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Experimental analysis of biodiesel synthesis from palm kernel oil: empirical model and surface response variables

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Abstract

This work studied the transesterification reaction of palm kernel oil to produce Biodiesel FAME, using as catalyst KOH incorporated as a potassium methoxide intermediate. The catalytic tests were performed modifying representative variables such as reaction temperature (°C), methanol/oil molar ratio, and catalyst content (%KOH). The experimental data were adjusted to a linear empirical model, finding that the best experimental condition was observed at 50 °C with a methanol/oil ratio of 5.5 and a% KOH of 0.8. Finally, the FAME was characterized by FTIR spectroscopy, gas chromatography, and ASTM quality control techniques for analysis of cold properties, transport properties, and combustion properties. The reaction rate was determined and a reaction mechanism was proposed based on the experimental results.

Keywords Biodiesel · Yield · Empirical model · Response surfaces · KOH

Introduction

Fossil fuels are the main causes of the excessive 46% increase in the atmospheric concentration of carbon dioxide that reached values of 410 ppm between 1850 and 2018. In consequence, some options have been proposed to reduce the GHG

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emissions associated with the transport sector, with participation in the biofuels market, which has seen a significant increase, although it is still a minority. Different routes have been researched for the production of alternative fuels from biomass for different applications and sectors, whether from physical, chemical, thermochemical or biotechnological processes [1–9]. The fermentation of sugars to obtain fuel alcohol and the transesterification of vegetable oils to produce biodiesel have been the most successful processes in industrial and commercial scale-up. It is also worth noting the recent impulse of the hydroprocessing of fats and lipids for the generation of hydrocarbons that can completely replace those derived from petroleum. However, the main focus has been on the use of these biofuels in the land transport sector, more specifically in the automotive sector.

Transesterification is a low-cost technology, commercially available and easy to assemble, but the characteristics of the biodiesel obtained are very sensitive to the type of oil used [6, 7]. This can be observed in Fig. 1, which shows that the global transesterification reaction is presented when the methyl esters of fatty acids or FAME are the product of interest of the reaction between a triglyceride and methanol, and is homogeneously catalyzed by potassium hydroxide [10, 11].

The main factors that affect the transesterification reaction are temperature, nature of the alcohol, alcohol/oil molar ratio, type and concentration of the catalyst, purity of the reactants, and stirring speed [12]. The most widespread transesterification process that has been industrialized uses homogeneous catalysts. Several researches have showed the advantages of using homogeneous catalysis, in which the conditions of temperature, alcohol/oil ratio and catalyst load have been optimized [13–15], but it requires a higher temperature and reaction time, which causes a high energy cost.

When mixed in a certain proportion with the fossil fuel, the methyl ester chains barely affect the functional properties of the fuel, since hydrocarbons lack the oxygen present in the FAME. This biofuel could be used in the aviation sector to partially replace Jet Fuel A1, as previous research has achieved substitutions of up to 20% with satisfactory results from the operational point of view [11, 16–20]. However, mixtures tend to substantially modify their fluidity, especially the elevation of

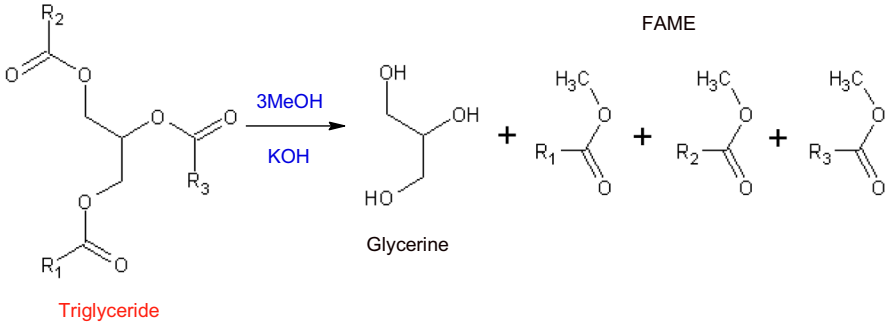


Fig. 1 Schematic representation of triglycerides transesterification using Methanol and KOH. Typical conditions: 60 °C, 1 h, atmospheric pressure

the freezing point, which leads to non-compliance with the established standards. This means that the cold properties (freezing point and cloud), critical to air safety, can be improved [21].

Currently, one of the major uses of vegetable oils, besides food and cooking, is in the production process of conventional biodiesel by transesterification, which is a known, commercially available and low-cost technology that has grown notoriously in the last 10 years in Colombia. This biofuel has an application for the land transport sector operating with Motor Fuel Oil (or ACPM as it is known in Colombia) [10], which uses palm oil. However, this process is not profitable at the industrial level, and has been commercialized and industrialized thanks to subsidies from countries that have been working against climate change. The problem lies in the high cost of essential raw materials like oils, which compete with the food sector, even though about 20% of oil production is used in non-food applications. Coconut oil, palm oil, and palm kernel oil are the main raw materials for the manufacture of oleochemicals [22].

Palm kernel oil is extracted by mechanical pressing or using solvents such as hexane from the palm kernel. From the almost 230 million tons of crude oil produced worldwide, about 3.5% corresponds to palm kernel oil (about 8 Mt), where Colombia contributes with 1.4% (almost 0.1 Mt). The average annual growth rate is 293 kt and 4.3 kt of crude palm kernel oil at the worldwide and at the national level, respectively. Crude palm oil corresponds to approximately 2% of the fruit and is one-tenth of the crude palm oil. In terms of total oils, palm kernel oil accounts for an average of 8% of production, 3% of consumption, and 16% of exports. It shows an annual increase of 36% in production, 20% in consumption and 59% in exports. Although the price of crude palm kernel oil is higher than the palm oil, the difference has been narrowing in recent years [1]. Recently, due to the implementation of policies that encourage the growth of areas cultivated with palm, within the biofuels program [23], an oversupply of products derived from palm, such as palm kernel oil, is being generated [24].

Regarding biofuels from oils, a biodiesel prepared from palm kernel or coconut oil can have a fairly high cetane number compared to other oils, which can make it attractive for biodiesel production, thanks to the present unsaturations [25]. Additionally, palm kernel oil contains a higher proportion of glycerides (tri, mono, and di) as well as fatty acids from short acyl chains than palm oil, so the demand for lauric acid in the country is replaced by palm kernel oil [26, 27]. This makes it attractive for applications other than those in the food industry, such as pharmaceuticals, cosmetics, fine chemicals, etc. [22, 28].

As the aeronautical sector is responsible for 2% of CO_2 emissions, 10% of fuel consumption worldwide, and 30% of air transport operating expenses are for fuel, the incorporation of biofuels emerges as a possibility [29–31]. To generate these substitutes, it is possible to use routes such as hydroprocessing to obtain biokerosene [2, 32–36], or transesterification (cheaper than the previous one, as it happens under moderate conditions of temperature and pressure) to produce mainly conventional FAME biodiesel of fatty acids of 12 and 14 carbons, compared with the 16 and 18 carbons generated with palm oil [10, 37, 38]. This means that the cold properties (freezing point and cloud) critical to air safety can be improved [21]. As a result, this biodiesel can

potentially become a bio-jet that partially replace Jet Fuel A1 in aviation fuel mixtures [39].

Studies on the use of FAME as an aviation fuel have been carried out, generating satisfactory results in up to 20% of the mixture [11, 16–20]. The Colombian Air Force has been a pioneer in conducting ground tests using Jet Fuel A1 mixtures with up to 50% FAME of palm oil in tubojet and tubohelix engine benches. However, there is still no certainty about the cold behavior of the mixture to give security to an air test, since the freezing point and cloud properties may not be within the specifications of the standard [40].

The literature reports that the optimization of the process conditions, the mechanism and the kinetics for the transesterification of palm kernel oil are limited compared with the information available for palm oil. The effect of the KOH concentration was studied but keeping constant the temperature at 60 °C and an ethanol/oil molar ratio of 3 in a time of 120 min. The highest yield obtained was 95.7% with 1% [41]. In a similar study, under the same conditions with 1% KOH, the optimal condition for the ethanol/oil molar ratio is 3 with a yield of 96% [42]. In most of the oil transesterification studies with both homogeneous and heterogeneous catalysts, the temperature, the alcohol/oil molar ratio and the amount of catalyst are mainly studied as the responsible variables that highly affect the optimization of the yield of FAME [43].

Thus, the current research aims to explore as first step the production of conventional FAME biodiesel from palm kernel oil transesterification, looking for the optimal conditions that allow the highest yield by varying the temperature, the molar ratio methanol-oil and the catalyst concentration. The biodiesel produced will be characterized. In subsequent works, the cold behavior of this generated biodiesel mixed in different proportions with Jet Fuel A1 will be evaluated to produce the necessary volume of biofuel required for the bench tests [44]. Future study of the reaction kinetics in bench tests must also be carried out.

Material and methods

Palm oil kernel characterization

In the development of experimental procedure, the following reagents and raw materials were used: refined palm kernel oil with an index 230–254 mg KOH/g oil, an iodine index of 17–19 and acidity index of 0.1% as acid. Besides, methanol for analysis was from the brand MERCK with CAS number 67-56-1, grade ACS.ISO, Ph Eur > 99.9% density at 20 °C of 0.791–0.793 g/cm^3, acidity < 0.0002 and alkalinity < 0.0002. Potassium hydroxide CAS number 1310-58-3, grade ACS was also used.

Experimental plant design

The experimental planning was designed from the selection of the main variables for biodiesel production, according to some works reports in the literature [45–47]. The reaction temperature, MeOH/Oil molar relationship and %KOH as

catalysts were selected as factors of experimental planning, performing aleatory experiments with the aim to evaluate the combined effects.

The levels for each factor were defined according to the review of other similar researches. In general, the temperature for reactions with a basic catalyst is close to the boiling point of alcohol, that is, between 40 and 60 °C. This is because the boiling point of methanol is 64.5 °C, so the reaction remains homogeneous at close temperature. At a higher temperature, the reaction not only lasts less time due to the higher speed, but there is also a higher conversion of biodiesel [48–52].

Regarding the alcohol/oil ratio, if it is less than the stoichiometric value (3), the conversion and productivity of methyl esters is very low. Considering that an excess of alcohol shifts the balance towards the formation of esters (right), the ratios higher than the stoichiometric ratio improve the conversion and the yield. However, ratios greater than twice the stoichiometric ratio (value of 6) do not significantly improve performance, instead they hinder the operations of separation of glycerol and purification of biodiesel [49, 50, 53].

Finally, when the concentration is less than 0.2%, the amount of alkaline catalyst makes the reaction rate very low. The concentration of the catalyst affects the conversion of biodiesel positively when it increases from 0.2 to 1% by mass, and negatively when there are high concentrations of catalyst because it favors the saponification reaction instead of the transesterification reaction. For example, when the KOH concentration is 1% mass, a 95.8% conversion of palm kernel oil is obtained; but the conversion is 85.2% when the KOH concentration is 1.25% [12, 41, 49].

A mixed level 3^3 factorial experimental design was made according to Table 1.

The treatment of the experimental data and the development of an empirical model was based on the following steps:

a. The accurate selection of response variables; usually, these variables are related to conversion and selectivity of the reaction, as well as the yield towards the production of the desired compound
b. Identification of the process variables that influence the selected response variable (YBD Yield); these variables and its levels are tabulated in Table 1.

Table 1 Conditions for catalytic tests reaction

Variable	Symbol	Levels	Values
Relationship MeOH/Oil	X_{RM}	− 1	3
		0	4.5
		1	6
KOH Content (%)	X_{KOH}	− 1	0.5
		0	1
		1	1.5
Reaction Temperature (°C)	T	− 1	47
		0	51
		1	55

c. Selection of an objective function and a maximum number of experiments, the empirical model is usually governed by Eq. 1.

$$y_{BD} = b + \sum_{i}^{n} a_{ii}x_i + \sum_{i=1}^{n} \sum_{j=2,j=2,j\geq i} a_{ij}x_{ij} \tag{1}$$

Here the independent variables are normalized in the [− 1,1] intervals, according to Eq. 2:

$$x = \frac{z - \frac{(z_{max}+z_{min})}{2}}{\frac{(z_{max}+z_{min})}{2}} \tag{2}$$

In this expressions, z are the current values of the variables, and represent the response variables, n is the number of independent variables, b is the independent term, a_{ii}, and a_{ij} are the regression coefficients related to the main effects, in general the maximum number of experiments should be greater than the number of parameters in the model. Finally, additional experiments should be included at the central point to estimate the experimental error and conduct the selected experiments.

Other additional steps are the Analysis of the experimental data using procedures and regression strategies, the elimination of the variables that are not significant and construction of a mathematical model that links the response and the statistically significant variables and finally the Checking of the effectiveness of the model verifying if the model is capable of representing the response within the experimental error.

Synthesis of potassium methoxide as transesterification reaction

In order to adjust the relationship OH-Oil for the experimental setup, the amount in grams of methanol corresponding to the alcohol-palm oil ratio proposed for each experiment was measured in a 100 ml Erlenmeyer. Afterward, the necessary percentage of potassium hydroxide according to the design of the experiments was carefully added to the container with methanol and stirred until the catalyst was completely dissolved.

Reactor setup and experimental procedure

In a batch reactor with controlled temperature and stirring, the transesterification reaction was carried out. First, 100 g of palm kernel oil were measured in an Erlenmeyer of 200 ml and stirred during 30 min. Subsequently, the intermediate was added to the reactor simultaneously for 5 min to start the reaction, which was prolongated under these conditions, according to experimental for 90 min. Afterward, the sample was cooled down to room temperature and its weight was recorded, then, it was added to a 200 ml decanter. By the effect of gravity, the glycerol falls to the bottom of the container due to its higher density, facilitating

the separation of biodiesel from the products. The glycerol sedimentation time was 12 h for each sample. The raw biodiesel obtained in the separation was washed 4 times with 50 ml of distilled water to remove the excess of methanol, traces of soap and some inorganic contaminants that may be present. The water with the dissolved impurities is separated by density difference through a decanter and the biodiesel obtained is weighed to determine its yield. It is important to carry out this process as soon as possible, since the reaction can continue at a slower rate at room temperature, with the possibility of moving to the left. Additionally, it is recommended to derivatize the sample and proceed to analysis immediately, otherwise store at − 2 °C.

The yield was calculated according to Eq. 3, where m_p is the mass of biodiesel purified and m_s is the mass of oil used for each experiment.

$$y_{BD} = \frac{m_p}{m_s} \tag{3}$$

Products characterization

Several tests were performed in the current study in order to characterize the fuel through the determination of its most relevant physical–chemical properties and composition. The physical–chemical properties evaluated were density (ASTM D7777-13"), flash point and combustion ("ASTM D92-12"), flammability measurements (“ASTM D93-15”), kinematic viscosity (“ASTM D445-15”), ash (“ASTM D482-13”), sulphated ash (“ASTM D482-13”), water and sediments (“ASTM D1796-11e1”), distillation (“ASTM D86-12”), heating value (“ASTM D240-14”), cloud point (“ASTM D-2500”) and refraction index (“ASTM D1218-12”). For an estimation of the composition, an analysis by gas chromatography and by infrared spectroscopy was carried out. First, it was necessary to prepare the samples under the method reported by Narvaez [54], as presented below. The sample was prepared by taking a tube and adding 5 mg of tricaprine (internal standard) and 20 mg of the biodiesel to be analyzed in the vial, then, 2 ml of hexane, 20 µl of BSTFA (derivatizing agent) and finally 2 drops of pyridine (as catalyst). Afterward, the tube rests at least 60 min to complete the test. The sample is manually injected in a 6820 GC chromatograph (Technologies Co. Ltd., Shanghai, China), which has a FID, consisting of a fused silica (0.3 0.53 mm) and a SGE HT-5 fused silica capillary column (12 0.53 mm × 0.15) (SGE International. Ltd., Victoria, Australia). The oven temperature is stabilized at 140 °C for 1 min, then, increases from 140 to 380 °C in 12 min. (ramp of 20 °C/min), and finally remains for 10 min. The injector temperature is 350 °C and the detector temperature is 390 °C. The carrier flow is 6 ml/min N_2, 40 ml/min H_2 and 360 m/min dry air. Data acquisition and processing is performed using software Cerity® (Technologies Co. Ltd., Shanghai, China). The infrared equipment is a BRUKER (UK) that analyzes with Fourier Transform, i.e. FTIR with numbers from 400 to 4000 cm^{-1}.

Results and discussion

Experimental analysis of Biodiesel production from palmist kernel oil

During the experimental runs of catalytic evaluation of palm kernel oil, the thermal conditions of the reactor were stabilized, as well as the preparation of the potassium methoxide intermediate. Some studies show that apparently the formation of micro-emulsions limits the mass transfer during the catalytic reaction [55]. Given this condition, the preparation of potassium methoxide intermediate is a fast strategy that allowed to reduce these diffusional limitations and, simultaneously, the experimental fluctuations of the experimental runs.

The experimental data obtained in the runs were preliminarily adjusted to the following empirical model, according to Eq. 4:

$$y_{BD} = \lambda + \lambda_0 X_{RM} + \lambda_1 X_{KOH} + \lambda_2 T + \lambda_3 X_{RM}^2 + \lambda_4 X_{KOM}^2 + \lambda_5 T^2 + \lambda_6 X_{RM} X_{KOM} + \lambda_7 X_{RM} T + \lambda_8 X_{KOK} T \quad (4)$$

Here $\lambda_i$are the values of the parameters obtained from the empirical model, X_{KOH} is the % of KOH used for the experimental runs, X_{RM} is the molar relationship MeOH/Oil used in the experimental runs and T is the reaction temperature. In this case, the objective function is the biodiesel yield (Y_{BD}). Initially, the first model fit gave the results shown in Table 2.

The model shows that some parameters are not significant, by means of a hierarchical treatment of the data. According to the value of the statistical significance (p), the final model is given by Eq. 5:

$$y_{BD} = \lambda + \lambda_0 X_{RM} + \lambda_2 T + \lambda_3 X_{RM}^2 + \lambda_6 X_{RM} X_{KOM} + \lambda_8 X_{KOK} T \quad (5)$$

The model obtained was achieved by the elimination, according to the value of *p,* and comparing the respective correlation and covariance arrays. The new parameters of the model with the values of significance are given in Table 3.

The final adjustment correlation matrix is shown in Table 4

Table 2 Parameters of the first empirical model of biodiesel yield obtained on STATISTICA V.8

	λ	λ_0	λ_1	λ_2	λ_3
Value	336.4005	16.51819	6.56936	11.0698	1.62252
error	239.6267	15.09435	31.51395	8.7980	0.55707
t	1.4039	1.09433	0.20846	1.2582	2.91259
p	0.2193	0.32370	0.84310	0.2639	0.03330
	λ_4	λ_5	λ_6	λ_7	λ_8
Value	1.90266	0.100000	3.00500	0.092027	0.203598
Error	4.90725	0.082348	1.52200	0.258634	0.570750
t	0.38772	1.214362	1.97438	0.355818	0.356721
p	0.71418	0.278829	0.10533	0.736497	0.735861

Table 3 Adjusted parameters of the empirical model for Biodiesel yield, after the second adjustment

	κ	κ_0	κ_2	κ_3	κ_6	κ_8
Value	62.04206	22.60855	−0.59331	−1.79952	−3.01619	0.258858
error	8.36744	2.83448	0.14604	0.29924	0.84068	0.076093
t	7.41470	7.97626	−4.06277	−6.01356	−3.58779	3.401850
p	0.00004	0.00002	0.00283	0.00020	0.00586	0.007850

Table 4 Correlation matrix of the estimated parameters

	κ	κ_0	κ_2	κ_3	κ_6	κ_8
κ	1.000000					
κ_0	**−0.607061**	1.000000				
κ_2	−0.610295	−0.212743	1.000000			
κ_3	0.609261	**−0.948798**	0.079005	1.000000		
κ_6	−0.000000	−0.296590	0.506382	−0.000000	1.000000	
κ_8	0.000000	0.288237	−0.521057	0.000000	**−0.971835**	1.000000

Bold values represent a stronger correlation between the calculated parameters

In the correlation matrix, the three correlations that most impact the parameters estimated in the model are given by the fit parameter κ_6 and the fit parameter κ_8, whose common variable is the KOH content in the reaction. This correlation can be explained because, as the molar ratio tends to be higher in the reaction, the reaction temperature is lower to keep the value of the yield high. In consequence, it presents a negative value (−0.97). The second correlation occurs between the fit parameter of the model κ_6 and the fit parameter κ_3, which relates the quadratic factor of the molar ratio of the transesterification reaction. This correlation is necessary in the model to keep the performance at a higher value, due to the quadratic nature of the fit and the calculated parameter κ_3, which is negative. Finally, the most important interactions of the correlation matrix show that the molar ratio variable is fundamental to maintain high yield values towards biodiesel production. An additional test of the model performance is the verification that the average residue assumes the value of zero in the experimental region, using the probability distribution t-. student, as shown in Table 5.

With 97.5% confidence, the cumulative probability values are given by Eq. 6:

$$P_{AC} = (t_1, 15) = 0,025\, P_{AC} = (t_1, 15) = 0,975$$
$$-2,131450 \leq \frac{\varepsilon - mean_{\varepsilon}}{\frac{S_N}{\sqrt{N_{\exp}}}} \leq 2,131450 \tag{6}$$

With n = 16, $\dot{\varepsilon}$ = 1,8E–05, S_N = 1,003,the mean interval results in:

$$-0.5346 \leq \frac{mean_{\varepsilon}}{} \leq 0.5347$$

Table 5 Residual values obtained using the function objective $Y_{BD} = \Lambda + \Lambda_0 X_{RM} + \Lambda_2 T + \Lambda_3 X_{RM}^2 + \Lambda_6 X_{RM} X_{KOH} + \Lambda_8 X_{KOH} T$

Run	Fame yield (%)		
	Experimental	Model	Error
1	85.8	85.4897	0.31032
2	98.7	100.204	− 1.50398
3	90.5	89.6429	0.85711
4	94.4	95.3086	− 0.90863
5	87.1	88.9041	− 1.80411
6	99.9	99.0941	0.80588
7	86.9	86.2285	0.67153
8	99.4	97.7563	1.64369
9	99.6	98.7513	0.84871
10	97.5	97.3448	0.15521
11	94.9	950,402	− 0.14022
12	95.1	95.7046	− 0.60458
13	95.2	96.7102	− 1.51022
14	97.2	96.7102	0.48978
15	97.4	96.7102	0.68978

The adjustment is satisfactory, and the average can assume zero values in the residues. Fig. 2 shows the fit of the residuals data, indicating that the model is suitable for the experimental data properly.

Fig. 2 shows that the assumption of the normally distributed error hypothesis is valid, although some residual values of some experiments do not follow the normal distribution. The model adequately satisfies the experimental data with slight correlations of the parameters. The correlation coefficient of the model was $R^2 = 0.985$. Fig. 3 shows the combined effects of variables of experimental planning over the biodiesel yield.

The curves show a point of inflection in the calculated model given by the best condition of biodiesel production. The effects on the temperature do not impact strongly in the yield, if it is considered that the search interval of the reaction temperature was small in comparison with the other variables. Clearly, the % KOH and the relation oil-methanol promote stronger combined effects on the yield. This is associated directly to the reaction rate if we consider that the reaction is dependent on the % of the catalyst and the Biodiesel produced.

The dependence on the reaction temperature affects the reaction kinetics. However, in the production of biodiesel using homogeneous phase catalysts, some authors have shown that other reactor parameters have a more significant impact [56, 57] as shown in Fig. 4.

The two temperature-dependent contour maps show that the condition that maximizes productivity for biodiesel is given by T = 50 °C and 0.8% of KOH as a catalyst in the potassium pentoxide precursor, maintaining high activity up to 54, where the reaction yield declines. The previous contour map depicts the reliability limits, where the values of %KOH and Molar ratio affect the yield. Values

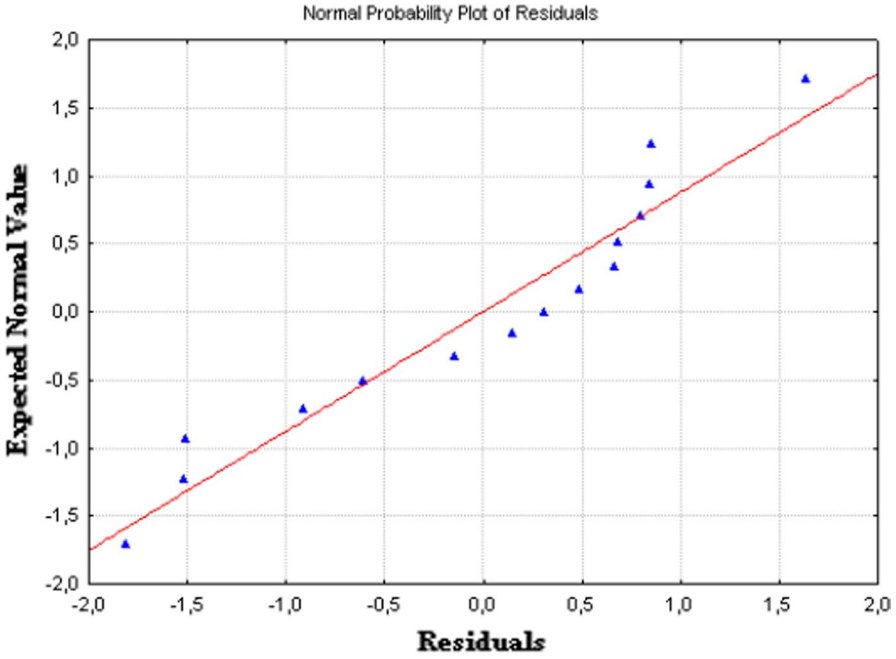


Fig. 2 Adjustment of the empirical model obtained of biodiesel yield $Y_{BD} = \lambda + \lambda_0 X_{RM} + \lambda_2 T + \lambda_3 X_{RM}^2 + \lambda_6 X_{RM} X_{KOH} + \lambda_8 X_{KOH} T$ over the experimental values observed

from 5.5 for the molar ratio and 0.8 for the % KOH increase the yield, while lower values affect the yield and its conversion, as corroborated in other studies [42].

Characterization of biodiesel

The catalytic conversion of biodiesel in homogeneous phase is clearly affected by the adequate selection of the variables. Table 6 shows the physicochemical properties of the biodiesel obtained in the most favorable test conditions.

Spectroscopy analysis and reaction mechanism

A chromatographic and spectroscopic analysis was carried out, which gives the following composition of methyl esters of fatty acids: 2.7% methyl caproate, 42.6% methyl laurate, 15.0% methyl myristate, 9.3% methyl palmitate, 0.3% methyl linoleate, 18.8% methyl oleate, 2.7% methyl stearate and 8.6% others. The above results show convergence with the initial composition of palm kernel oil, which has a similar fatty acid profile: 3.4% capric acid (C_{10}), 47.8% lauric acid (C_{12}), 16.3% myristic acid (C_{14}), 8.5% palmitic acid (C_{16}), 2.0% stearic acid (C_{18}), 15.4% oleic acid (C_{18}) and 2.4% linoleic acid (C_{18}). The spectroscopy results from Fig. 5 allow to establish correlations, considering that C–H, C–O and C–C molecular vibrations

Fig. 3 Response surfaces of the experimental factors of the biodiesel production process, over the reaction yield predicted by the empirical model: **a** Effect of reaction temperature and MeOH/Oil ratio (X_{RM}), **b** Effect of reaction temperature and % KOH

are a consequence of olefin fractions, oxygenated compounds and fatty acids for palm kernel oil, FAME and potassium methoxide.

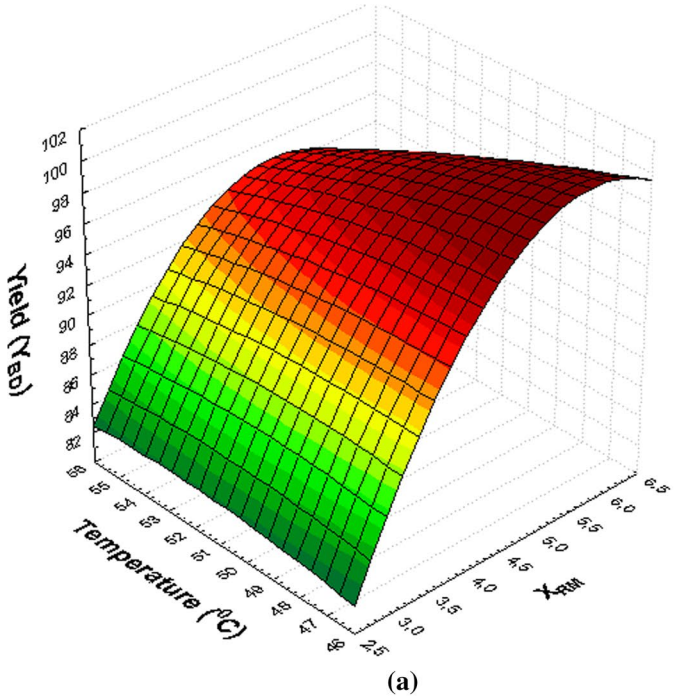
The IR spectra showed C–O stretch at 720 cm^{-1}, related with C–O binding from methyl esters [45]. The C=C stretch at 890 cm^{-1} is attributed to double bonds on hydrocarbons. C–O, C–C and C–H vibration groups are attributed to carbonyl components from hydrocarbon-free methyl esters [46]. Other vibrations in 1200, 1400, 1480, 1740, 2830 and 2925 are finally attributed to the presence of molecular stretching in C–H for hydrocarbons and ethers [47], single bonds in esters designated by C–O vibrations, together with C–H and O–H bending and C=O in C–H esters [48, 49]. Table 7 shows the IR assignment for palm kernel oil, FAME obtained and potassium methoxide

Weak peaks were found at 721 and 878 cm^{-1} related to the presence of aromatic alkyl and benzene derivatives. The presence of esters was observed in the region between 1016 and 1361 cm^{-1} and at 1741 cm^{-1}. Additionally, carboxylic compounds were identified between 2858 and 2922 cm^{-1} due to Alkyl C=H stretching. Finally, the peaks in 1361 and 1461 cm^{-1} showed the presence of phenols, aldehydes and alkene groups with a lower concentration.

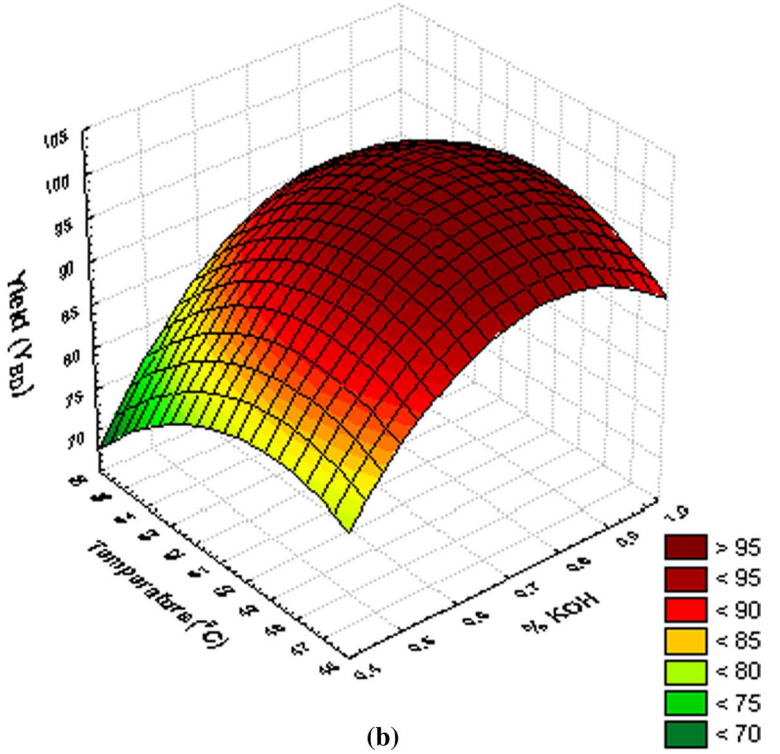
The FTIR for palmist kernel oil showed the presence of volatile compounds in various vibrations, such as blends of alkanes, aldehydes, alcohols, esters and carboxylic acid derivatives. The change of vibrations explains the reaction path. For instance, when the C–H stretching intensity changed in the same region of FTIR spectrum, the vibrations for C=O stretching and C–C stretching changed at the same time. The role of potassium methoxide as intermediate is able to change the electronic configuration of triglycerides of palm kernel oil to compounds of FAME

The GC results exhibits the following fatty acid methyl ester composition: 2.7% methyl caproate, 42.6% methyl laurate, 15.0% methyl myristate, 9.3% methyl palmitate, 0.3% methyl linoleate, 18.8% methyl oleate, 2.7% methyl stearate and 8.6% others [50]. Additionally, and according to the FTIR spectrum, possible components are present in the sample of biodiesel obtained, such as methyl octadecanoate ($C_{19}H_{38}O_2$), methyl 3-nonenoate ($C_{10}H_{18}O_2$), methyl nonanoate ($C_{10}H_{20}O_2$) and hexanoate ($C_7H_{14}O_2$), as well as carboxylic acids (butanoic, hexanoic, decanoic and undecanoic) and other saturated and unsaturated esters. This characterization is consistent with that of other research carried out with palm kernel oil [58, 59]. Other studies have reported different reactivity, thus changing the nature of palm kernel oil, the catalysts and the main conditions of reactions, as shown on Table 8.

Ogogo et al. (2018) [60] showed a similar kinetic approach by surface response models with a higher KOH concentration and temperature reaction than those used in this work. Alamu et al. (2007) [41] showed that FAME yield depends on KOH%, without considering the effect of reaction temperature and methanol/oil molar ratio on the reaction kinetic. The next year, Alamu (2008) [42] reported the effect of alcohol/oil molar ratio on FAME yield under typical conditions of transesterification.


Yield (YBD)
102
100
98
96
94
92
90
88
86
84
82
Temperature (°C)
55
54
53
52
51
50
49
48
47
46
XMM
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5

(a)


Yield (YBD)
105
100
95
90
85
80
75
70
Temperature (°C)
% KOH
0.5
0.6
0.7
0.8
0.9
1.0
> 95
< 95
< 90
< 85
< 80
< 75
< 70

(b)

Fig. 4 Contour maps of reaction yield as a function of experimental variables **a** combined effect of MeOH/Oil ratio (X_{RM}) with reaction temperature **b** combined effect of %KOH with reaction temperature

These works keep some conditions fixed, therefore, it is not possible to analyze the combined effects on reaction kinetics, since experimental fluctuations represent fluctuating directions associated with the reaction mechanism [61].

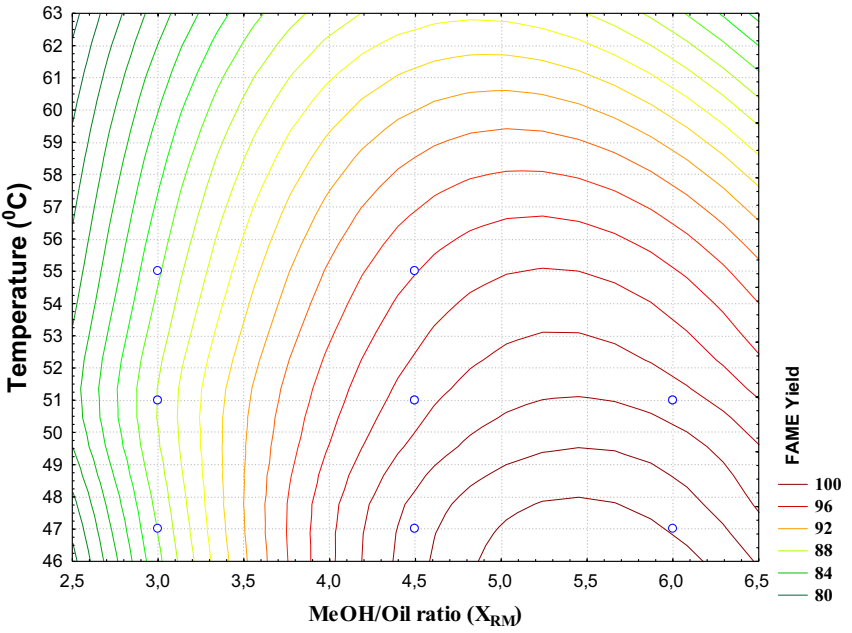
A reaction path [62] can be described according to Fig. 6.

The first step is the attack of a methoxide ion on the carbonyl group of the triglyceride. Second, the intermediate compound reacts with a methanol to produce a methoxide ion. Finally, in the third step, the intermediate compound is rearranged to form an ester and a diglyceride. These three steps are reproduced in three reversible stages [51]. The first stage corresponds to the previous one from triglycerides to diglycerides. In the second stage, the generated diglycerides are also attacked by the methoxide ion to generate another intermediate that, when reacting with another methanol molecule, generates a methoxide. Finally, when reorganized, an ester and a monoglyceride are produced. It is expected that these three steps happen in the third stage, where the monoglycerides that appear will end up giving another ester and glycerin.

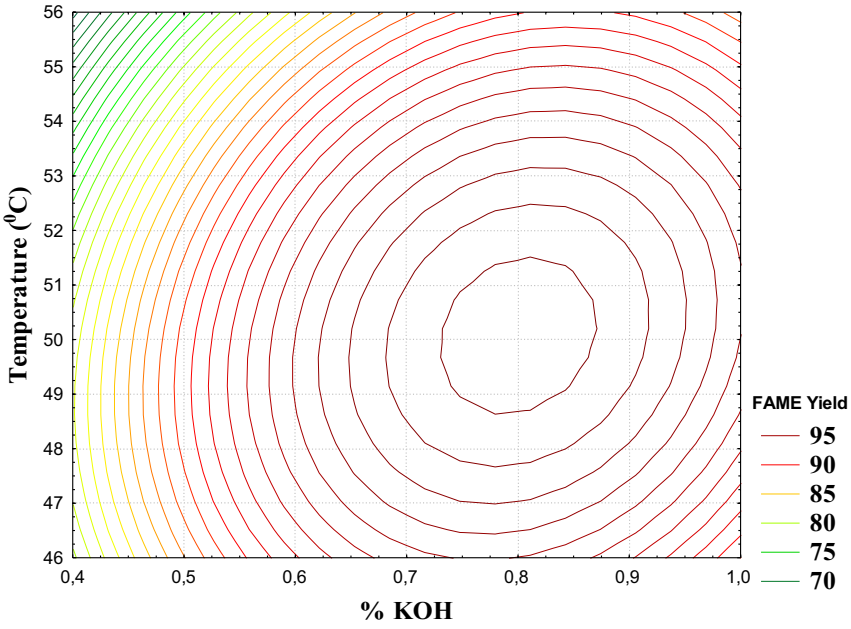
In general terms, for each triglyceride molecule, 3 methanol molecules are consumed, 3 methyl esters are produced and 1 glycerol molecule, while the hydroxide participates as a catalyst. The step that controls the process was found to be the conversion of triglycerides to diglycerides [63]. The catalyst concentration mainly affects the third stage of conversion of monoglycerides to ester and glycerin [54].

Conclusions

According to the experimental model and the statistical study of the experimental data obtained in the batch reactor employing the palm kernel oil transesterification process, it can be concluded that the increase in the quantity of methanol favors the yield to a critical value of the quantity of catalyst. According to the design of experiments, it was found that for the transesterification of palm kernel oil at 500 rpm during 1.5, the highest yield of 99.9% was obtained at 50 °C, 6 mol methanol/mol oil and 0.8% of potassium hydroxide concentration for oil, since the highest variability occurs as a function of this variable. The effect of temperature was not decisive in the process, due to the low range of exploration of this variable. However, the adverse effects of higher temperatures could affect the selectivity and, therefore, a greater range is not contemplated. According to the analysis tests made in the laboratory, the biodiesel met the characteristics and standards required according to the different ASTM standards mentioned in the current document. The cold properties were not evaluated since this parameter must be measured for biofuel mixtures allowed by the current aviation legislation. The spectroscopic and chromatography results allowed to determine that the approximate fatty acid methyl ester composition of the palm kernel oil biodiesel


Temperature (0C)
MeOH/Oil ratio (XRM)
FAME Yield
100
96
92
88
84
80

(a)


Temperature (0C)
% KOH
FAME Yield
95
90
85
80
75
70

(b)

Table 6 Characterization of biodiesel produced from palm kernel oil

Property	Method	Units	Measured value	Limits (ASTM Reported values)
Density at 15 °C	ASTM D7777-13	g/cm^3	0.89	0.860–0.900
Kinematic viscosity at 40 °C	ASTM D445	cSt	4.22	1.9–6.0
Flashpoint. Open Cup	ASTM D92-12	°C	119.00	Minimum 93
Flashpoint. Closed Cup	ASTM D93-15	°C	107.00	Minimum 100
Water and sediments	ASTM D1796-11E1	V%	0.00	Maximum 0.05
Total Ash	ASTM D482-13	wt%	0.07	Maximum 0.18
Sulphated ash	D874-13A	wt%	0.01	Maximum 0.02
Average boiling temperature	D86-12	°C	140.00	Maximum 360
Cloud point	ASTM D-2500	°C	7.00	Report
Refractive index	ASTMD1218-12	–	1.43	N/A
Heat value	ASTM D240-14	MJ/kg	37.40	N/A
Specific gravity	N/A	–	0.887	N/A

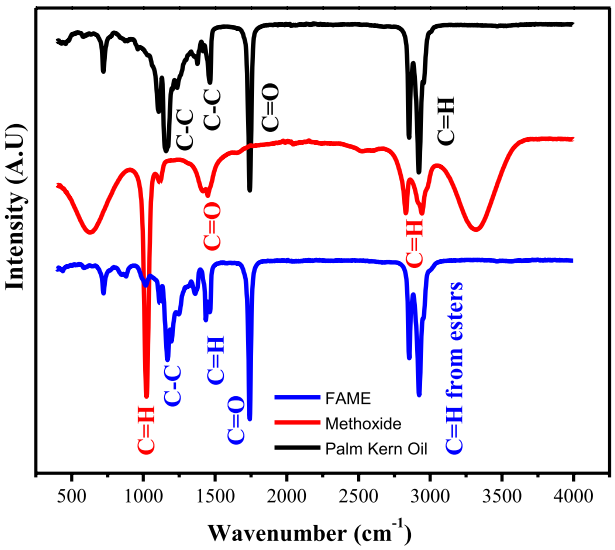


Fig. 5 FTIR spectra of biodiesel obtained from palm kernel oil at T = 50 °C, P = 1 atm, $X_{RM} = 6$, and 0.8% KOH (blue line); palm kernel oil at ambient temperature (black line), and potassium methoxide intermediate (red line). (Color figure online)

generated has mainly 43% methyl laurate (C12:0), 15% methyl myristate (C14:0), 9% methyl palmitate (C16:0) and 19% methyl oleate (C18:1), following the fatty acid profile of the palm kernel oil. Other vibrations in 1200, 1400, 1480, 1740,

Table 7 Spectroscopic characterization and quantification of palm kernel oil and Biodiesel FAME

Palm Kernel oil		FAME	
Assignment	Wavenumber (cm^{-1})	Assignment	Wavenumber (cm^{-1})
Cis –CH=CH-	721	Aromatic C–H Bending	721
C–C stretching	1158	Benz.1, 2, 4-trisub	878
C–C stretching	1464	Alcohol/Phenol O–H stretching	1016
C=O stretching	1743	C-O stretching	1113
C–H stretching	2852	Esters C=O stretching	1167
C–H stretching	2920	Esters C–O stretching	1196
		CH_3–O–C(=O)R stretching	1361
		CH_3–O–C(=O)R(CH_3) stretching	1436
		CH_3–O–R–(CH_3) stretching	1461
		Esters C = O & C–O Stretching	1741
		Alkyl C–H Stretching	2858
		Alkyl C–H Stretching	2922
Average composition			
IUPAC NAME	%mass	% Free mass	% Molar
Methyl decanoate	2.7	2.95	3.78
Methyl dodecanoate	42.6	45.61	51.90
Methyl Tetradecanoate	15.0	16.41	16.16
Methyl Hexadecanoate	9.3	10.18	8.98
Methyl 9-octadecenoate	2.7	2.95	2.36
Methyl octadecenoate	18.8	20.57	16.56
9,12 methyl octadecadienoate	0.3	0,33	0.27
Others	8.6	–	–
Average reaction rate	1.18×10^{-3} mol/s		

Table 8 Comparison of biodiesel yield reported using palmist kernel oil

T	MeOH/Oil	KOH	FAME Yield	Cloud point of biodiesel obtained (°C)	References
52.5	6	5	94.07	8	[60]
60	5	0.75	95.8	6	[41]
60	0.22	1	96	6	[42]
50	5.5	0.8	99.4	7	This work

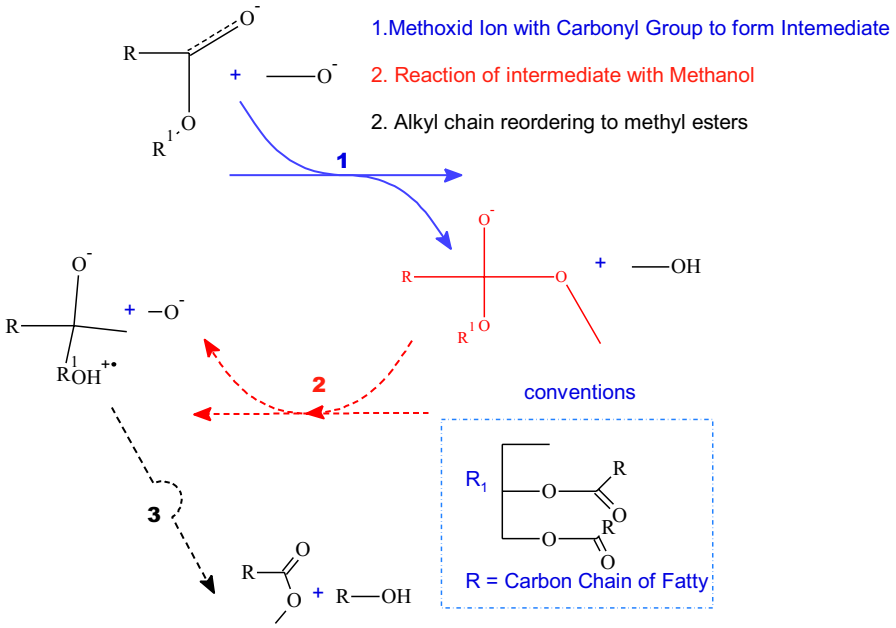


Fig. 6 Proposal of reaction path for biodiesel synthesis from palm kernel oil with MeOH/KOH

2830 and 2925 are finally attributed to the presence of molecular stretching in C–H for hydrocarbons and ethers, single bonds in esters designated by C–O vibrations, along with C–H and O–H bending, C=O in C–H esters, And a small percentage of C–H stretching and bending in alkanes.

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Compliance with ethical satndrads

Conflict of interest The authors declare that they have no conflict of interest.

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