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REGULAR ARTICLE

An experimental investigation of the mechanical variables influence on soybean biodiesel production using the response surface methodology

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Abstract

Biodiesel is a fuel derived from renewable sources such as vegetable oils, animal fats, or residual oils. Although it is a potential source of energy, the efficiency of the production of this fuel depends on several factors, including variables associated with the stirring and mixing process of the reactions. The proper choice of these variables can avoid the formation of vortices, favor the flow direction and the homogeneity of the mixture, and, consequently, contribute to a higher yield of biodiesel. In this context, the present work investigated the effect of agitation and mixing on the production of soybean biodiesel from the analysis of parameters: impeller (blade - turbine), stirring speed (150 rpm – 300 rpm), and baffle (with-without). For this, a 2^3 factorial experimental design was carried out for the methylic and ethylic routes. In the reactions, the oil: alcohol molar ratio, amount of catalyst, time, and temperature were fixed. Experimental results indicated higher yields for reactions via the methylic route (more than 93%). Through the statistical analysis, it was also verified that the presence of a baffle and the use of a turbine impeller were the variables of greater statistical significance for the methylic and ethylic routes, respectively. These results showed that the variables considered had a significant impact on the yield of the reactions, although the reaction conditions remained constant, which reinforces that only the control of stirring and mixing parameters can promote optimal yields of the reactions, reducing costs with reagents, operating time, or temperature control.

Keywords

Biodiesel; impeller; stirring speed; Soybean oil

Introduction

In recent decades, the use of fossil fuels has significantly contributed to the worsening of the greenhouse effect due to the high amount of pollutant gases released into the atmosphere as a result of the burning of these fuels. Given these environmental impacts and the finite nature of petroleum-derived sources, several research groups are investigating potential alternative and renewable sources with high energy potential. In this context, biodiesel stands out. This is obtained from various raw materials, such as vegetables, animal fat, and residual oils, which favors the diversity of material and location of production of this fuel (Andrade, Errico and Christensen 2017; Chen and Lee, 2018; Maran and Priya, 2015).

Biodiesel consists of a mixture of mono-alkyl esters, obtained from the reaction of transforming oils and fats in the presence of a catalyst (Ferella et al., 2010). Since, due to the high viscosity, the raw materials cannot be used directly in diesel engines. Various routes for biodiesel production are cited in the literature such as transesterification (Melo et al., 2020; Gabriel et al. 2020), esterification (Sun et al., 2015; Saeikh and Vinjamur, 2014), electrolysis (Rafati et al., 2019; Farrokheh et al., 2022) and thermal cracking (Serin et al., 2016). Among them, the production by transesterification is predominant because it is an economic process with a high yield in biodiesel. In this technique, biodiesel can be produced from animal fat, vegetable oils, or algae using short-chain alcohol and a catalyst to accelerate the reaction (Singh et al., 2020; Falowo et al., 2021).

Several studies investigate the influence of different variables on the yield of the transesterification reaction, such as raw material characteristics, type of alcohol, type and amount of catalyst, oil: alcohol ratio, reaction time, and temperature (Kumar et al., 2019; Sundaramahalingam et al., 2021; Thakkar, Kachhwaha and Kodgire, 2022). Given a large number of factors, it is a challenge to define which parameters are most relevant to produce biodiesel, capable of providing a

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high yield and keeping the production cost competitive. In this context, many studies used optimization tools, such as experimental design and response surface methodology, to determine favorable operating conditions for biodiesel production, aiming at a higher conversion of esters to favor a high reaction mass yield (Ajala et al., 2017; Andrade, Errico and Christensen, 2017; El-Gendy et al., 2015; Fracari et al., 2009; Hamze, Akia and Yazdani, 2015; Maran and Priya, 2015; Musa, 2016; Onukwulli et al., 2017).

Recently, Silva Neto et al. (2020) investigated the optimal conditions of temperature, oil: alcohol ratio, and percentage of catalyst in the production of biodiesel from chicken wastes using the Central Composite Design. Amruth and Sudev (2019) investigated the parameters of time, reaction temperature, and oil: alcohol ratio to obtain the maximum yield of fish oil biodiesel, using the response surface methodology. The same tool was applied by Sanchez et al. (2018) to optimize the methanol volume, catalyst concentration, and hexane volume used in the water extraction for biodiesel production from wet microalgal biomass.

Although many researchers investigated the effects of different parameters in the biodiesel production process, most of them focused on the chemical reaction variables. However, discussions focused on chemical parameters, in addition to reaction time and temperature, can provide optimal reaction conditions, but usually result in a higher cost associated with reagents or energy expenditure, which compromises the competitiveness of the biodiesel produced against other fuels. In this sense, it is essential to consider other factors of the production process, such as the mechanical parameters of the reactor, related to the phenomena of agitation and mixing, which are little discussed in the literature.

From the study of the reactor's mechanical variables, it is possible to optimize the yield of biodiesel production by keeping the chemical reaction conditions fixed, thus avoiding additional costs with reagents, which is one of the main problems to make biodiesel competitive in the fuel market. Efforts are directed towards safer and reduce the development of technologies capable of optimizing the biodiesel production process (Chen and Lee, 2018; Fracari et al., 2009; Sánchez et al., 2018).

In this scenario, some recent works have addressed the effect of parameters such as agitation speed, baffle, and impeller. Peiter et al. (2020) studied the effect of these variables on the reaction time and identified the period in which they exert more influence on the production of soybean biodiesel. However, the authors carried out experiments with high proportions of reactants (oil/alcohol molar ratio 1:10 and 1.5% of catalyst in relation to the mass of oil) and high temperature (70 ºC), conditions that already tend to favor the yield, but make the reaction more expensive.

Given the lack of further investigations on the subject, this article expands the discussions on the effect of stirring and mixing variables. In this sense, it aims to optimize the process conditions to achieve maximum reaction efficiency by adopting fixed and mild reaction conditions, reducing the associated costs. For this purpose, a 2^3 full factorial design was used. The independent variables were stirring speed (150 rpm - 300 rpm), impeller type (turbine or blade) and baffle presence (with or without). The yield of esters, obtained via gas chromatography, was the output variable. From the experimental data, the effect of each one of the variables and the interactions between them on the yield were investigated using statistical techniques.

Materials and methods

The materials used in the experiments were soybean comercial oil, ethanol PA (Dinâmica®, Indaiatuba, SP, Brazil), metanol PA (Dinâmica®, Indaiatuba, SP, Brazil), sodium hidroxide (Dinâmica®, Indaiatuba, SP, Brazil), magnesium sulfate (Dinâmica®, Indaiatuba, SP, Brazil) and hydrochloric (Dinâmica®, Indaiatuba, SP, Brazil).

Factorial experimental design for biodiesel production

The mechanical properties of the reaction system in a pilot unit to produce biodiesel was evaluated to optimize the soybean biodiesel production process over the variable of interest which is the yield.

A factorial experimental design was applied considering two levels and three variables (2^3) in duplicate, resulting in 16 experiments for each route. The three variables investigated were stirring speed, impeller type, and baffle presence. The complete factorial experimental design 2^3 is shown in Table 1.

Table 1. Variables for the study of soybean biodiesel production.

.	Variables		
Level	**Impeller**	**Baffle**	**Stirring Speed (rpm)**
-1	blade	without	150
+1	turbine	with	350

Level -1 represents the lower limit and level +1 represents the upper limit of each variable. Further, I represents the type of impeller used, B the presence or absence of baffles, and SS the stirring speed of the blend. Two qualitative variables were considered (impeller, I, and baffle, B) and a quantitative variable (stirring speed, SS). Table 2 presents the complete factorial experimental planning 2^3 by the variables and levels defined.

Table 2. Complete factorial experimental planning 2^3.

Experiment	Impeller (x_1)	Baffle (x_2)	Stirring Speed (rpm) (x_3)
1	-1	-1	-1
2	+1	-1	-1
3	-1	+1	-1
4	+1	+1	-1
5	-1	-1	+1
6	+1	-1	+1
7	-1	+1	+1
8	+1	+1	+1

Transesterification reaction

For the transesterification reactions, commercial soybean oil was used. The batch was carried out in a jacketed bench reactor, coupled to a thermostated bath to keep the system temperature constant. Stirring was controlled with a mechanical stirrer. Figure 1 presents a representation of the pilot unit for biodiesel production and the variables considered in this study.

The process variables that remained constant during the reactions were temperature (303.15 K), amount of catalyst (0.5 %), and molar ratio oil/alcohol (1: 5). The reactants used were ethylic and methylic alcohol (according to the production route), sodium hydroxide P.A, as a catalyst, magnesium sulfate as a drying agent, and HCl P.A. After the purification step, the biodiesel remained in the oven at 333.15 K for remove moisture. The experiments were realized randomly to minimize experimental errors.

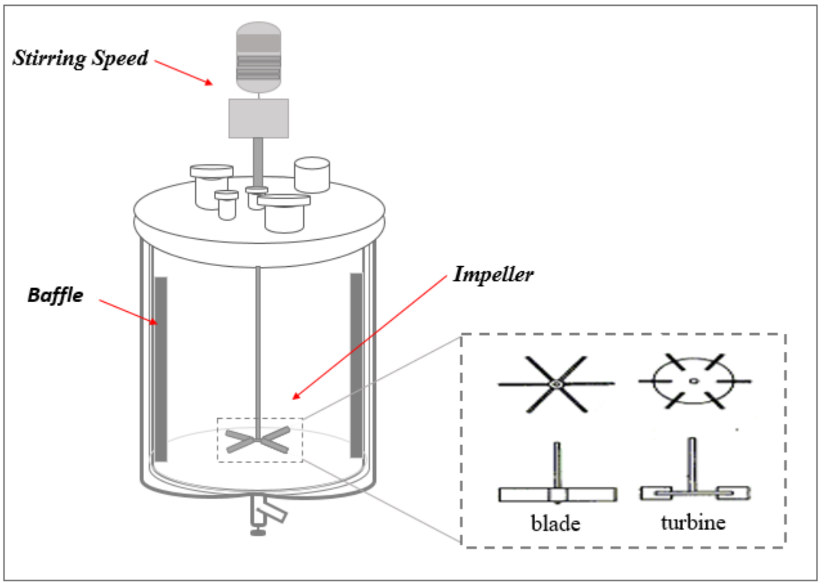

Figure 1. Pilot unit for biodiesel production.

The determination of yields was by gas chromatography using the SHIMADZU GC-Plus model chromatograph with a flame ionization detector and a 2.2 m column with injector temperature of 523.15 K, flame ionization detector temperature of 613.15 K, column temperature 323.15 K, column pressure of 6kPa. The entrainment gases used were hydrogen, nitrogen, and synthetic air. The internal standard used was tricaprylin, at the concentration of 0.8 g/10mL of hexane. The biodiesel samples had a mass of approximately 0.15 g and were diluted in 1mL of the internal standard solution, and 1μL of the sample was injected into the chromatograph. The injections were made in duplicate. The yields of the esters were calculated as (Equation 1):

$$\eta = (m_{tricaprylin} * A_s * f * 100)/A_{tricaprylin} * m_s \quad (1)$$

Where $m_{tricaprylin}$ is the weight of the internal standard, A_s is the sum of the areas of the peaks for the esters contained in the samples, f is the response factor, $A_{tricaprylin}$ is the area of the peak referring to the internal standard, m_s is the weight of the sample.

Finally, the yield results obtained were evaluated using a graphical tool for data analysis, STATISTIC 7.0.

Results and discussion

The average yield in esters for the 16 experiments for ethyl and methylic routes is presented in Table 3.

Table 3. Yield in esters for ethyl and methylic routes.

Experiment	Y_e (%)	Y_m (%)
1	89.3± 4.95	81.45±0.50
2	89.5±2.83	85.2±1.41
3	83.05±4.03	87.05±5.16
4	78.45±2.76	84.55±6.29
5	83.55±4.03	87.7±0.00
6	89.3±4.24	85.5±1.70
7	82.95±2.48	89.15±2.76
8	93.1±0.42	93±3.53

According to Table 3, the methylic and ethylic routes presented close results, with an average standard error equal to 3.22, although methanol is more reactive due to the shorter chain. Only the Table 3 results are not enough to explain the effect of mixing and stirring on the biodiesel yield. So, to obtain a quantitative analysis of the effect of variables, the yield in esters (the dependent variable) was evaluated statistically as a function of the effects of independent variables (X1, X2 e X3). As result, Pareto charts were obtained, considering a confidence interval of 95%, as shown in Figure 2.

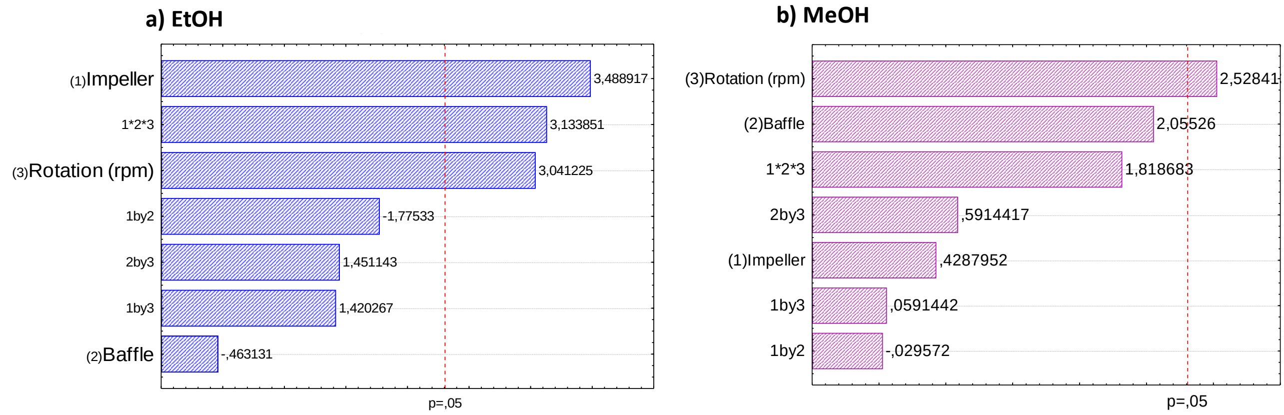

Figure 2. Pareto charts for the factorial experimental design 2³ for the study of the effect of mechanical variables in biodiesel production by a) ethyl and b) methylic routes.

To evaluate the statistical significance of the effects, for confidence of 95%, it is considered statistically significant, an effect whose absolute value is greater, t_N x s (effect), where t_N is the Student distribution for N degrees of freedom and s (effect) is the experimental error for an effect. Applying this criterion, according to Figure 2, mechanical variables affect the reaction differently depending on the type of alcohol used.

Significant individual effects of baffles and stirring speed were observed for the ethyl route, as shown in Figure 2a. The significance of the combined effect of the interaction of the 3 independent variables was also verified. These effects positively influenced the biodiesel yield, which corroborates the statistically significant effect of the interaction of the 3 variables, as evidenced by the analysis of the Pareto chart. The highest yields were achieved in experiments 1, 2, 6 and 8 shown in Table 3, with a maximum yield of 93.1 ± 0.42% performed to test 8 using a higher stirring speed (350 rpm), turbine-type impeller and baffles.

Also, according to Figure 2a, the predominant effect on the production of ethyl biodiesel is attributed to the type of impeller. Comparing the yields, a variation of approximately 16.3 % between the minimum and maximum yields. The maximum corresponds to the use of the turbine-type impeller. The choice of impeller type is crucial to ensure that the fluid follows a preferential path and may also depend on other parameters to be considered, such as stirring speed, physical characteristics of the fluid, and the geometry of the reactor. Thus, the best results indicate that for the characteristics of the reactor used, the turbine reactor provided the best energy supply to the system through stirring. This result corroborates with the data from Adeyemi, Mohiuddin and Jameel (2011) who investigated the influence of impeller geometry on the yield response.

Regarding the stirring speed, it was demonstrated that the increase in the degree of stirring increased the yield of esters, since this parameter is directly associated with the increase in the contact between the alcohol and oil molecules, making the transfer of more efficient mass. However, it is not possible to generalize since very high agitation speeds can decrease reaction rates and consequently reduce the process yield, Hosseini, Nikbakht and Tabatabaei (2012).

Although the presence of baffle was not significant as the main variable, the interaction combined with impeller type and stirring positively influenced the ester yield. The literature points out that the use of baffles improves the fluid dynamics of the process, since it promotes a more efficient mixing of the reactants by redirecting the flow inside the reactor and preventing air from entering the system due to the formation of vortices on the surface, which is reflected in a positive result for both yield and heat transfer efficiency. In addition, the use of baffles reduces system energy consumption and increases the mixing temperature when compared to a baffle less system. The mass transfer in these systems is more efficient in these systems when combined with the use of paddle impellers, a result also reported by Wongjaikham et al. (2021).

However, for viscous fluids, such as biodiesel, the effect of the presence of baffle is less pronounced and, although it potentiates the effect of agitation and mixing, it is not significant compared to the others. This statement is confirmed by the experimental data, since the baffle effect was not a significant parameter for any of the evaluated routes. In addition, similar considerations were obtained by other authors, such as Peiter et al. (2020) who reported that the presence of baffle reduced the yield of esters in the first two minutes of the transesterification reaction of soybean oil and after that time it did not exert any further influence on the process.

For the methyl route (Figure 2b), only the stirring speed had a significant influence on the efficiency of the process. It was observed that increasing the stirring speed resulted in an increase of up to 15.4% in the biodiesel yield. The highest efficiency (93 ± 3.53%) was achieved when the stirring used was equal to 350 rpm, turbine-type impeller and baffles, test 8. This observed behavior may be a consequence of the better reactivity of the methylic route so that the chemical variables and the conditions of the transesterification reactions were sufficient to achieve high conversions into biodiesel, even though the mechanical conditions could be an unfavorable scenario for the blending process.

Figure 3 presents a summary of the percentage increase in biodiesel production as a function of the mechanical variables investigated in this study. According to this Figure, the biodiesel produced by the ethylic route had a more accentuated increase (+16.3 %) than the methylic route (+15.4 %)

indicating that the yield of esters using ethyl alcohol is more influenced by the study variables. These results agree with the discussion provided by Figure 2, which indicated a greater effect of significant variables on yield by the ethylic route compared to the methylic route.

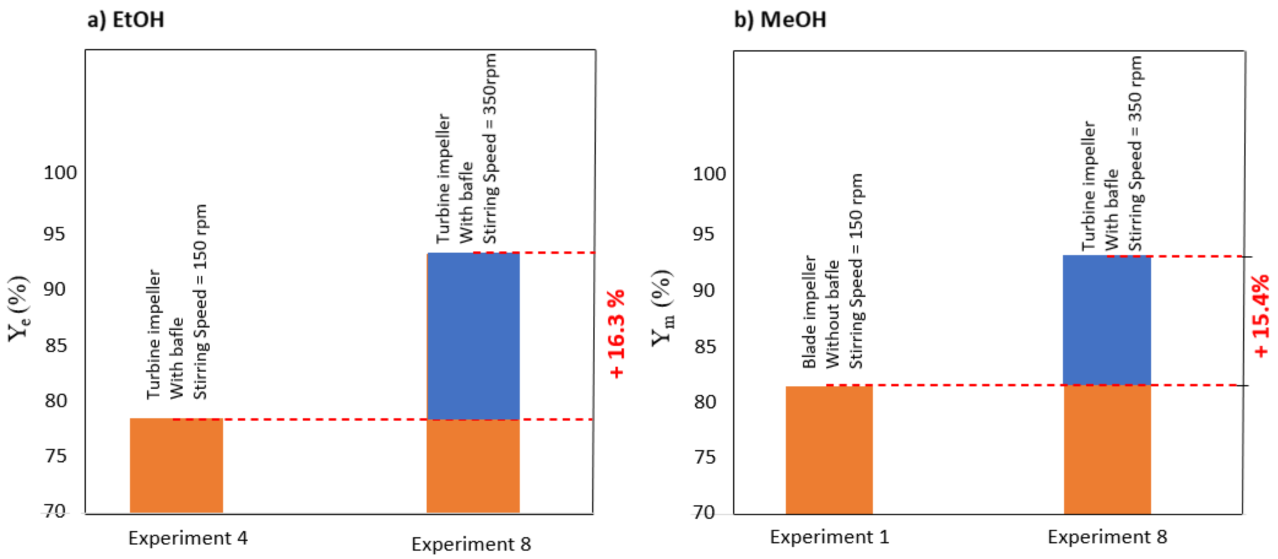

Figure 3. Percentage increase in biodiesel as a function of mechanical variables for a) ethylic and b) methylic routes.

The analysis provided by the Pareto chart and the variation between the maximum and minimum yields observed in Table 3 corroborates the confirmation of the relevant effect of mechanical variables on the yield in the transesterification reactions. However, it is worth mentioning that the higher yields obtained were also favored by the previously defined operating variables, such as temperature, molar ratio, reaction time, and amount of catalyst.

Based on the statistical analysis of the ester yield results, empirical models were obtained. For the ethylic route, the model in question provided a linear relationship between the yield and impeller use, stirring, and the interaction of the three factors for the ethylic route, according to Equation 2. For the methylic route, a model with fewer parameters was obtained, since only the stirring showed significance for confidence of 95 %, as shown in Equation 3.

$$Y_e(x_1x_2x_3) = 84.7625 + 2.8250x_1 + 2.4625x_2 + 2.5375x_1x_2x_3 \quad (2)$$

$$Y_m(x_1) = 86.7000 + 2.1375x_3 \quad (3)$$

As they are empirical models, the equations are suitable to describe the results only in the operational range of the experiments used as in the prediction. It is important to mention that the values of the variables x_1 and x_2 may only assume the values of -1 and +1, as they refer to the qualitative baffle variables and the types of impellers. Thus, the few parameters present in Equation 3, for the production of soybean biodiesel by the methylic route, indicate that for the established operating conditions, the yield variation is only dependent on the stirring speed. However, for other reactional conditions, this model will most likely not represent the real condition of the system, so continuous studies with increased levels of several variables are necessary.

In Figure 4, the values predicted by the empirical models and the values observed experimentally are compared and it is possible to evaluate the normal distribution of the experimental points around the line y=x, which corresponds to an ideal model.

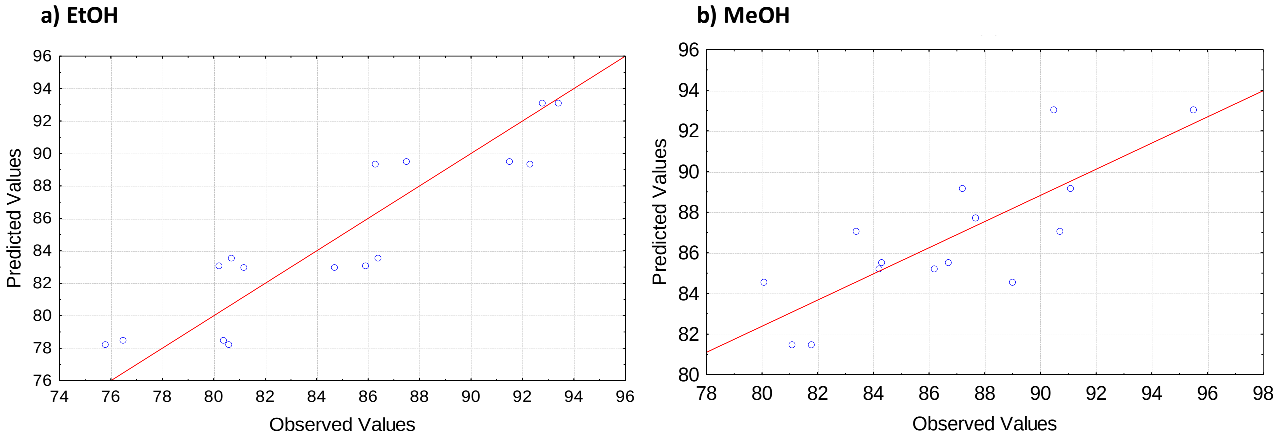

Figure 4. Predicted versus observed values for the yield response variable in esters by a) ethylic and b) methylic route.

It can be inferred from Figure 4 that the points are randomly arranged next to the line, which may be indicative of few model residuals, but also indicates that for this set of experimental data, the models were able to represent the yield trend as a function of mechanical variables.

For a more detailed analysis of the optimum point for biodiesel production, the response surface method was also used. The response surface shows how two factors influence the response and the direction in which a better response is obtained for the biodiesel yield in the transesterification reaction. Response surfaces for the methylic and ethylic routes are shown in Figure 5.

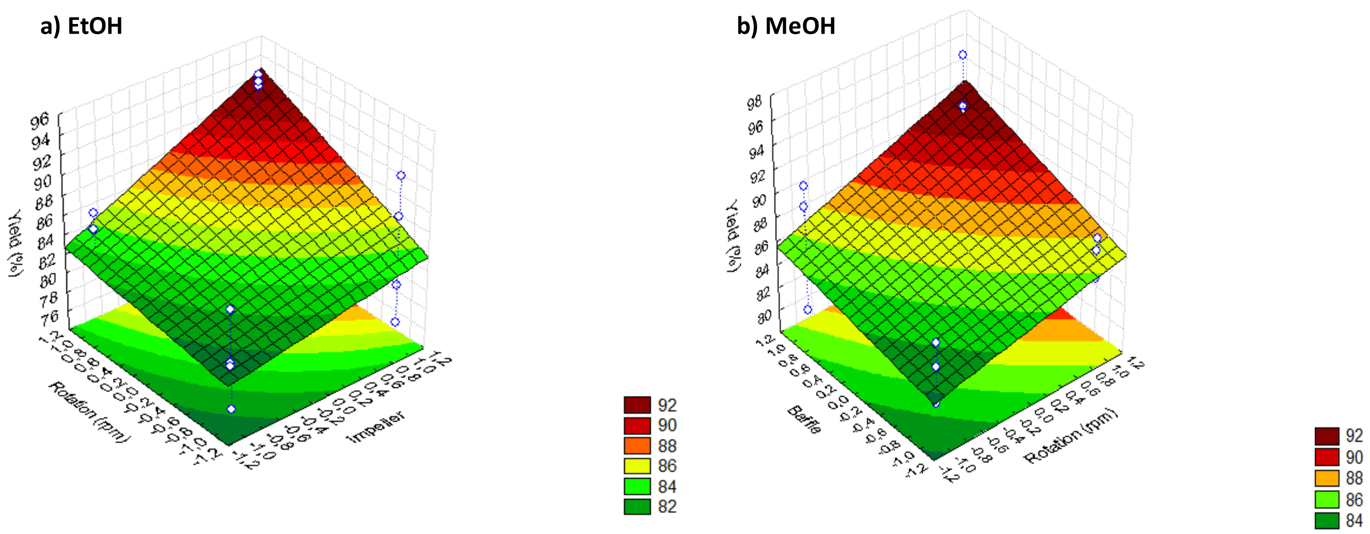

Figure 5. Response surface for a) ethylic and b) methylic route.

In the surface response (Figure 5), the intense red color region represents the reaction conditions that provided the highest conversions. In Figure 5a, the baffle variable was kept fixed, since this variable was not statistically significant. Thus, the variables considered were the type of impeller and stirring. The response surface suggests that the model tends to an optimum point of high performance when there is an increase in stirring speed and use of the turbine impeller. The increased stirring speed improves biodiesel yields due to the greater number of collisions between oil and alcohol molecules during the reaction time, thus favoring the transesterification kinetics. Furthermore, the best performance with the turbine impeller agrees with the literature (Peiter et al., 2020) and is related to a considerable reduction in the reaction time.

For the methylic route, the response surface (Figure 5b) related stirring, the only statistically significant variable according to Pareto, and, in the absence of another significant parameter, the baffle, which was the closest variable to the significance line. Analogously to the ethylic route, a trend towards an optimal point was noticed as the stirring variable reached higher levels of the design.

Conclusions

In this study, the factorial design was applied to determine the influence of the mechanical variables on the transesterification reaction of soybean oil via the methylic and ethylic routes. The results were discussed by statistical techniques such as Pareto chart and response surface methodology. The analysis of the results showed that to produce biodiesel via the ethylic route, the stirring speed and the type of impeller presented statistically significant effects on the reaction. While for the reactions via the methylic route, only the stirring was significant for the conversion of biodiesel esters. In terms of yield, the most significant results were for tests that used a turbine-type impeller, use of a baffle, and stirring speed of 350 rpm for the ethyl and methylic route.

This study demonstrates that the factorial design was efficient in the screening of significant variables applied in the optimization by the response surface method and can be a useful tool in the experimental data treatment that related more than two variables in a way that it was possible to evaluate the behavior of these individuals and their interaction relations and, finally, to predict the conditions required to obtain a higher yield in esters.

Thus, the results show the possibility to evaluate only the effect of the reactor's mechanical variables to optimize the biodiesel production process. These changes can be enough to provide a high yield of the process without the need for changes in chemicals, temperature, pressure and time, which may prove to be an alternative to reducing process costs. Although the present study has contributed to the discussions on the effect of mechanical variables on the transesterification reaction, it is understood that other system parameters can be considered for an even broader discussion on the topic.

As the next steps, variables such as reaction time, molar ratio, and temperature can be varied concomitantly to obtain a configuration that provides even higher yields. This would allow the simultaneous investigation of the effect of agitation and mixing, through the mechanical variables, combined with the reaction conditions, since the reaction parameters are strongly discussed in the literature. In this way, it will be possible to arrive at a statistical model that considers not only the reaction variables, as the literature usually discusses, but also the reactor configuration, so that these combined factors are considered to find the optimal conditions for maximum yield, which may also be of wide application in the design and operation of industrial plants.

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