

Physical and chemical properties

The physicochemical properties were evaluated according to the methods established by the American Society for Testing and Materials (ASTM), the results compared to the European standards and the Brazilian National Agency of Petroleum, Natural Gas and Biofuels (ANP).

The investigation was performed following parameter characterization for the oil: viscosity, humidity, acidity index, and saponification index were evaluated, presented in Eqs. (2)-(5):

$$\nu = tK \quad (2)$$

where ν is a viscosity (mm²/s), t is the time (s); K is constant (0,1125 mm²/s²).

The humidity was determined in Eq. (3), where U is humidity, N is difference between final and initial mass, P is initial mass:

$$U = \frac{100N}{P} \quad (3)$$

Eq. (4) presented the acidity index, where IA is acidity index (mg NaOH/g), V is the volume of NaOH (ml), f is correction factor, M is molarity and m is the mass of the sample.

$$IA = \frac{28.2VfM}{m} \quad (4)$$

The saponification index, IS , is presented in Eq. (5) where A is volume of the sample titration, B is volume of the white sample, f is correction factor of the solution HCl 0,5 M, P is initial mass of the sample:

$$IS = \frac{26.6f(B-A)}{P} \quad (5)$$

The density was analyzed by ASTM D 4052 and the refraction index was checked by Abbe refractometer.

RESULTS AND DISCUSSION

Physical and chemical properties

Transesterification is influenced by the physical and chemical characteristics of the raw materials, so some properties of biodiesel can be predicted from the analysis. The results found in this study are shown in Table 1.

Table 1. Physical-chemical properties of chicken oil and biodiesel of chicken oil

Property	Chicken oil	Chicken biodiesel
Refractive index	1.4646±0.0001	-
Viscosity 40 °C (mm ² /s)	48.46±0.2	5.839±0.2
Density 20 °C (kg/m ³)	917.30±0.5	884.35±0.5
Humidity (%)	0.398±0.0018	-
Acidity index (mg KOH/g)	0.759±0.05	0.716±0.05
Saponification index (mg KOH/g)	191.51±0.8	-

Compared with soybean oil, which is the main raw material for biodiesel production in Brazil (ANP), the oil of chicken had similar density and a lower viscosity, which is satisfactory for the production of biodiesel, since high values of viscosity and density can cause problems related to the fuel injection in the engine, damaging its performance [22,23].

For base-catalyzed transesterification, the glycerides and alcohol must be substantially anhydrous; the water causes partial saponification that produces soap, consuming the catalyst and reducing the catalytic efficiency, in addition to causing increased viscosity, gel formation and difficulty in reaching separation of glycerol [19,24]. Humidity in biodiesel acts increasing the acidity and subsequent formation of soap, as well as causing corrosion of fuel supply systems, microbiological growth and hydrolysis of the methyl ester [25]. Humidity was not a problem, neither in terms of favoring side reactions nor reagent consumption.

The acid number is defined as the number of mg of potassium hydroxide needed to neutralize 1 g of the sample. For transesterification to be efficient, the amount of free fatty acids present in the raw material must be less than 3% [26]. Researchers suggest that to use an alkaline catalyst, the level of free fatty acids in the raw materials should be reduced to less than 1% [27,28], but as can be seen in Table 1, the index found was less than 1 mass%, thus, no oil treatment step was necessary.

Most of the raw materials used for biodiesel production have higher values than the one found in this study. The chicken oil had an adequate acid index, with a low presence of free fatty acids, indicating a good raw material for the biodiesel production, without compromising the transesterification process.

The saponification index of the chicken oil found in this study was 190.51 mg KOH/g. Like the viscosity and the density, the saponification index does not have a standard value set in resolution of the ANP, but compared to the literature, the value found is within the parameters [29]. A lower saponification index leads to a higher yield in the reaction, due to the decrease of the parallel reactions that can lead to the formation of soap. This index directly influences the amount of alcohol in the transesterification since an excess of alcohol is used to avoid the formation of soap.

Gas chromatography

The gas chromatography technique is used to determine the fatty acid profile of the raw material. Table 2 shows the oil profile used as raw material for this study. Chicken oil is composed of 25 to 35% of saturated fatty acids and 40 to 75% of unsaturated fatty acids [30,31]. Thus, with the known composition and after a transesterification reaction, the respective triglycerides were obtained. Thus, through the percentage of each component of the oil, the molar mass was obtained (Table 2). With 95.5% of the oil composition known and that this percentage corresponds to 820.592 g/mol, this basis of calculation was used to obtain the total molar mass of 859.26 g/mol.

Table 2. Composition of fatty acids and triglycerides present in chicken oil

Fatty acid	Content (%)	Triglyceride	Correspondent molar mass (g/mol)
Myristic	0.6	Trimyristate	4.332
Palmitic	22.3	Tripalmitate	179.738
Palmitoleic	4.5	Tripalmitoleate	36.000
Stearic	4.9	Triestearate	43.610
Oleic	35.4	Trioleate	312.936
Linoleic	26	Trilinoleate	228.280
Linolenic	1.8	Trilinolenate	15.592
Others	4.5	-	-

Ester content and optimization

The variables studied in the experimental design, their respective levels, and the results of ester concentration in the biodiesel of chicken oil obtained are shown in Table 3.

Table 3. Experimental matrix for factorial design 2³, with triplicate at the central point and 6 axial points, with maximum conversion of esters

Case	Temperature °C	Proportion	Catalyst content %	Ester content %
B1	35 (-1)	1;5.0238 (-1)	1.25 (-1)	73.46
B2	65 (1)	1;5.0238 (-1)	1.25 (-1)	51.82
B3	35 (-1)	1;10.9762 (1)	1.25 (-1)	58.28
B4	65 (1)	1;10.9762 (1)	1.25 (-1)	86.83
B5	35 (-1)	1;5.0238 (-1)	2.25 (1)	92.88
B6	65 (1)	1;5.0238 (-1)	2.25 (1)	52.88
B7	35 (-1)	1;10.9762 (1)	2.25 (1)	91.59
B8	65 (1)	1;10.9762 (1)	2.25 (1)	67.91
B9	50 (0)	1;8 (0)	1.75 (0)	88.99
B10	50 (0)	1;8 (0)	1.75 (0)	89.02
B11	50 (0)	1;8 (0)	1.75 (0)	89.09
B12	24.8 (-1.68)	1;8 (0)	1.75 (0)	86.67
B13	75.2 (1.68)	1;8 (0)	1.75 (0)	59.52
B14	50 (0)	1;3 (-1.68)	1.75 (0)	68.95
B15	50 (0)	1;13 (1.68)	1.75 (0)	75.34
B16	50 (0)	1;8 (0)	0.91 (-1.68)	68.86
B17	50 (0)	1;8 (0)	2.59 (1.68)	41.34

The data obtained in the experimental design were treated statistically and, in this way, it was observed the need of adding the axial points to the planning due to the non-representativeness of the linear model to the obtained data was observed.

Through CCD planning it was possible to analyze the effects of the studied variables, allowing to verify statistically their significance for biodiesel production. The Pareto graph (Figure 1) shows the effects of variables that are statistically important, as well as presenting the effects of two-to-two interactions between these variables.

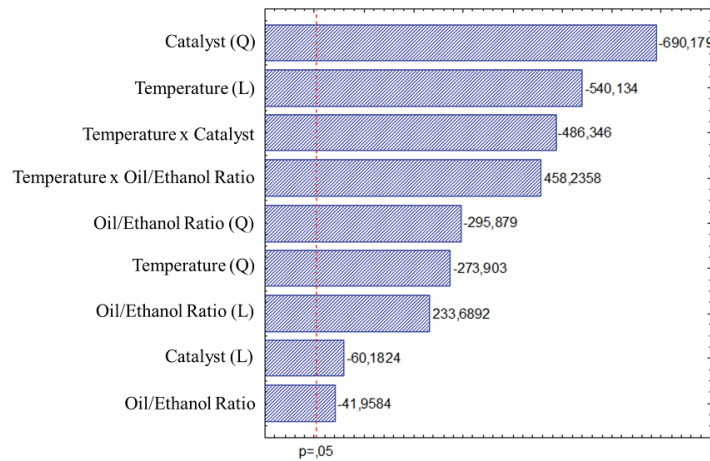


Figure 1. Pareto graph.

The effect of a variable is more significant the further it is on the right side of the line $p = 0.05$, which represents a significance level of 95%, so those behind the line are not considered to be statistically significant. Once the significant effects have been defined, we can evaluate their interference in the response variable by studying the Response Surface Methodology (RSM), analyzing two variables simul-

taneously. For the comparison of ester conversion in biodiesel as a function of catalyst concentration and reaction temperature, the oil/ethanol ratio was kept fixed at the center point (Figure 2).

For the comparison of the ester conversion as a function of the oil/ethanol ratio and catalyst concentration, the reaction temperature was maintained at the central point of the planning (Figure 3).

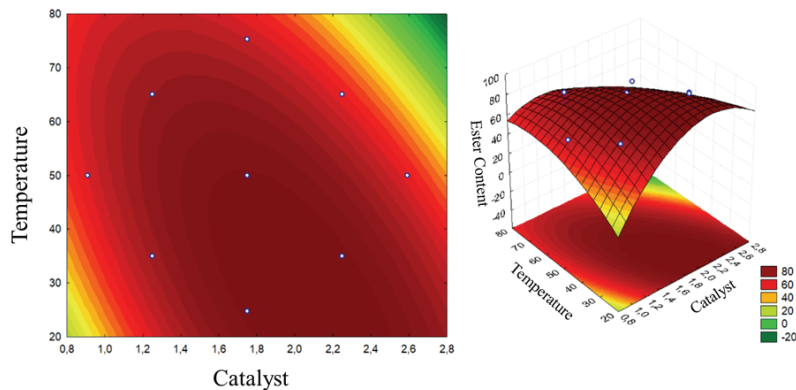


Figure 2. Response surface for ester conversion as a function of catalyst concentration and temperature with a fixed oil/ethanol ratio of 1:8.

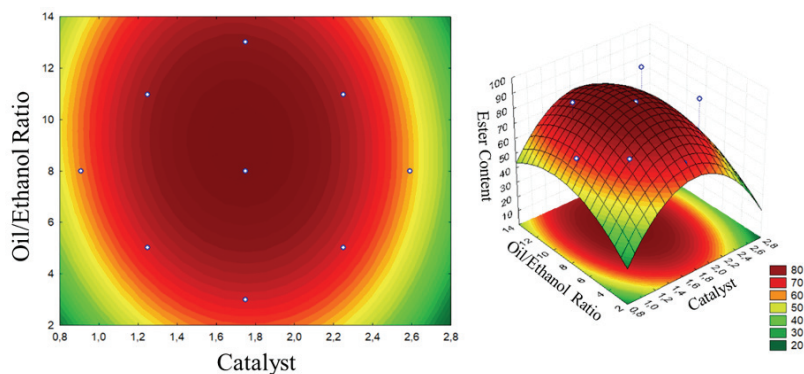


Figure 3. Response surface for ester conversion as a function of catalyst concentration and the oil/ethanol ratio with a fixed temperature of 50 °C.

Finally, the ester conversion was evaluated as a function of the oil/ethanol ratio and reaction temperature, with the catalyst concentration at the center point (Figure 4).

In Figures 2 and 3 we can see that the optimum biodiesel conversion range is between 1.8 and 2.2%, for the catalyst concentration. High concentrations of the catalyst tend to form salts of fatty acids, making the purification process difficult and causing product losses. The low concentrations of the catalyst are insufficient for the reaction to occur, reducing the yield [32].

Figures 3 and 4 show that the optimum biodiesel conversion range is between 1:5 and 1:9, for the oil/ethanol ratio. Low oil/ethanol ratios reduce the yield due to the reversibility of the reaction and, on the other hand, the excess alcohol deactivates the performance of the catalyst [33]. Thus, an optimized mole ratio will reduce the cost of producing biodiesel, decreasing the amount of ethanol used. In Figures 2 and 4 it is observed that the temperature extremes reduce the reaction yield for some analyses and the optimum conversion range is between 20 and 40 °C.

Because it is a fat, at low temperatures the chicken oil is in a solid state, which makes the reaction difficult. The reaction temperature is normally dependent on the type of alcohol used, so the temperature is kept below the boiling point of the alcohol, keeping its level constant in the reaction bottle [3].

With the obtained data it was possible to obtain an optimization for biodiesel production for the variables and levels studied, that can be expressed by Eq. (6) (Table 4):

$$C_{Ester} = -136.203 + 1.930T_L - 0.019T_Q + 0.43R_L - 0.511R_Q + 208.924C_L - 42.94C_Q + 0.186T_LR_L - 1.176T_LC_L - 0.512R_LC_L \quad (6)$$

where C_{Ester} corresponds to ester concentration in biodiesel (g/g), R_L , the linear ratio oil/ethanol (g/g), R_Q , the quadratic ratio oil/ethanol (g/g), C_L , the linear catalyst concentration (g of cat./g of oil), C_Q , the quadratic catalyst concentration (g of cat./g of oil), T_L , the linear temperature (°C) and T_Q , the quadratic temperature (°C).

Table 4. Regression coefficients of predicted quadratic polynomial model

Term		Regression coefficient	Standard error
Intercept	β_0	-136.203	96.02255
Linear	β_1	1.930	1.83354
	β_2	0.843	8.57747
	β_3	208.924	56.11116
Quadratic	β_{11}	-0.019	0.01433
	β_{22}	-0.511	0.36412
	β_{33}	-42.194	12.90127
Interaction	β_{12}	0.186	0.08576
	β_{13}	-1.176	0.51050
	β_{23}	-0.512	2.57290

Physical-Chemical properties of biodiesel

Table 1 shows data on the physicochemical characterization of biodiesel obtained from optimized operating conditions (temperature: 30 °C, catalyst concentration: 2.1%, oil/ethanol ratio: 1:5.5). It can be observed that the biodiesel of chicken oil is in accordance with the specifications of the *ANP* Resolution N° 45/2014 in all the tests.

The value obtained for the specific mass, 884.35 kg/m³, complies with *ANP* Resolution 45/2014, which defines the limit between 850 and 900 kg/m³. The value obtained is close to the diesel value of 853 kg/m³. The density of the oil varies according to the nature of the raw material and, in determining the

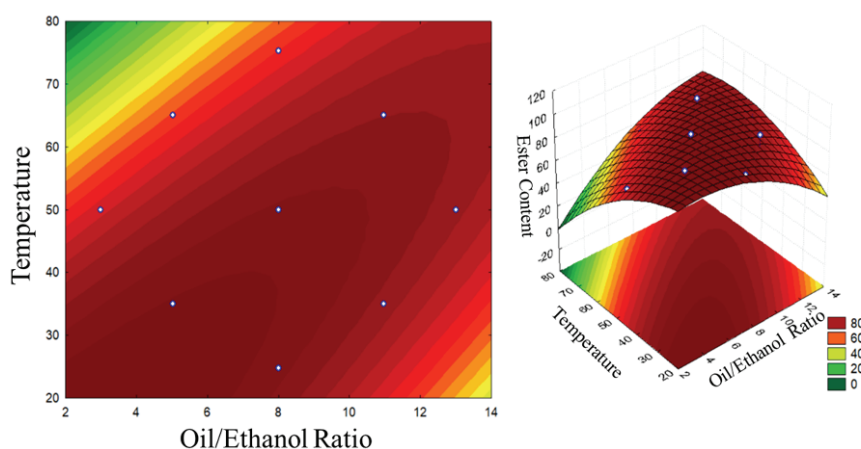


Figure 4. Response surface for ester conversion as a function of temperature and the oil/ethanol ratio with a fixed catalyst concentration of 1.75%.

density of the biodiesel, it is possible to guarantee the longevity and the good functioning of the engine due to the influence on the fuel atomization efficiency [15,34,35].

The value obtained for the viscosity, 5.839 mm²/s, complies with the ANP Resolution 45/2014, which defines the limit between 3.0 and 6.0 mm²/s, the value of the range established for conventional diesel. The viscosity of the biodiesel is influenced mainly by the experimental conditions and the extension of the transesterification reaction. It is a parameter that affects the atomization of the fuel at the time of injection in the combustion chamber and, ultimately, the formation of deposits in the engine [3,15,24,36].

Viscosity and density are important parameters in the biodiesel quality and may be related to the purity of the product obtained, assuming that there are no possible interactions between the ethyl ester and the glycerides. Factors such as incomplete reaction, the presence of mono-, di- and triglycerides or the presence of glycerin, due to inefficient purification, promote changes in viscosity and density. In this way, we can associate one of these physical properties with the degree of purity of the prepared biodiesel oil, being an efficient, fast, and low-cost analytical method for determining the quality and purity of biodiesel.

For the acid value, the obtained value of 0.716 mg KOH/g is within the limits allowed by the ANP. The acid control is extremely important to prevent corrosion of the engine. In addition, the monitoring of the acidity of biodiesel is also of great importance for its storage since the change of values in this period can mean the presence of water. Viscosity and, mainly, the period of induction of Rancimat are parameters that can be used to monitor the oxidative degradation of the biodiesel during the time of storage [15].

About 3.33 chickens are produced per m², so about 33,300 chickens per ha are produced. The average unit weight of chicken is 2.7 kg, where 8% is greasy residue. As 43.8% (data obtained in this work), about 3,150 kg are obtained per ha. As the production of chicken for slaughter occurs five times a year, we have a final value of about 15,750 kg of oil per ha per year. Comparing this yield with those of other oilseeds, we can theoretically produce about 25 times more chicken oil per ha than soybean oil, the main raw material for biodiesel production in Brazil [11,24,34].

CONCLUSION

The studies show that the reuse of the fatty residues of chicken is a viable alternative for the pro-

duction of biodiesel, since it has an excellent extraction yield and a high productivity - approximately 25 times higher than soybean oil per hectare, the most used raw material for biodiesel production in Brazil. The factorial design was efficient in the screening of significant variables to a 95% confidence interval and it was possible to describe the process. The central composite design resulted in optimized conditions being determined for the maximum biodiesel chicken wastes yield where the concentration of the catalyst is 2.1% by mass, the oil/ethanol ratio is 1:5.5 and the reaction temperature is 30 °C. From the equation obtained (Eq. (6)) it is possible to predict the conditions required to obtain a higher yield.

The biodiesel of chicken oil presented adequate characteristics for its use, since its specifications agree with specification parameters listed in Technical Regulation N° 7/2008 of the National Agency of Petroleum, Natural Gas and Biofuels.

Nomenclature

<i>A</i>	Volume of the sample titration, mL
ABPA	Brazilian Association of Animal Proteins
ANOVA	Analysis of Variance
ANP	Brazilian National Agency of Petroleum, Natural Gas and Biofuels
ASTM	American Society for Testing and Materials
<i>B</i>	Volume of the white sample, mL
β_i	Linear coefficient
β_{ii}	Quadratic coefficient
β_{ij}	Interaction coefficient
β_0	Intercept coefficient
CCD	Central composite design
C_{Ester}	Ester concentration, g/g
C_L	Linear coefficient of catalyst concentration, g of cat./g of oil
C_Q	Quadratic coefficient of catalyst concentration, g of cat./g of oil
<i>f</i>	Correction factor
<i>I</i> A	Acidity index, mg NaOH/g
<i>I</i> S	Saponification index, mg KOH/g
<i>K</i>	Constant, mm ² /s ²
<i>m</i>	Mass of the sample, g
<i>M</i>	Molarity, mol/mL
<i>N</i>	Final and initial mass difference, m
<i>P</i>	Initial mass, m
R_L	Linear coefficient of ratio oil/ethanol, g/g
R_Q	Quadratic coefficient of ratio oil/ethanol, g/g
RSM	Response Surface Methodology
<i>t</i>	Time, s
T_L	Linear coefficient of temperature, °C
T_Q	Quadratic coefficient of temperature, °C
<i>U</i>	Humidity, %

ν	Viscosity, mm ² /s
V	Volume of NaOH, mL
x_i	Independent variable
x_j	Independent variable
y	Model response

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LUIZ DANIEL DA SILVA NETO¹
PAULO VICTOR FERREIRA LOZ²
JOÃO INÁCIO SOLETTI²
DAYANA DE GUSMÃO COELHO²

¹Department of Chemical Engineering,
Federal University of São Carlos
Rodovia Washington Luís, São Carlos -
São Paulo, Brazil

²Laboratory of Separation and Process
Optimization Systems, Technology
Center, Federal University of Alagoas,
Maceió, Alagoas, Brazil

NAUČNI RAD

STATISTIČKA OPTIMIZACIJA PROIZVODNJE BIODIZELA IZ PILEĆEG OTPADA I ETANOLA

Za dobijanje biodizela, jedan od najvažnijih faktora je kvalitet ulja koje se koristi u proizvodnji. Međutim, i druge faktore, kao što su cena i raspoloživost, treba, takođe, uzeti u obzir. Otpadno pileće ulje je vrlo obećavajuća mogućnost za ovu proizvodnju, jer potiče iz otpada. Cilj ovog rada bio je razvoj metodologije za ekstrakciju i karakterizaciju otpadnog pilećeg ulja i njegovu primenu za proizvodnju biodizela etanolizom, kao i optimizacija procesnih uslova proizvodnje biodizela primenom metodologiji površine odziva kombinovanom sa centralnim kompozitnim planom. Određene su karakteristike ulja: viskozitet, gustina, kiselost, vlažnost uzorka, indeks refrakcije i saponifikacioni broj, kao i karakteristike biodizela: viskozitet, gustina, kiselost i sadržaj estera. Pri optimalnim uslovima proizvodnju biodizela iz pilećeg ulja (koncentracija katalizatora 2,1%, odnos ulje/etanol 1:5,5 i reakciona temperatura 30 °C) postignuto je 95% konverzije. Fizičko-hemijske karakteristike dobijenog biodizela zadovoljavaju ANP standarde.

Ključne reči: biodizel; ileći otpad; etanoliza; optimizacija.