Performance of biodiesel production by means of Ultrasonic Transesterification

Desempeño de producción de biodiesel por medio de Transesterificación Ultrasónica

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Abstract

Introduction— In recent years, the use of renewable energies and eco-friendly fuels has increased, among which one of the best performance is biodiesel; the paper shows an upgrade in the efficiency and effectiveness laboratory level's biodiesel obtaining.

Objective— Evaluating the production of biodiesel employing ultrasound is presented, leading to improve the response time and efficiency of the reaction, concerning the conventional method using only temperature.

Methodology— In the transesterification process, castor oil, methanol, and potassium hydroxide are used, obtaining biodiesel and glycerin. A factorial design with two levels for transit time, mixing temperature, and ultrasound intensity were applied in an instrumented scale reactor to control these variables.

Results— In the tests, values close to the reference stoichiometric value of the reaction were obtained. The statistic indicates a normal behavior of data, and identifies it as a factor of incidence in the efficiency of the reaction to the intensity of the ultrasound, concerning the response time of the reaction, the mixing temperature and the intensity of ultrasound.

Conclusions— The efficiency of the reaction concerning the studied factors, it only depends on the ultrasound obtaining up to 95.7% of the stoichiometric value; and the response time of the reaction depends on the temperature and ultrasound, obtaining times of formation of product four times faster.

Keywords— Biodiesel; Efficiency; Incidence factor; Mixing temperature; Ultrasound

Resumen

Introducción— En los últimos años, el uso de energías renovables y combustibles ecológicos ha aumentado, entre los cuales uno de los mejores resultados es el biodiesel; el artículo presenta una mejora en la eficiencia y la eficacia en la obtención de biodiesel a nivel de laboratorio.

Objetivo— Evaluar la producción de biodiesel por medio de ultrasonido, lo que lleva a mejorar el tiempo de respuesta y la eficiencia de la reacción, con respecto al método convencional que usa solo temperatura.

Metodología— En el proceso de transesterificación, se utilizan aceite de ricino, metanol e hidróxido de potasio; obteniendo biodiesel y glicerina. Se aplicó un diseño factorial con dos niveles de tiempo de tránsito, temperatura de mezcla e intensidad de ultrasonido en un reactor a escala instrumentado para controlar dichas variables.

Resultados— En las pruebas, se obtuvieron valores cercanos al valor estequiométrico de referencia de la reacción. La estadística indica un comportamiento normal de los datos y lo identifica como un factor de incidencia en la eficiencia de la reacción a la intensidad del ultrasonido; con respecto al tiempo de respuesta de la reacción, la temperatura de mezcla y la intensidad del ultrasonido.

Conclusiones— La eficiencia de la reacción con respecto a los factores estudiados, solo depende de que el ultrasonido obteniendo hasta el 95.7% del valor estequiométrico; y el tiempo de respuesta de la reacción depende de la temperatura y el ultrasonido, obteniendo tiempos de formación del producto cuatro veces más rápidos.

Palabras clave— Biodiesel; Eficiencia; Factor de incidencia; Temperatura de mezcla; Ultrasonido

I. Introduction

The search to reduce dependence on fossil fuels has led to the implementation of clean energy and fuels with a lower carbon footprint. Such as natural gas, gasohol, and biodiesel; the latter has gradually increased its production due to its low production costs and minimal contamination [1], which implies searching for new alternatives or improvements to obtain them. Biodiesel, when used in its synthesis, an oil of natural origin [2], a catalyst and short-chain alcohol, makes it necessary for its precursors to having a particular set of characteristics [3]; this is how conventional techniques for oil conditioning employ acid catalysts or homogeneous bases [4]. The use of these catalysts has technical and environmental disadvantages, by the processes of neutralization and filtration to remove salts that are formed [5], generating additional costs in the separation and purification of the final products. Obtaining biodiesel is a transesterification process in which a triester (oil) is transformed into a methyl ester.

The variables of highest consideration in the transesterification reaction are the alcohol/oil molar ratio [6], percentage of catalyst and temperature [7], its effect being evaluated by kinetic studies [8], with little use of statistical designs. For the production of biodiesel, the alcohol/oil molar ratio varies from 0.1:1 - 24:1; the concentration of the catalyst is between 0.25% -6% by weight concerning the oil; the temperature is commonly set at 333.15 K, but intervals between 310.15 K - 348.15 K are found [9]. The production processes of biodiesel are divided into batches or continuous [10], [11]. The continuous processes use reactors of stirred tank CSTR (Continuous Stirred Tank Reactor) [12] and tubular PFR (Plug Flow Reactor) [13]. The studies focus on controlled laboratory reactions, with batch reactors with small reaction volumes (less than 3 L), to characterize the different factors that influence the reaction [14].

To reduce processing times and improve efficiency in obtaining biodiesel, we have identified two variables to be optimized independently of the concentration, according to the Arrhenius equation [15], related to the rate of product formation: reaction temperature and mixing intensity. The reaction temperature in alcoholysis depends on oil and alcohol employed [16]; increase temperature improves performance and reaction time, but the boiling point of alcohol should not be exceeded due to its evaporation and formation of bubbles. That limit reaction at the alcohol/oil/biodiesel interphases; the ultrasound-assisted transesterification is performed at a temperature among 313.15 K - 333.15 K [17]. The intensity of microwave mixing accelerates endothermic reactions [18], through efficient heating, low levels of environmental contamination, and reproducible procedures, but there is a risk of explosions due to overheating [19]. On the other hand, ultrasound increases the transfer of mass and the rate of chemical reactions [20], reducing risks. Several studies compare reactions of ultrasound-assisted transesterification with other techniques for the production of biodiesel, such as mechanical agitation and microwave irradiation [21], thermal temperature increase [22], and hydrodynamic cavitation [23]. With ultrasound performance for making biodiesel the highest, obtaining short-term processes [24], regardless of the catalyst and reagents [25], [26].

The research's objective is to measure the incidence of ultrasound and temperature on the efficiency and rate of response in the biodiesel production, with preset values of reagents; whereby the experimental work will be carried out in an ultrasound reactor, which allows controlling the sources of variation of each stage of the reaction inside the transesterification tank. The reaction that takes place inside the tank is the most significant stage in the process, in which the increase in reaction response time is expected compared with conventional techniques. In this way, the experiments to be performed will be subjected to ambient temperature (295.15 K) and 315.15 K, combined with ultrasonic agitation and transit times of 4 and 6 minutes. All this to determine the influence of each factor on efficiency, and reaction response time, using the design of experiments methodology, and statistical analysis.

II. Materials and Methods

A. *Alcoholysis reaction*

Of the three classes of transesterification, this work focuses on alcoholysis (Fig. 1). A reaction by which a molecule of triglyceride, a significant component of vegetable oil, reacts with light alcohol, under the action of a catalyst to produce a mixture of esters of fatty acids and glycerin [27].

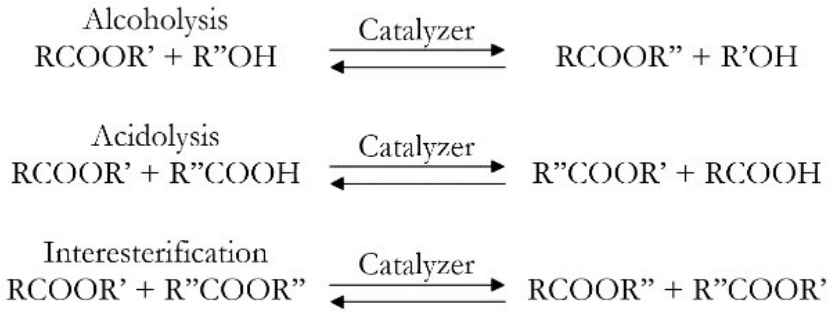

Fig. 1. General Transesterification reactions.
Source: Adapted from [27].

The transesterification reaction of vegetable oil with methanol, to produce methyl esters of fatty acids and glycerin, depending on the stoichiometry of the reaction. For each mole of transesterified triglyceride, three moles of methanol are needed, obtaining three moles of methyl esters and one mole of glycerin. The reaction consists of three consecutive reversible stages: initially, triglyceride is converted into diglyceride, for later conversion to monoglyceride, and glycerin is obtained as a final product (Fig. 2).

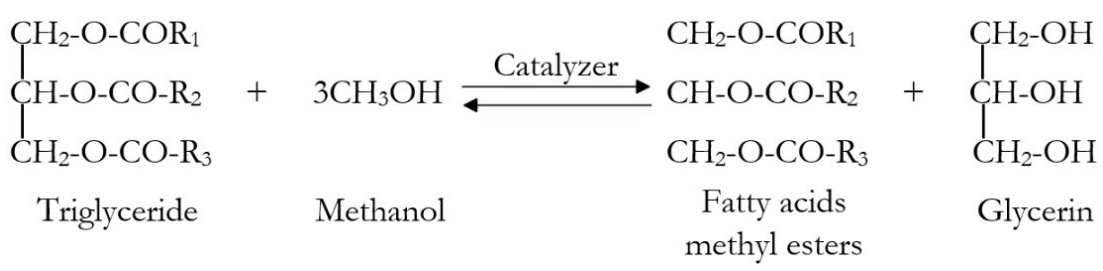

Fig. 2. Schematic reaction of transesterification of vegetable oils.
Source: Adapted from [27].

The applications of glycerin are an additive or raw material in food products, tobacco, and pharmaceutical drugs, for the synthesis of trinitroglycerin, alkyd resins, and polyurethanes [28]. The methyl esters are transformed into a large number of raw materials for their subsequent synthesis and different chemical products (Fig. 3).

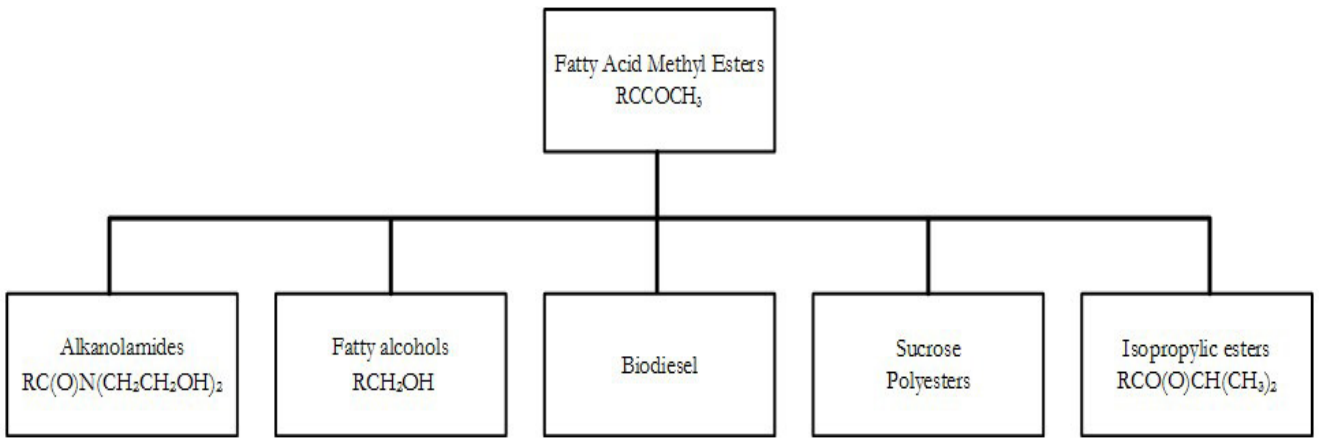

Fig. 3. Products obtained in transesterification.
Source: Adapted from [27].

One of the products obtained from methyl esters (Fig. 3) is biodiesel [29]; its production is subject to different variables that alter the quality and performance of the final product.

The raw material to be used in this research is 130 mL of castor oil, 31 mL of methanol, and 3 grams of potassium hydroxide, obtaining biodiesel and glycerin. According to the stoichiometry of the reaction, the amount of reference glycerin is 9.5 mL [30]. When mixing the substances, a glass beaker and a manual stirring with a glass rod are applied for one minute before subjecting it to the different experimental conditions.

B. *Acoustic cavitation*

The time in obtaining biodiesel and the quality of the final product depends on how the substances present in the reaction are mixed. The mixing rate is essential throughout the reaction process since, in the transesterification tank, a system of two immiscible liquid phases is formed between the oil and the alcohol/catalyst solution [31]. The agitation types also affect the performance of the reaction [32]. Another form of mechanical agitation consists of the irradiation of the mixture with ultrasound; this mechanism is known as acoustic cavitation. The mixing intensity of the substances in this work is carried out with an ultrasonic actuator to increase the efficiency of the transesterification reaction (Fig. 4) [33].

Fig. 4. Process's experimental setup.
Source: [33].

When the liquids are subjected to ultrasound, the sound waves propagated in the medium generate fluctuating cycles of high pressure (compression) and low (rarefaction). During the low-pressure cycle, high-intensity ultrasonic waves create small vacuum bubbles, which by cavitation grow to their resonance size generating a violent collapse during a high-pressure cycle. Reaching locally high temperatures (approx., 5000 K) and pressures (approx., 2000 atm) together with the propulsion of liquid streams up to 280 m/s [34]. The temperature effects in solids and liquids improve the transference of components among reagents. The pressure's rapid change generates bubbles in liquids to ensure greater contact area. High propulsion streams guarantee a flow constant of reagents avoiding localized saturation of components.

C. *Experimental design*

The purpose of the proposed factorial design is to determine the influence of transit time, temperature, and ultrasound, on the efficiency and response time of the reaction. Two levels are established for each factor: {a1, a2} for the transit time factor (A), {b1, b2} for the temperature (B) and {c1, c2} for the ultrasound (C), (Table 1), the number of possible combinations is 8 [35].

Table 1. Factorial design 23 of the experiments.

Factor	Levels
A: Transit time	a1: 4 minutes
	a2: 6 minutes
B: Temperature	b1: 295.15 K
	b2:315.15 K
C: Ultrasound	c1: 0 kHz
	c2: 20 kHz
# Experiment	Combination
1	a1-b1-c1
2	a1-b2-c1
3	a1-b1-c2
4	a1-b2-c2
5	a2-b1-c1
6	a2-b2-c1
7	a2-b1-c2
8	a2-b2-c2

Source: Authors.

Three tests are performed on each of the possible combinations, and the volume of glycerin formation is measured every minute; the results of the standard experiments (1 and 5) are omitted (Table 1), leaving the tests distributed for both transit times:

- *Test* 1-3: Three tests are carried out at a temperature of 315.15 K, to determine the influence of temperature on the reaction.
- *Test* 4-6: Three tests are carried out at room temperature (295.15 K) with ultrasound (20 kHz), to determine the influence of ultrasonic radiation on the reaction.
- *Test* 7-9: Three tests are carried out at a temperature of 315.15 K and the presence of ultrasonic radiation to determine the joint influence.

III. Results and Discussion

In the experimentation process, the glycerin formation data are recorded until reaching a stable state in each test (times and efficiency are compared in each experiment). Initially, a descriptive analysis of the data is performed, then a formal analysis is performed with MINITAB [36], to determine the most relevant factor in each experiment. The data are presented by transit time, and in each treatment, the final volume of glycerin and efficiency of the developed test are related. The efficiency of the reaction is obtained by comparing the final value of glycerin with the reference value (9.5 mL).

A. *Transit times*

In the experiments, the factors: temperature and ultrasound alternate in the developed tests, the stabilization time of the reaction is 90 minutes the final volume of glycerin is related to the efficiency of the reaction (Table 2).

Table 2. Results of transit time tests.

Test	Glycerin Volume [mL]		Reaction Efficiency [%]	
	a1	a2	a1	a2
1	7.1	6.3	74.7	66.3
2	6.9	6.6	72.6	69.5
3	7	6.3	73.6	66.3
4	8.3	8.7	87.3	91.5
5	8.7	9.1	91.5	95.7
6	8.1	8.9	85.2	93.6
7	8.5	9	89.4	94.7
8	8.5	8.8	89.4	92.6
9	8.4	8.7	88.4	91.5

Source: Authors.

For the transit time of 4 minutes: in tests 1-3, of similar glycerin comprised between 6.9 mL - 7.1 mL, the maximum efficiency is 74.7%; in tests 4-6, final glycerin volumes between 8.1 mL - 8.7 mL are obtained, the maximum efficiency is 91.5%; finally, in tests 7-9, final glycerin volumes between 8.4 mL - 8.5 mL are obtained, the maximum efficiency is 89.4%. The highest volume of glycerin formation is presented in test 5 (a1-b1-c2) with a value of 8.7 mL (Fig. 5a).

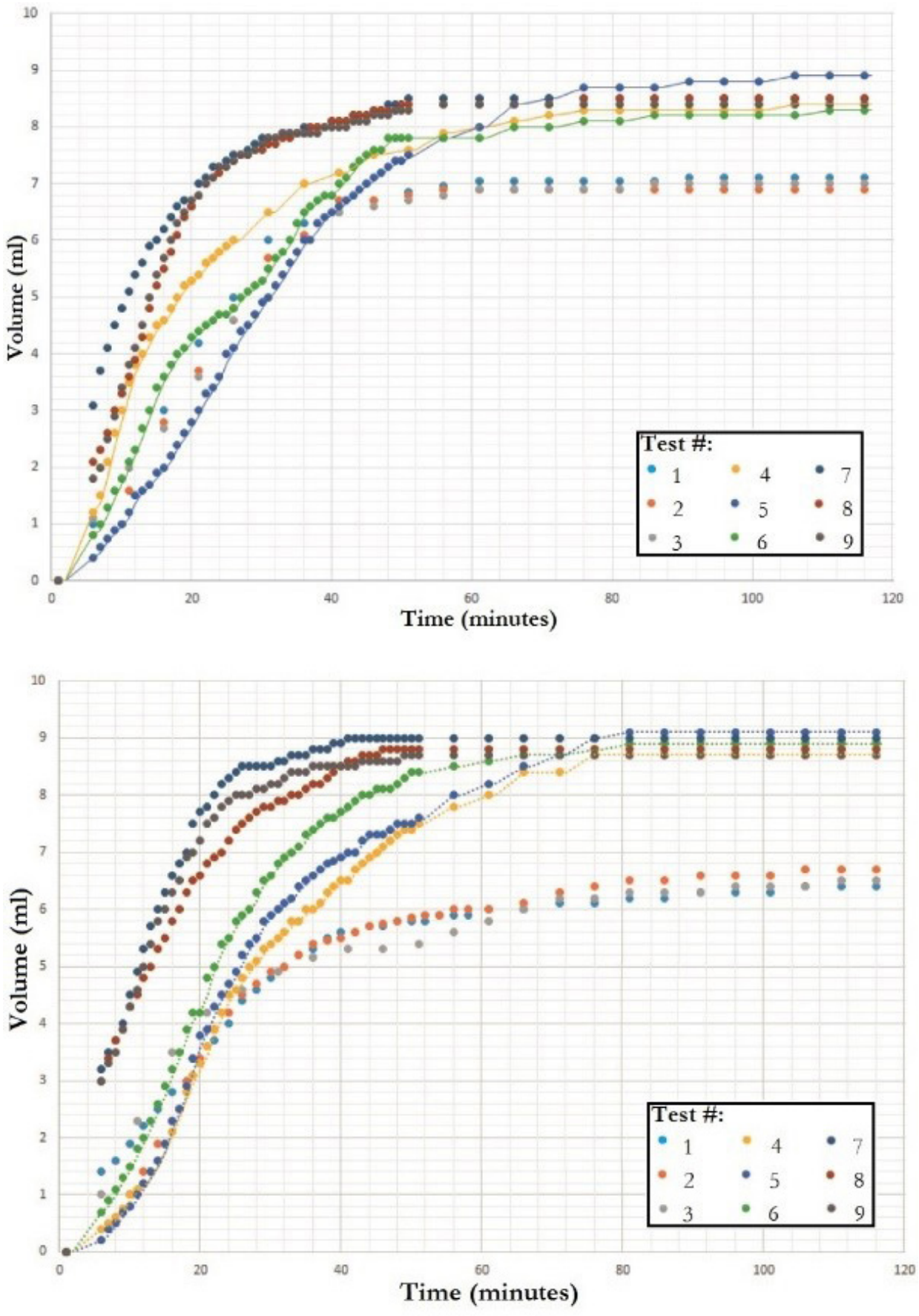

Fig. 5. Kinetics of the alcoholysis reaction. a) Transit time of 4 minutes. b) 6-minute transit time.
Source: Authors.

For the transit time of 6 minutes: in tests 1-3, final glycerin volumes are obtained between 6.3 mL - 6.6 mL, the maximum efficiency is 69.5%; in tests 4-6, final glycerin volumes are obtained between 8.7 mL - 9.1 mL, the maximum efficiency is 95.7%; in tests 7-9, final glycerin volumes are obtained between 8.7 mL - 9 mL, the maximum efficiency is 94.7%. The highest volume of glycerin formation is presented in test 5 (a2-b1-c2) with a value of 9.1mL (Fig. 5b).

When analyzing the percentage change in efficiency over time, the influence of the factors studied in the tests can be seen; of each group of tests, one is randomly chosen (Table 3).

Table 3. Efficiency vs. Time Results.

Transit time	Test	Pending [%Effi/mins]	Time [mins]	Variable of interest
t1	1	2.12	33	Temperature
	6	2.07	30	Ultrasound
	9	3.95	16	Ultrasound & Temperature
t2	1	1.35	60	Temperature
	5	1.99	30	Ultrasound
	9	4.85	14	Ultrasound & Temperature

Source: Authors.

B. *Influence of ultrasound and temperature on the reaction*

To determine the statistical influence of each experimental factor in the tests that were conducted [36], each one is related to the responses under evaluation: Efficiency (value recorded in 90 minutes) and Time (measured until glycerin is 90%) of the final theoretical volume) (Table 4) (Fig. 6).

Table 4. Worksheet of factorial experiments.

Temperature [K]		Ultrasound [kHz]		Efficiency [%]		Response Time[mins]	
a1	a2	a1	a2	a1	a2	a1	a2
295.15	295.15	0	0	72.5	72.5	68	68
315.15	315.15	0	0	73.6	67.4	43	46
295.15	295.15	20	20	88	93.6	42	61
315.15	315.15	20	20	89	92.9	28	22

Source: Authors.

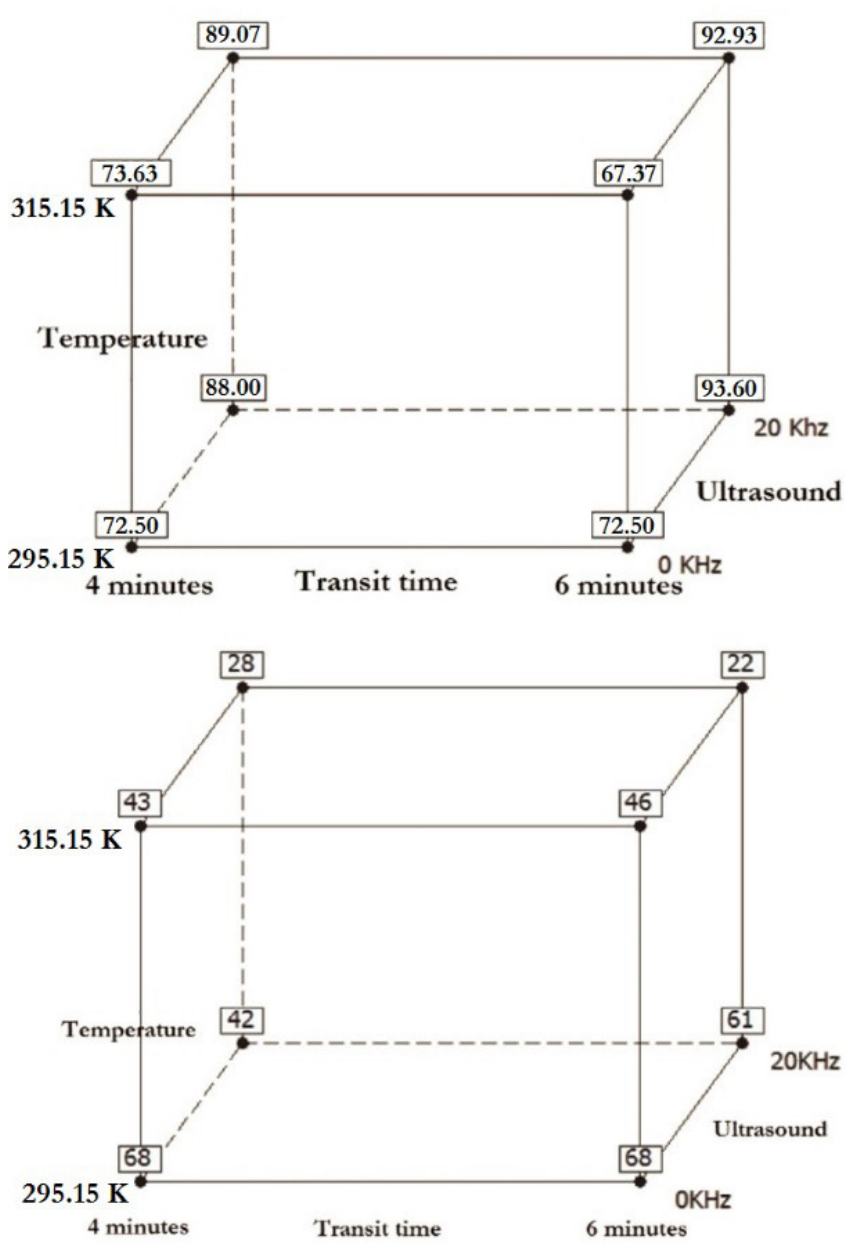

Fig. 6. Graphical representation of the factorial experiment. a) Efficiency. b) Response Time.
Source: Authors.

Among the three factors studied related to efficiency, ultrasound is the one with the highest incidence, temperature factors, and transit time does not affect (Fig. 7a). Temperature and ultrasound have a higher impact on the response time of the reaction. The transit time factor does not show an appreciable effect (Fig. 7b).

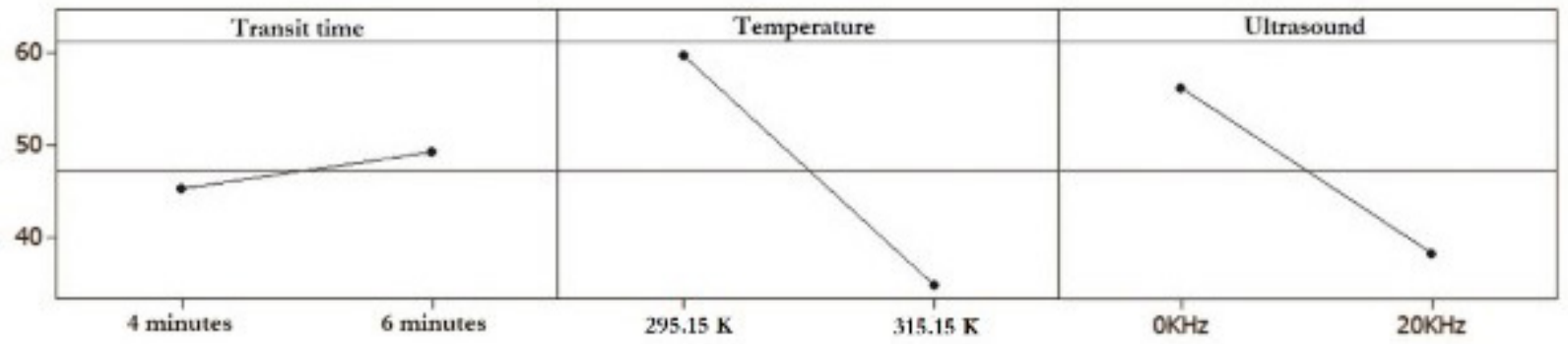

Fig. 7. Main effects of the factors in the reaction. a) Efficiency. b) Response time.
Source: Authors.

Table 5. ANOVA results.

Reaction variable	Factor	Value-P	Value-F
Efficiency	Transit time	0.743	0.01
	Temperature	0.962	0.01
	Ultrasound	0.001	96.82
Response time	Transit time	0.441	0.09
	Temperature	0.006	8.77
	Ultrasound	0.018	2.67

Source: Authors.

Variance analysis is used to know which variables are experimentally significant (Table 5). Regarding the P-value about the efficiency: the transit time and temperature are not significant factors; only ultrasound is a significant factor. Concerning response time, transit time is not a significant factor, Temperature and ultrasound are significant factors. This information is corroborated with the regression equations (1)-(2), for both efficiency and response time, have an R-square of 100%, and the normal probability of the factors diagram (Fig. 8).

$$Efficiency = 57.65 + 3.41A + 0.68B + 0.72C - 0.16AB + 0.016AC - 0.023BC + 0.0056\,ABC \quad (1)$$

$$Response\ time = 102.1 - 1.65A - 1.55B - 6.89C + 0.075AB + 1.24AC + 0.17BC - 0.035ABC \quad (2)$$

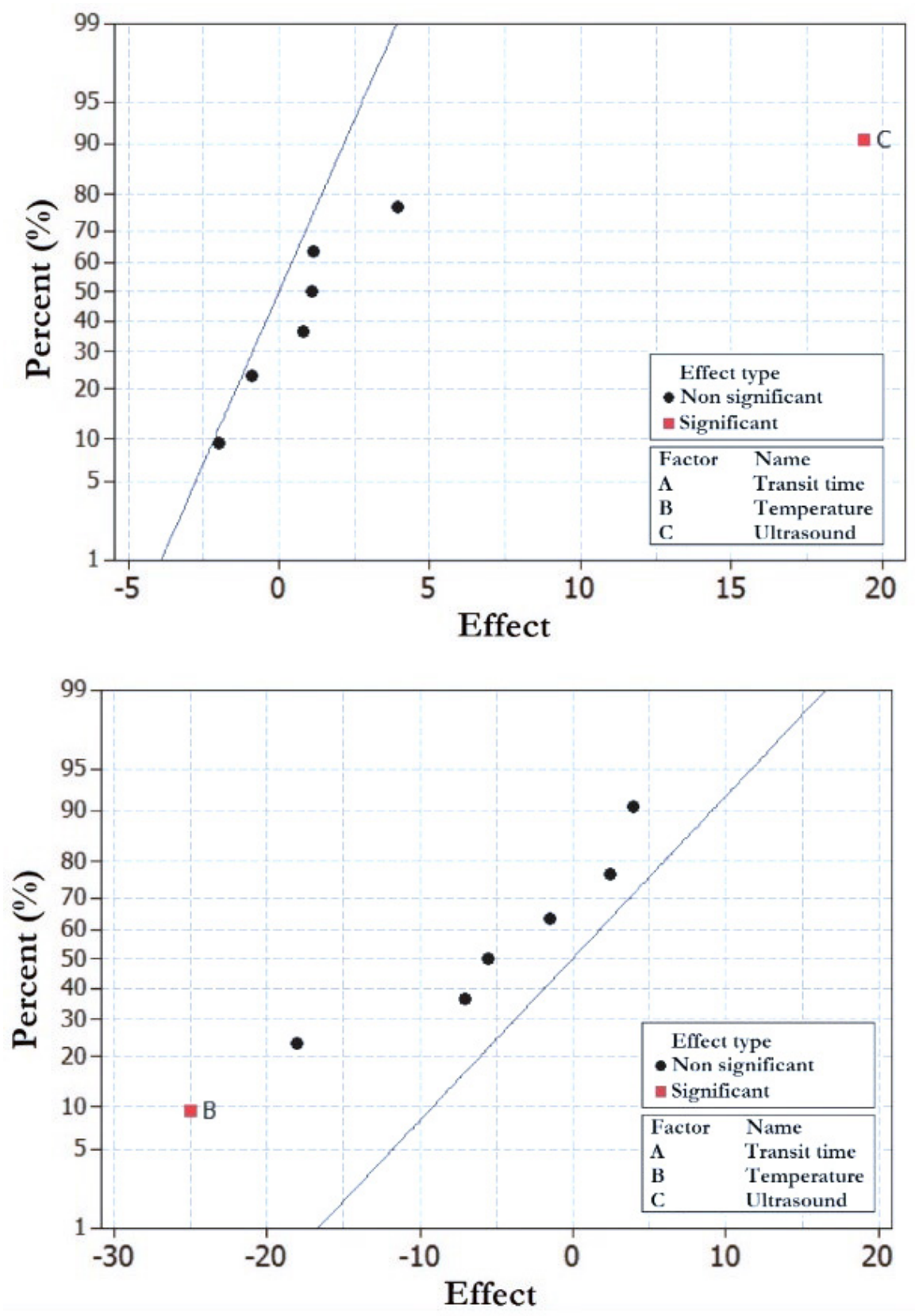

Fig. 8. Normal probability of effects on the reaction. a) Efficiency. b) Response time.
Source: Authors.

With the Pareto diagram, the magnitude and importance of the factors involved in the reaction and their possible combinations are determined (Fig. 9). The diagram shows the absolute value of the effects and draws a reference line in the graph. Any effect that extends beyond this baseline is potentially significant.

According to the above, the efficiency is influenced by ultrasound; this happens because the liquid's cavitation guarantees an effective contact area with a constant conversion among reagents. The response time is influenced by ultrasound and temperature; the temperature's effect is the process acceleration by the increment of the internal moving of reagents by the heat given.

The most relevant effect in the study of efficiency is ultrasound, followed to a lesser degree by the combination: transit time and ultrasound. In the study of the response time of the reaction, the most relevant effect is the temperature followed by the ultrasound, and to a lesser degree, the combination: transit time, temperature, and ultrasound (Fig. 10).

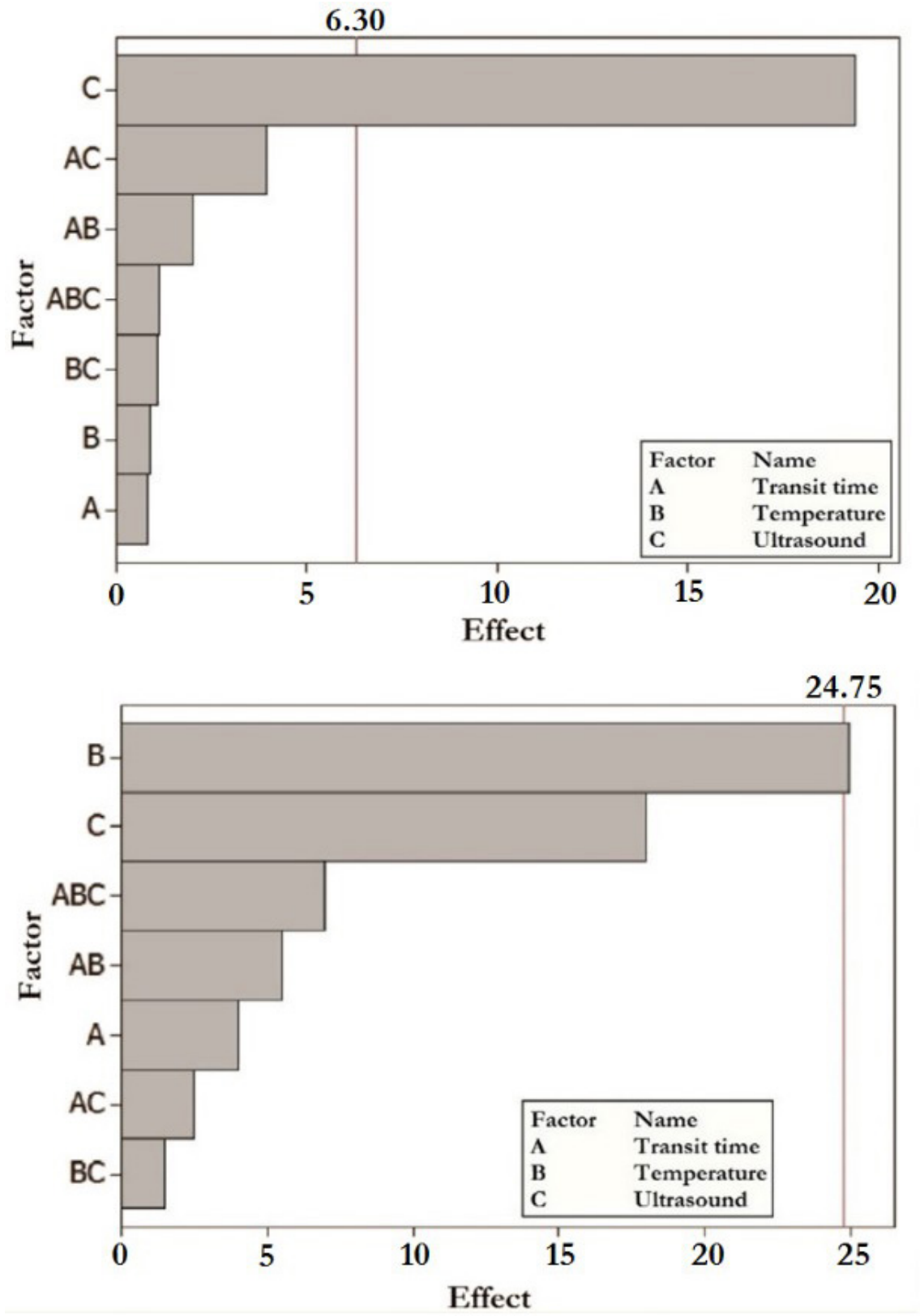

Fig. 9. Pareto diagram of the effects on the reaction. a) Efficiency. b) Response time.
Source: Authors.

Previous researches have implemented experimental designs to optimize the production of biodiesel. The Universidad de Córdoba (Colombia) [37], a design type 3^2 with variations of the stirring speed and catalyst concentration is proposed, reaching a conversion rate of 90%. At the Complutense University (Spain) [38], a design type 3^2 was implemented, with temperature and catalyst concentration variation reaching a maximum conversion of 98%. In Universidade Federal do Rio Grande do Norte (Brasil) [39], a design type 5^4 with variations of molar ratio, catalyst concentration, ultrasound amplitude, and pulse vibration is followed, with a 100% conversion rate. NITT and PSG College of Technology (India) [40], a design type 2^3 is established, with a variation of the molar ratio, catalyst concentration, and reaction time, achieving a maximum conversion of 98.9%; the biodiesel characterization in the research above is by gas chromatography and nuclear magnetic resonance. In this research, the biodiesel characterization is carried out indirectly with the measurement of glycerin formed [41]. Under the proposed experimental design, a conversion rate of 95.7% was obtained, but guaranteeing an increase in the process response speed to obtain an efficient and effective reaction.

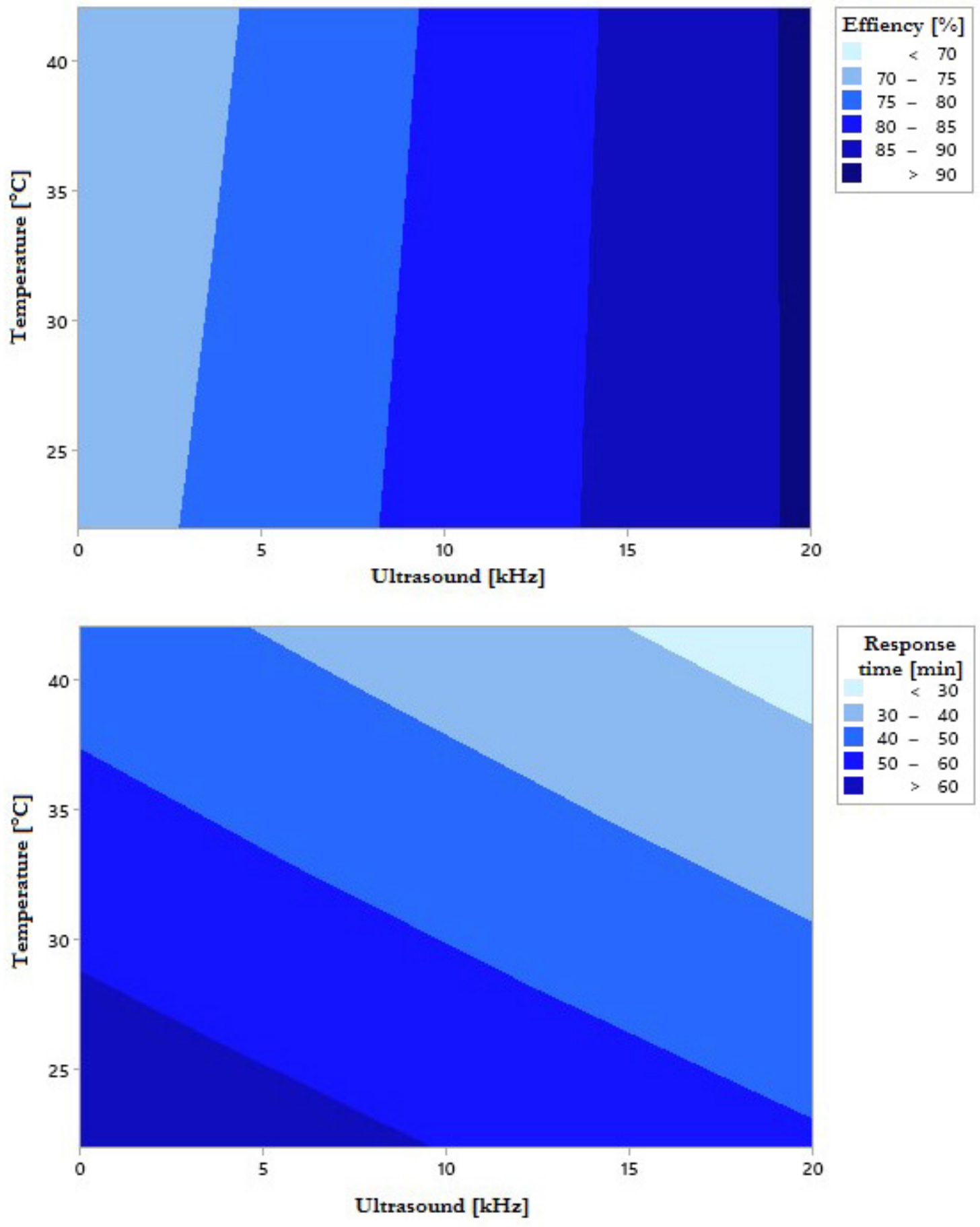

Fig. 10. Contour plot depending on temperature and ultrasound. a) Efficiency. b) Response time.
Source: Authors.

IV. Conclusions

A design of factorial experiments was carried out to determine the influence of temperature, ultrasound, and transit time on the efficiency and response time of the transesterification reaction of castor oil for the biodiesel production. The analysis of variance ANOVA, with a 95% reliability, determined the efficiency of the reaction concerning the studied factors; it only depends on the ultrasound; and the response time of the reaction depends on the temperature and ultrasound, obtaining times of formation of product four times faster.

The influence of the mixing temperature, the ultrasound intensity, and the transit time on the efficiency and response time of the transesterification of castor oil for the production of biodiesel was evaluated, using the design of factorial experiments and ANOVA analysis. It was determined that the efficiency of the reaction depends on the ultrasound; for its part, the reaction response time depends on the temperature and ultrasound, obtaining a production four times faster than the benchmarks.

In the tests carried out, the highest efficiency of the obtained reaction was presented with a transit time of six minutes, with the incidence of ultrasound at room temperature with a value of 95.7%. The highest reaction response time was obtained with a transit time of six minutes with an incidence of ultrasound at 315.15 K, with 4.85% formation per minute, for 14 minutes.

A biodiesel production recipe was accomplished, under indirect measurement of glycerin, with response rate analysis. With this reached a biodiesel production method with low cost in the obtaining and characterization of the final product. Both aspects are relevant at the industrial level; further research will focus on a bigger scale implementation and better conversion percentage.

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