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EXPERIMENTAL EVALUATION OF BIODIESEL PRODUCTION FROM SOYBEAN OIL BY MICROWAVE RADIATION METHODOLOGY

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Abstract. *Biodiesel production in the world needs new and more efficient production methodologies. The main producers of biodiesel in the world carry out the process in the conventional way, although several researchers have identified that the organic reactions produced by heating of the irradiation with microwaves, present reduced times, and similar yields than the conventional process. In this way, the main goal of this work consists in the development of a methodology to produce a biodiesel-type biofuel by microwave irradiation, using national raw material (soybean oil), methyl alcohol and potassium hydroxide as catalyst. An experimental design was carried out with two control variables, oil:alcohol molar ratio, and reaction time. The results showed that all efficiencies were above of 95%. Highlighting 3 experiments, biodiesel 3, with oil:alcohol molar ratio 1:12 and transesterification time of 2 minutes, in which a mass efficiency of 100,69% was obtained, biodiesel 4, with oil:alcohol molar ratio 1:6 and transesterification time of 2 minutes, with a mass efficiency of 97.9%, and biodiesel 6, with an oil:alcohol molar ratio of 1:12 and transesterification time of 0.34 minutes with an efficiency of 100%. The latter being the most promising result of the present work, showing a higher conversion within the parameters required by regulatory standards of the oil with methyl esters in a short time. In addition, for the three mentioned biodiesels, their physicochemical properties were evaluated, in which they presented values within the limits allowed by both national and international biodiesel standards. The gas chromatography mass spectrometry analysis (GC-MS) was also conducted, showing higher content of methyl esters. The energy analysis of biodiesel production using microwave irradiation technology was presented as well. In these conditions, it can be concluded that through the methodology developed in the present work it was possible to produce biodiesel with higher percentages of conversion to methyl esters, complying the standards for use in internal combustion engines.*

Keywords: *Biodiesel. Transesterification. Microwave irradiation. Mass performance. Chromatography*.

1. INTRODUCTION

Fossil fuels are one of the biggest sources of pollution in the world. The main efforts to mitigate contamination by the product of burning these fuels are not delivering the desired results, the increase in energy demand increases consumption, causing an increase in the greenhouse effect on the planet (Elango et al., 2019). In this way, some cleaner and renewable energy alternatives are being discussed and studied. (Muthukumaran et al., 2017). The implementation of renewable energies presents almost zero emissions of the main polluting gases, in addition to having the potential to meet energy demand. (Kim et al., 2015). Currently, research focuses its efforts on the development of new technologies to produce biofuels (Li et al., 2019). Different experimental works found in the literature have reported favorable increases in air decontamination by using biodiesel-type biofuels. Vegetable oil fuels are very attractive due to their environmental benefits, such as low sulfur and other polluting gases. (Santos et al., 2009).

Biodiesel is a type of renewable and biodegradable fuel produced from raw materials such as vegetable oils (such as soy, sunflower and canola oil) and animal fats. It is obtained through a process called transesterification, consisting of the reaction of these raw materials with an alcohol, usually methanol or ethanol, in the presence of a catalyst. The result is methyl or ethyl esters. These esters can be mixed with petroleum diesel in different proportions. (Nayab et al., 2022).

In addition, biodiesel has the advantage of being easy to apply in vehicles, as it does not require any modifications to the engine. (Smit et al., 2010). Biodiesel production by conventional heating requires more energy. In this way, alternatives have been sought to solve this problem, such as heating by microwave irradiation. (Li et al., 2019; Xiang et al., 2017), in which it is possible to considerably reduce the time of reactions and organic syntheses. This type of study is important because currently, Brazil is an important producer of biodiesel in the world, in addition, it has the largest reserves of raw materials, such as oils, alcohols, and others, contributing to the diversification of fuel production in the country. Brazil (Stevanato et al., 2020).

The factorial planning tool is a useful analytical strategy, and its main application lies in screening the most relevant variables of a given analytical system. After this screening process of the most significant variables, experiments are carried out that allow refinement and a better understanding of the system under study. (Muthukumaran et al., 2017). Within this context, data analysis along with experimentation is an essential procedure for understanding the tools, especially those aimed at optimizing processes. (Stafoggia et al., 2008). In the proposal of an experimental design, it is fundamental that it be able to provide exactly the type of information that the analyst wants. Thus, the most important activity is not in data analysis, but in designing the experiments in which these data are to be obtained. In this sense, the present work aims to experimentally evaluate the production of biodiesel from Brazilian soybean oil, through two control variables in the production of biodiesel, the heating time and the content of oil in alcohol, in addition to presenting the results of the different physical-chemical properties important for the use and future implementation in internal combustion engines.

2. MATERIALS AND METHODS

2.1 Materials

For the development of the project, the raw materials presented in Table 1 were used. In which the most relevant information of the reagents and the environmental conditions in the production of biodiesel is presented.

Table 1. Main information for the biodiesel production process.

Material	Reagent	Molecular weight (g/mol)	Density (g/ml)
Alcohol:	Methyl alcohol (99,8 %)	46	0,789
Oil:	Soybean Oil	871	0,93
Catalyst:	Potassium hydroxide (KOH)	56,11	--
Total reaction volume:	300 ml	--	--
Room temperature	20 °C	--	--
Atmospheric pressure	101325 kPa	--	--

2.2 Biodiesel Production

A biodiesel production methodology was developed using raw materials from Brazil, which reduces energy consumption and production time. Biodiesel was produced using microwave radiation. (Fia & Amorim, 2021), in the Figure 1. The experimental bench is presented; composed of an agitation system, the mixer is made of materials that do not interfere with microwave radiation, which are Teflon and glass, in addition, it has a cooling system composed of a heat exchanger, with the main objective of lowering the temperature of the gases produced by the chemical reaction inside the reactor, with water flow in the range of 10 to 15 degrees Celsius, which is controlled by a thermal bath. The transesterification reaction takes place inside a reactor previously loaded with the raw material, its shape is circular, with a flat bottom and a capacity of 2 liters, whose material is glass. The reactor heating source is a conventional microwave oven with a maximum operating power of 1400 W.

Figure 1. Prototype experimental biodiesel production bench.

The biodiesel production scheme is shown in Figure. 2. The experimental prototype has two raw material reactors, one initially with soybean oil preheated to 80°C, and the second reactor has a mixture of methanol and catalyst (KOH), previously mixed. The set features a glass head with three entries, the first is intended for the supply of raw material, the second to house the stirrer, and the third connects the system with the condenser, which recovers the alcohol evaporated during the heating process. This cooling system maintains the temperature of the water flow at 10°C, being driven by a submerged pump inside the thermal bath with a storage tank with a capacity of 15 Liters.

The mixing of the raw materials and the rapid heating of the microwave oven leads to a faster and more optimized transesterification. Once the reaction is finished, the biodiesel goes to the first decantation phase in which the glycerin is extracted. This process takes one hour, and involves placing the substance from the reactor inside a funnel with a capacity of 2000 ml. After separation, the biodiesel is purified with distilled water. In this procedure, the water is preheated to 90 °C, then, the biodiesel is placed in the separation funnel and then hot water is added, in proportions 1:2, the washing was done with 3 repetitions or until realizing that the water did not there were more particle trails. This is done with the aim of removing impurities and remains of the catalyst present in the biodiesel. With purified biodiesel, the physicochemical properties are determined using specific standards (Cavalcanti-Oliveira et al., 2015).

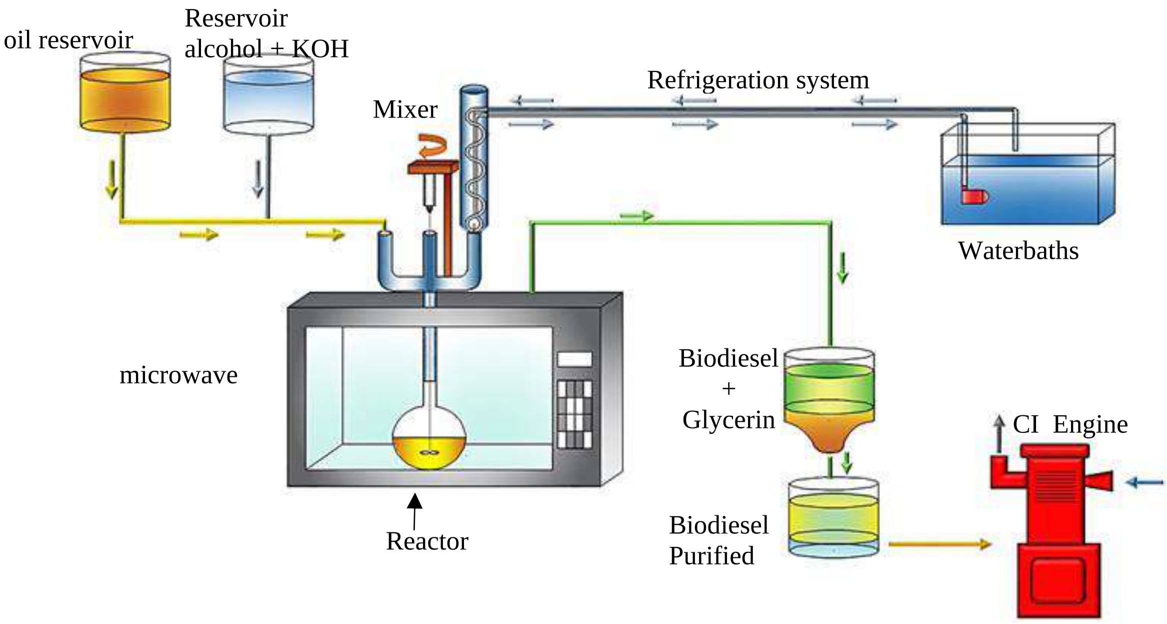

Figure 2. Schematic diagram of the experimental bench for biodiesel production using microwaves.

2.3 Experimental planning method

An experimental design was made to determine the best ratios for biodiesel production, Table 2 contains the factorial design for the biodiesel production variables with 11 experiments. As planned, it started with proportions of 1:4.76, with 300 ml of total solution. Subsequently, experiments were carried out with proportions 1:6, 1:9, 1:12, 1:13.2, obtaining complete transesterification with a mass ratio of 1% with the KOH catalyst based on the weight of the oil present in each experiment, obtaining better experimental results in the chosen oil:alcohol proportions. In addition, the time variation was the most important parameter in this study.

Another of the most important variables in biodiesel production is temperature. A constant oven reaction power was established for all tests at 60% of its nominal power of 1400 Watts, so the working power was 840 Watts, which represents a constant operating temperature of 70 °C. In addition, when the oil is loaded into the reactor, it is preheated for 1 minute at maximum power, raising the temperature of the soybean oil to 100 °C.

Table 2. Factorial design for biodiesel production ratios.

Variables	$-\sqrt{2}$	-1	0	1	$\sqrt{2}$
Molar ratio oil:alcohol	1:4,76	1:6	1:9	1:12	1:13,2
Time	0,34	2	6	10	11,65

On Table. 3. The factorial planning matrix for the biodiesel production variables is presented, carried out for the different experiments planned in this work, with a total of 11 experiments.

Table 3. Factorial Design Matrix for biodiesel production molar ratios.

Experiment	**Time (minutes)**		**Molar ratio oil:alcohol**	
0 (x3)	0	6	0	1:9
1	+1	10	+1	1:12
2	+1	10	-1	1:6
3	-1	2	+1	1:12
4	-1	2	-1	1:6
5	+	11,65	0	1:9
6	-	0,34	0	1:9
7	0	6	+	1:13,2
8	0	6	-	1:4,76

2.4 Characterization physicochemical properties of biodiesel

The standardization of the process for obtaining biodiesel by microwave radiation applied by the authors (Buasri et al., 2015; Xiang et al., 2017)present approaches for the characterization of before and after the transesterification reaction to verify the ranges of biofuel standards met by different international standards, comparing with other works (Lokman et al., 2015; Nguyen et al., 2021). As microwave radiation is used in organic reactions, such as biodiesel production, due to the acceleration of heating and higher concentration of reaction energy. These speeds up the chemical reaction due to selective absorption of energy at (Krishnan & Rajan, 2017), which generates a significant reduction in the transesterification reaction time and may present improvements in the physicochemical characteristics of the biofuel.

Continuing with the methodology used in measuring the properties of biodiesel, the biodiesel samples with the best characteristics of the transesterification reaction were measured, in addition, the characteristics of a standard fluid were measured to have a comparison with the literature, in which chose oleic acid. Subsequently, the physicochemical characteristics of soybean oil were measured. Table 4 presents the results obtained from the physicochemical properties in this work, with the standards of the Brazilian standard ANP 45/2014, the American standard ASTM D6751-15 Grade 1-B and 2-Bd, and the European standard EN14214 :2012.

Table 4. Biodiesel quality standards.

Parameter	**Unit**	**Brazil Resolution ANP 45/2014**	**U.S ASTM D6751-15 Grade 1-B e 2-Bd**	**European Union EN14214:2012**
density at 15ºC, min. - max.	kg/m^3	850 – 900	--	860 - 900

Dynamic viscosity at 40ºC, min. - max.	mm^2/s	3,0 - 6,0	1,9 - 6,0	3,5 - 5,0
Acid index, max.	mg KOH/g	0,5	0,5	0,5
Iodine index, max,	g I_2 /100 g	--	--	120
Ester contente, min.	% Mass	96,5	96,5	96,5

3. RESULTS AND DISCUSSION

3.1 Analysis Factorial design of biodiesel production relationships

The results obtained in the tests that constituted the factorial design are presented in Table 5. It is observed that there was little significant variation in the percentages of methyl esters in the different reaction conditions used. It was also identified that the values ranged from 93.13% to 100.69%. However, it is important to differentiate the yield of the mass ratio in the biodiesel when washed and compared to the unwashed one. Thus, washed biodiesel was chosen for data analysis.

Table 5. Biodiesel quality standards.

test	Time (minutes)	molar ratio	Yield raw (%)	Yield washed (%)
0	6,00	1:9	106,04	97,47
1	10,00	1:12	108,35	101,32
2	10,00	1:6	97,18	93,13
3	2,00	1:12	111,5	100,69
4	2,00	1:6	102	97,9
5	11,65	1:9	108,36	95,44
6	0,34	1:9	105,7	100,7
7	6,00	1:13,2	110,78	95,09
8	6,00	1:4,76	96,57	94,09
Repetition 0	6,00	1:9	107,85	98,78
Repetition 1	6,00	1:9	104,57	97,73
Repetition 2	6,00	1:9	105,53	97,13

When comparing reactions with lower alcohol content, specifically tests 2, 4 and 8, which have a 1:6 molar ratio, the time is longer for test 2, with 10 minutes of reaction compared to 2 minutes test 4, in addition, showed a difference in yields, with 4.77%, which represents an important influence on the reaction time for the proposed content. Likewise, tests with a higher alcohol content were analyzed, in which the yields of the transesterification reaction with methyl alcohol and the catalyst content, in this case potassium hydroxide, did not represent a large variation in the reactions. This represents an efficient constant production of biodiesel with the presented characteristics, keeping the potassium hydroxide constant at 1% in weight of the soybean oil. The most important result in the experimental planning was the production of biodiesel in a short time, which translates into a low production cost, in addition, the yield variation was not significant, but, making a comparison of tests 1 and 3, the which have the same 1:12 molar ratio with a high methyl alcohol content, both showed similar transesterification reaction yields with 101.32% and 100.9% conversion, but the time showed a noticeable change. Test 3 performed the reaction in 2 minutes and test 1 in 10 minutes, in the same way that tests 2 and 4 presented reaction times of 10 and 2 minutes, respectively, with the same reaction percentages. These results indicated that the residence time of the reaction in the microwave oven with the variable response of the molar ratio became the most important factors, since the reaction mixture remains longer under favorable reaction conditions, in addition to increasing the effective shocks. between the reacting molecules, allowing to reach higher conversion values. These results are consistent with those obtained in the literature, where longer residence times contributed to higher ester yields. (Nayab et al., 2022).

To complement the analysis of the results, as well as optimize the conditions of the transesterification reaction using microwave irradiation heating technology, it is necessary to quantify the influence of each independent variable on the response variable, as well as their interactions and their quadratic terms. For this, a multiple regression was performed using the Statistic software, obtaining an empirical equation that represents the variation in the ester content in the transesterification reaction, as a function of the independent variables studied. The equation obtained for the grouped data, expressed in coded variables, is given by the Equation (1):

$$BO\ (\%)\text{: } 97{,}4357-1{,}4489X+1{,}5534\cdot Y+0{,}8061XX+1{,}35XY-0{,}9443YY \quad (1)$$

It presented the square of the multiple correlation coefficient (R2) equal to 0.99, indicating that 99% of the variability of the data was obtained, with this value and knowing that the residues were random and independently distributed, it is observed that the equation is statistically consistent. The empirical equation shows that the variables reaction time (X) and alcohol:oil molar ratio (Y) are significant and affect the yield in esters, as well as the interaction between residence time and molar ratio (XX), in addition, such as the interaction between reaction time and molar ratio (XY) and the interaction between temperature and oil:alcohol molar ratio (YY). The values of the coefficients obtained for each variable confirm the previously discussed trends. For the variable oil:alcohol molar ratio (Y), positive coefficients were obtained, showing that they positively affect the system response, while for the reaction time (X), a negative coefficient was obtained, indicating that this variable negatively affects the ester content. Through Equation (1) obtained, it is also possible to state that the reaction time is the variable that most affect the response since the coefficient value obtained was the highest (11.65) and oil:alcohol molar ratio (1:9). In Fig. 3, the response surface for the yield of biodiesel produced as a function of the variables reaction time and oil:alcohol molar ratio is presented.

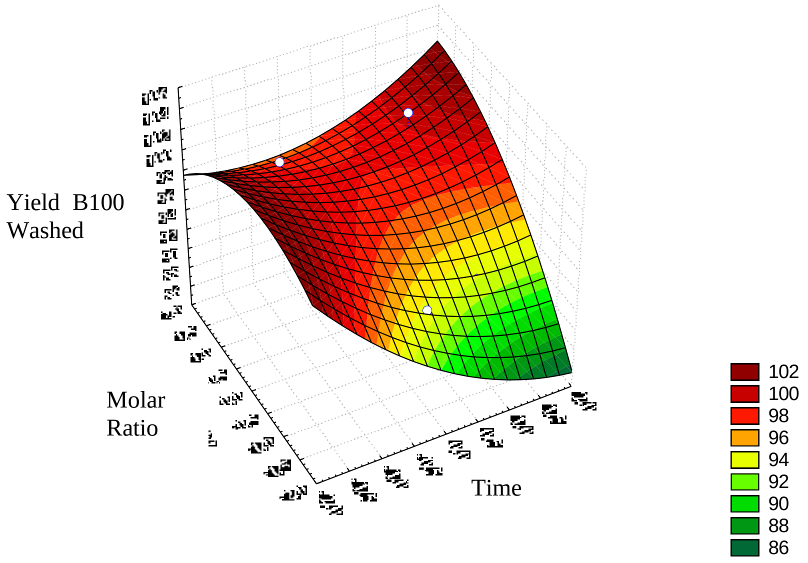

Figure 3. Response surface for the produced biodiesel yield

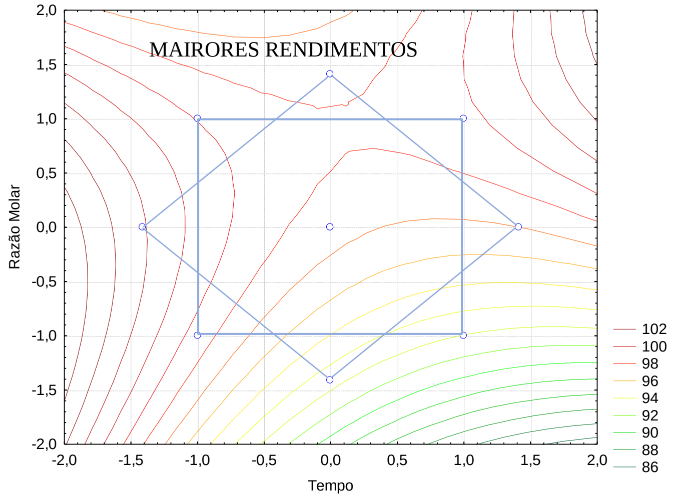

Figure 4. Contour graph obtained for the optimization of variables, transesterification time and molar ratio.

With the help of Equation (1), and Figure. 3, it was possible to identify the region of maximum yield of the biodiesel transesterification reaction by microwave irradiation technology, as can be seen in Fig. 4, finding the best parameters for the transesterification times of 2 minutes for the molar ratio experiments (1:6, 1:9, 1:12) these experiments maintain a yield superior to 97% of reaction in the transesterification, already reported by some authors (Geris et al., 2007; Waudby & Zein, 2021), but in this context, the results of low transesterification time are important, which are surprising and with few reports in the literature found on this type of research work, in the case of the time of 0.34 minutes.

3.2 Physicochemical properties of biodiesel

In this section, the results of the characterization of the 3 tests with the lowest reaction time are presented, in this case, tests 3, 4, and 6, in addition, the characterization of soybean oil, the main raw material in the production of biodiesel and a standard for checking the tests, oleic acid.

Density

The density found in this work is within the standards of the Brazilian and international norm EN on biofuels, with a maximum value of 0.9092 g/mL. It is vitally important to verify this parameter, which directly influences the combustion of the engine, since a low specific mass causes delays in the ignition of the combustion and this, in the long term, considerably deteriorates the integrity of the engine. (Krishnan & Rajan, 2017). In Table 6, the results of the specific mass measured in the samples are presented.

Table 6. Results of biodiesel specific mass and samples.

Samples	Biodiesel 3	Biodiesel 4	Biodiesel 6	Soybean oil	Oleic acid
Density (g/mL)	0,8719 ± 0,0004	0,8751 ± 0,0002	0,8742 ± 0,0003	0,9092 ± 0,0004	0,8856 ± 0,0002

Kinematic and dynamic viscosity

It is desired that biofuels have a low viscosity, otherwise, they could affect the operation of the fuel injection system in the internal combustion engine. Furthermore, the viscosity of the oil acts as a parameter in determining the time in the transesterification reaction, establishing a relationship between viscosity and biodiesel conversion. (Stevanato et al., 2020). Biodiesel 6 presented a kinematic viscosity value of 5.2952 mm^2/s, whose value is within the ranges established by the ASTM standard (1.9-6.0 mm^2/s) which is optimal for engine operation, biodiesel 6 is the sample with a molar ratio of 1:9 so with a time in the transesterification oven of 0.34 seconds. Biodiesel 3 obtained not-so-favorable results concerning viscosity, whose value obtained was 6.2808 mm^2/s, thus, it was identified that this parameter was outside the range established by the standard. This experiment corresponds to a sample with a molar ratio of 1:12 and 2 minutes of transesterification in the oven. In Table 7, the main results of the calculated viscosities are presented. Biodiesel 6 presented a kinematic viscosity value of 5.2952 mm^2/s, whose value is within the ranges established by the ASTM standard (1.9-6.0 mm^2/s) which is optimal for engine operation, biodiesel 6 is the sample with a molar ratio of 1:9 so with a time in the transesterification oven of 0.34 seconds. Biodiesel 3 obtained not so favorable results with viscosity, whose value obtained was 6.2808 mm^2/s, thus, it was identified that this parameter was outside the range established by the standard. This experiment corresponds to a sample with a molar ratio of 1:12 and 2 minutes of microwave transesterification.

Table 7. Results of dynamic and kinematic viscosity of biodiesel and samples.

Samples	Biodiesel 3	Biodiesel 4	Biodiesel 6	Soybean oil	Oleic acid
Kinematic viscosity (mm^2/s)	6,2808 ± 0,0401	5,6014 ± 0,0664	5,2952 ± 0,0484	68,4380 ± 0,0185	37,2580 ± 0,0906
dynamic viscosity (mPa·s)	5,5325 ± 0,0515	0,8751 ± 0,0002	0,8742 ± 0,0003	59,7780 ± 0,1609	33,2440 ± 0,0785

Acid index

The different samples presented for the measurement of the acidity index, presented favorable data, the results reported for acidity values of the soybean oil used for the production of biodiesel are in accordance with the quality limits established for use as raw material in the production of biodiesel, in all experiments the values of the acidity index are within the standards of the Brazilian standard (ANP 0.5 mg KOH/g) and the international standard (EN, ASTM 0.5 mg KOH/g). In Table 8, the results of the acid index of the chosen samples are presented.

Table 8. Biodiesel acid index results and samples.

Samples	Soybean oil	Oleic acid	Biodiesel 3	Biodiesel 4	Biodiesel 6
Index Acid (mg KOH/g)	0,2336 ± 0,0009	244,2939 ± 0,6130	0,3379 ± 0,0627	0,2345 ± 0,0017	0,1165 ± 0,0010

Iodine Index

According to (Geris et al., 2007) the health requirements for vegetable oils and fats require that, for an oil to be marketed in Brazil, it must comply with the safety parameters for human consumption, being necessary that they are between the values of 120 and 143 g of iodine / 100g. Thus, the low amount of unsaturation in soybean oil is attributed to adulterations made with fats or to hydrolysis processes and oxidative rancidity caused by long periods of storage, as shown by the study, in which it was identified that after 180 days of storage, the iodine value of virgin soybean oil dropped from 123 to 115.41 g of iodine / 100g.

In Brazil, there is no requirement regarding the iodine index limits for biodiesel, so the current legislation only requests the investigation of this parameter (ANP Resolution No. 14 of 11.05.2012). According to the European Standard, the iodine value must be close to 120 g I2 100 g^{-1}. The results obtained in the measurement of the iodine index are considerably positive, the biodiesel samples were within the limits established by the European standard, in Table 9. the results of the iodine index of the chosen samples are presented.

Table 9. Results of iodine index of biodiesel and samples.

Samples	Soybean oil	Oleic acid	Biodiesel 3	Biodiesel 4	Biodiesel 6
Iodine index (g I_2/100 g)	87,84 ± 0,8000	82,2000 ± 0,6500	102,3700 ± 0,8500	112,5180 ± 0,8500	103,2100 ± 0,8500

Refraction index

The refraction index, the results of this analysis are compared with (Lima et al., 2010), in which for a soybean oil biodiesel, a refractive index of 1.47 was found, whose result is in agreement with those obtained in this work. . In Table 10, the results of the refractive index of the chosen samples are presented.

Table 10. Results of the biodiesel refraction index and the samples.

Samples	Soybean oil	Oleic acid	Biodiesel 3	Biodiesel 4	Biodiesel 6
Refraction index	1,4755 ± 0.,0006	1,4640 ± 0,0021	1,4565 ± 0,0006	1,463 ± 0,0046	1,4658 ± 0,0023

Turbidity

Turbidity is a physical property of liquid solutions which is associated with the transparency of these fluids. The presence of suspended materials interferes with the passage of light through the fluid, leading to a reduction in the turbidity parameter. In biodiesel, greater turbidity refers to the presence of small, dissolved water droplets or fine particles of suspended contaminants. Table 11 shows the turbidity results of the chosen samples.

Table 11. Results of turbidity of biodiesel and samples.

Samples	Soybean oil	Oleic acid	Biodiesel 3	Biodiesel 4	Biodiesel 6
Refraction index	0,02 ± 0,01	0,02 ± 0,01	0,02 ± 0,01	0,02 ± 0,01	0,02 ± 0,01

3.3 GC-MS Chromatography Analysis

Biodiesel produced by microwave irradiation methodology, with the best physical-chemical characteristics presented so far, are the experiments of low reaction time, so to guarantee its conversion from oil to methyl esters and to determine the content of impurities, it was GC-MS characterization test was performed. In Figures. 5.a,b,c, the chromatograms are presented for the three chosen biodiesel samples, in this case sample (a) -biodiesel 3, sample (b) -biodiesel 4, sample (c)-biodiesel 6. In Table 11, the quantification of the content of methyl esters of total fatty acids in biodiesel samples is presented, according to the EN14103 standard, which determines the quantification of esters in biodiesel.

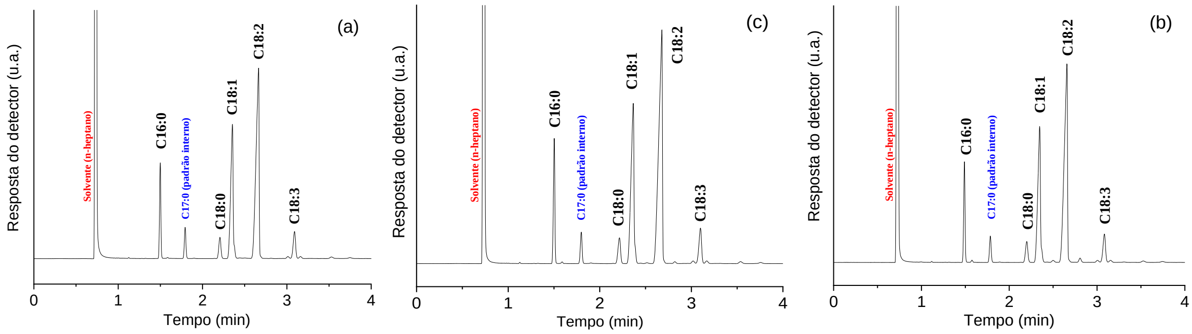

Figure 5. Chromatograms obtained for the analyzed biodiesel samples: (a) (biodiesel 3); (b) (biodiesel 4); and (c) (biodiesel 6).

Table 11. Total fatty acid methyl ester content of biodiesel samples, EN14103 standard.

Samples	% FAME
Biodiesel-1 (Experiment 3) Molar ratio oil:alcohol 1:12	97,68
Biodiesel-2 (Experiment 4) Molar ratio oil:alcohol 1:6	84,43
Biodiesel-3 (Experiment 6) Molar ratio oil:alcohol 1:9	90,66

The results represent an important indicator of the conversion of methyl esters in the transesterification reaction carried out by the planned methodology. All three samples have an optimal conversion. As a result, it was identified that the higher the molar ratio, the higher the content of methyl esters in biodiesel. In this case, the sample with the lowest content was sample 2, it had a content of 1:6. The results in samples 1 and 3 showed a molar ratio of 1:12 and 1:9, respectively, the higher the oil:alcohol molar ratio, the greater the complete conversion. The EN 14214 and ANP 07/2008 standards require a minimum free ester content of 96.5% for the results obtained. Thus, only the sample of biodiesel 3 with molar ratio oil:alcohol 1:12 complies with the requirement.

4. CONCLUSIONS

The Design of Experiments was presented as a tool of great importance for the study of biodiesel production with microwave irradiation. In this approach, not only the effects of individual variables are considered, but also their interactions. The two variables defined for the planning carried out, reaction time and oil:alcohol molar ratio, proved to be significant in the content of methyl esters. The different tests showed excellent performance in the transesterification reaction, with a yield above 95%, the variable reaction time proved to be the most influential one for tests 3, 4, and 6 with reaction times of 2 minutes and 0, 34 minutes.

The physical-chemical properties of biodiesel present properties within the limits allowed in the ABNT standard and international standards for all the analyzed characteristics (density, specific mass, dynamic and kinematic viscosities), with this it is possible to guarantee an excellent injection in the diesel engine. internal combustion. The other chemical parameters calculated, such as the iodine value, the refractive index, and the acidity index, showed adequate development with the use of the KOH catalyst in the indicated proportions (1% in weight in oil), whose result is in agreement with.

The production of biodiesel with microwave irradiation presents good results in a short time. Three of them were chosen for chromatography analysis, Biodiesel 3 (molar ratio 1:12, time 2 minutes and yield 100.69%), in which the best amount of methyl esters was observed with 97.68%, that is, a higher amount of biodiesel presented, the biodiesel experiment 4 (molar ratio 1:6, time 2 minutes and yield 97.9%) which presented an amount of 84.43% of methyl esters, the biodiesel experiment 6 (molecular ratio 1 :9, time 0.34 minutes and yield 100.7%) showed 90.66% amount of methyl esters. This was the sample with the shortest reaction time and with an excellent amount of biodiesel.

5. ACKNOWLEDGEMENTS

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6. REFERENCES

Buasri, A., Rattanapan, T., Boonrin, C., Wechayan, C., & Loryuenyong, V. (2015). Oyster and pyramidella shells as heterogeneous catalysts for the microwave-assisted biodiesel production from jatropha curcas oil. *Journal of Chemistry*, *2015*. https://doi.org/10.1155/2015/578625

Cavalcanti-Oliveira, E. D., Silva, P. R., Rosa, T. S., Moura, N. M. L., Santos, B. C. P., Carvalho, D. B., Sousa, J. S., Carvalhinho, M. T. J. E., Castro, A. M., & Freire, D. M. G. (2015). Methods to prevent acidification of Macaúba (Acrocomia aculeata) fruit pulp oil: A promising oil for producing biodiesel. *Industrial Crops and Products*, *77*, 703–707.

Elango, R. K., Sathiasivan, K., Muthukumaran, C., Thangavelu, V., Rajesh, M., & Tamilarasan, K. (2019). Transesterification of castor oil for biodiesel production: Process optimization and characterization. *Microchemical Journal*, *145*, 1162–1168.

Fia, A. Z., & Amorim, J. (2021). Heating of biomass in microwave household oven - A numerical study. *Energy*, *218*.

Geris, R., Alessandra Carmo dos Santos, N., Andrade Amaral, B., de Souza Maia, I., & Dourado Castro José Roque Mota Carvalho, V. (2007). Biodiesel de soja-reação de transesterificação para aulas práticas de química orgânica. Em *Quim. Nova* (Vol. 30, Número 5).

Kim, S. E., Lim, Y. H., & Kim, H. (2015). Temperature modifies the association between particulate air pollution and mortality: A multi-city study in South Korea. *Science of the Total Environment*, *524–525*, 376–383.

Krishnan, R. Y., & Rajan, K. S. (2017). Influence of microwave irradiation on kinetics and thermodynamics of extraction of flavonoids from Phyllanthus emblica. *Brazilian Journal of Chemical Engineering*, *34*(3), 885–899.

Li, K., Chen, G., Chen, J., Peng, J., Ruan, R., & Srinivasakannan, C. (2019). Microwave pyrolysis of walnut shell for reduction process of low-grade pyrolusite. *Bioresource Technology*, *291*.

Lima, A. L., Lima, A. P., Portela, F. M., Santos, D. Q., Neto, W. B., Hernández-Terrones, M. G., & Fabris, J. D. (2010). Parâmetros da reação de transesterificação etílica com óleo de milho para produção de biodiesel. Em *Artigo/Article Artigo/Article Ecl. Quím* (Vol. 35, Número 4).

Lokman, I. M., Rashid, U., & Taufiq-Yap, Y. H. (2015). Production of biodiesel from palm fatty acid distillate using sulfonated-glucose solid acid catalyst: Characterization and optimization. *Chinese Journal of Chemical Engineering*, *23*(11), 1857–1864.

Muthukumaran, C., Praniesh, R., Navamani, P., Swathi, R., Sharmila, G., & Manoj Kumar, N. (2017). Process optimization and kinetic modeling of biodiesel production using non-edible Madhuca indica oil. *Fuel*, *195*, 217–225.

Nayab, R., Imran, M., Ramzan, M., Tariq, M., Taj, M. B., Akhtar, M. N., & Iqbal, H. M. N. (2022). Sustainable biodiesel production via catalytic and non-catalytic transesterification of feedstock materials – A review. *Fuel*, *328*.

Nguyen, T. N., Khoa, N. X., & Tuan, L. A. (2021). The correlation of biodiesel blends with the common rail diesel engine's performance and emission characteristics. *Energies*, *14*(11).

Santos, F. F. P., Rodrigues, S., & Fernandes, F. A. N. (2009). Optimization of the production of biodiesel from soybean oil by ultrasound assisted methanolysis. *Fuel Processing Technology*, *90*(2), 312–316.

Smit, R., Ntziachristos, L., & Boulter, P. (2010). Validation of road vehicle and traffic emission models - A review and meta-analysis. Em *Atmospheric Environment* (Vol. 44, Número 25, p. 2943–2953).

Stafoggia, M., Schwartz, J., Forastiere, F., & Perucci, C. A. (2008). Does temperature modify the association between air pollution and mortality? A multicity case-crossover analysis in Italy. *American journal of epidemiology*, *167*(12),

Stevanato, N., Ferreira de Mello, B. T., Massa, T. B., & da Silva, C. (2020). Potencial do nabo forrageiro para biodiesel: extração do óleo e produção de ésteres. *Revista UNINGÁ Review*, *35*, eRUR2872.

Waudby, H., & Zein, S. H. (2021). A circular economy approach for industrial scale biodiesel production from palm oil mill effluent using microwave heating: Design, simulation, techno-economic analysis and location comparison. *Process Safety and Environmental Protection*, *148*, 1006–1018.

Xiang, Y., Xiang, Y., & Wang, L. (2017). Microwave radiation improves biodiesel yields from waste cooking oil in the presence of modified coal fly ash. *Journal of Taibah University for Science*, *11*(6), 1019–1029.