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A Study on Production of Biodiesel from Waste Frying Oil

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Biodiesel is a highly effective fuel with the same efficiency such as diesel oil derivative only accompanied by moderate engine modifications. This type of biofuel can be obtained from renewable sources such as vegetable, animal or frying oils. In this context, waste frying oils transesterification was studied to obtain the maximum value of biodiesel. Transesterification reactions were carried out between 70 to 90 min using waste frying oils (WFOs), methanol, and sodium hydroxide as catalyst. In order to determine the best conditions for biodiesel production, a series of experiments were carried out, using catalyst percent and temperatures in a range of 0.16 to 1.84% and 40 to 90°C, respectively. The highest biodiesel production was obtained using catalyst percent, temperature and reaction time of 0.5%, 80°C and 90 min, respectively. By statistical experimental design was possible to determine a mathematical model to predict the best operational conditions to maximize biodiesel production.

1. Introduction

In Brazil, the main raw material used for the biodiesel process is soybean. According to Ministry of Agriculture soybean production has been exceeded 82 million tons which could lead Brazil to be largest producer of biodiesel in the world (Silva et al., 2018). However, many negative factors have been interfered to increase the production such as large land for planting and its impact with the food industry. Therefore, other raw material alternatives have gained prominence as frying oil (Silva Filho et al, 2018). This type of residue has excellent technical characteristics such as low economical value, easy storage and many pre-treatment processes with same biodiesel production scheme from soybean oil. Daily, waste frying oil are discarded, almost totally in sanitary sewers without control water pollution and this generate an impact environmental. Moreover, the waste oil causes a reduction in the diameter of ducts due to solidification and sewer obstruction.
In cases when is discarded in landfill reaches the water table, water resources, rivers, streams and dams. Studies have reported for one litter of waste frying oil can contaminate about 25,000 litters of water (Bimendra et al., 2017). According to Da Silva et al., (2017), in Brazil about 2 % of waste frying oil is collected, that is, 98% of this material is irregularly discarded in sewerage. Several governmental entities with private companies are organized to incentive for waste frying oil collection programs from the Brazilian population, and according to the Brazilian Union of Biodiesel and Bioquerosene (Ubrabio, 2018) the growth of the collection of used oil will contribute to the generation of new jobs and the regularization recyclable materials. For this reason, in this work is study the operational variables effects to aim biodiesel production from waste frying oil by experimental methodology.

2. Materials and experimental methodology

Waste Frying oil samples were obtained from a local restaurant at Rio de Janeiro City. The experimental was carried in the laboratory of the university Estácio de Sá and the procedures separed in three stages: pre-treatment, physicochemical characterization and transesterification reaction.

2.1 Pre-treatment

The raw material derived from the waste frying oil has a high fatty acid content and transesterification catalytic reaction is not enough to separate the phases. For this reason, to increase the reaction efficiency is necessary use sodium or potassium to avoid the saponification process of fatty acids (Da Rosa, 2013).

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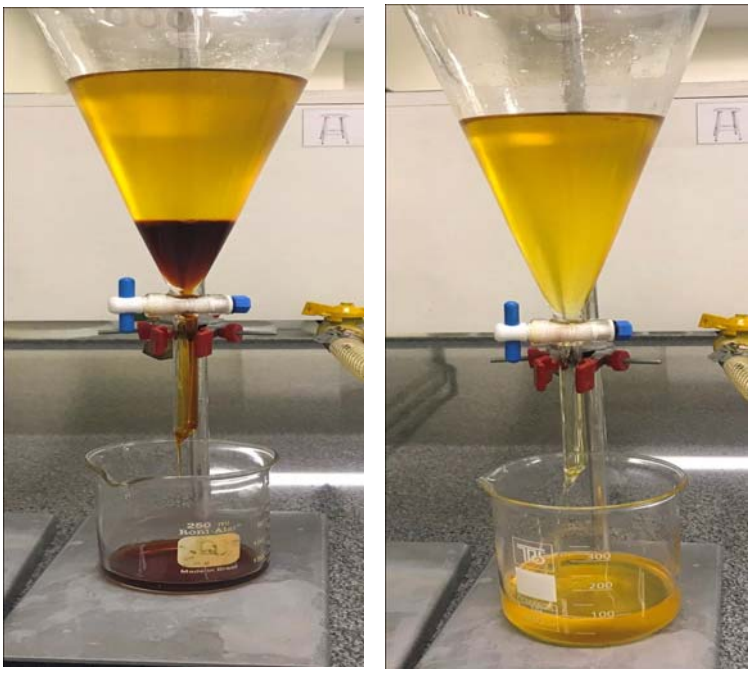
Samples have contained several contaminants such as water, free fatty acids, particulates and phospholipids. First, sample were dried and filtered to eliminate water and solid particulates. After that, free fatty acid (FFA) were measured by acid-base titration as number of potassium hydroxide (KOH) milligrams required to neutralize the FFA present in 1 g of sample. Finally, densities of samples were calculated. Biodiesel produced from vegetable and animal oils is less compressible than petroleum derivatives because they are denser.



(a) *(b)*

Figure 1: transesterification reaction products: (a) glycerine phases; (b) biodiesel.

2.2 Transesterification reaction

In Brazil is commonly used the alkaline transesterification to produce biodiesel. However, other processes can also be used such as cracking and esterification. The effect of catalyst, temperature and reaction time and their quadratic and cubic interactions on the yield of biodiesel and glycerine of waste frying oil samples has been investigated. The catalyst percentage, temperature and reaction time were carried out in the range of 0.5 – 1.0 % w, 50 – 80 °C and 70 – 90 min, respectively. Table 1 shows the experimental statistical planning with the process variables. The statistical software STATISTICA 7 from Statsoft Inc was used to create the design matrix.

For each experiment 300 ml of oil and 60 ml of methanol anhydrous (high purity) were used. Moreover, catalyst and methanol were mixed. The percentage of catalyst is proportional to grams of 300 ml of oil. In the other hand, alcohol and catalyst mixture was carried out at 50 ° C in a closed Erlenmeyer flask prior to mix with the oil. After the oil was heated to desire temperature, alcohol and catalyst mixture were added to flask on the reaction time according to experimental statistical planning. Finally, transesterification product was transferred to separatory funnel about 24 hours until form two phases (Figure 1). Biodiesel and glycerin from each experiment were collected and weighed.

The glycerin is a common residue from of oils used to produce biodiesel and it is generated on average from 10% to 12% in relation to mass of vegetable or animal oil. Glycerol is known as glycerol or propanetriol, an organic compound belonging to the alcohol function, has several applications, mainly in the cosmetics pharmaceutical industry due to be a great humectant (Maia, 2015).

Table 1: Design matrix with independent process variables

Experiment	Catalyst (%w)	Temperature (°C)	Time(min)
1	0,5	50	70
2	0,5	50	90
3	0,5	80	70
4	0,5	80	90
5	1,5	50	70
6	1,5	50	90
7	1,5	80	70
8	1,5	80	90
9	0,16	65	80
10	1,84	65	80
11	1	40	80
12	1	90	80
13	1	65	63
14	1	65	97
15	1	65	80
16	1	65	80
17	1	65	80

3. Results and discussion

The analysis of statistical design of experiment to identify the significant variables was performed by Statistica 7.0 ® software. Table 2 shows the results of density, yield of biodiesel and glycerin of the waste frying oil transesterification reaction.

Table 2: Density, yield of biodiesel and glycerin of the waste frying oil transesterification reaction

Experiment	Biodiesel (% w)	Glycerine (% w)	Density (g / ml)
1	9,87	90,20	0,870
2	9,54	91,39	0,860
3	60,23	40,60	0,970
4	98,43	13,60	0,960
5	14,61	87,24	0,880
6	9,83	91,41	0,880
7	79,76	30,39	0,940
8	78,62	30,98	0,930
9	13,47	88,34	0,880
10	16,46	85,58	0,900
11	10,25	90,43	0,850
12	61,86	40,95	0,970
13	14,59	87,20	0,900
14	10,07	90,89	0,880
15	11,63	89,87	0,870
16	8,97	92,21	0,870
17	11,49	90,03	0,870

3.1 Statistical analysis of biodiesel conversion

The linear (L) and quadratic (Q) effect of the temperature as well as the quadratic effects of the catalyst and time was most significant at a 95% confidence level. The linear effect of the catalyst and the combination between temperature and catalyst are not significant. The Pareto chart (Figure 2) shows the individual effects of the variables as well as their interactions. The effect will be significant to the right of the red line at 95% confidence level.

Figure 3 shows the results of response surface as a function of most significant term to biodiesel and glycerine, respectively. According to statistic results, the linear effect of catalyst percent is not significant. In order to optimize the biodiesel production, the reaction must be operated in a range of temperature of 60 - 90 °C. Furthermore, Figures 3(b) shows biodiesel conversion versus temperature and time. It is observed that response is rising with an increase in temperature and reaction time.

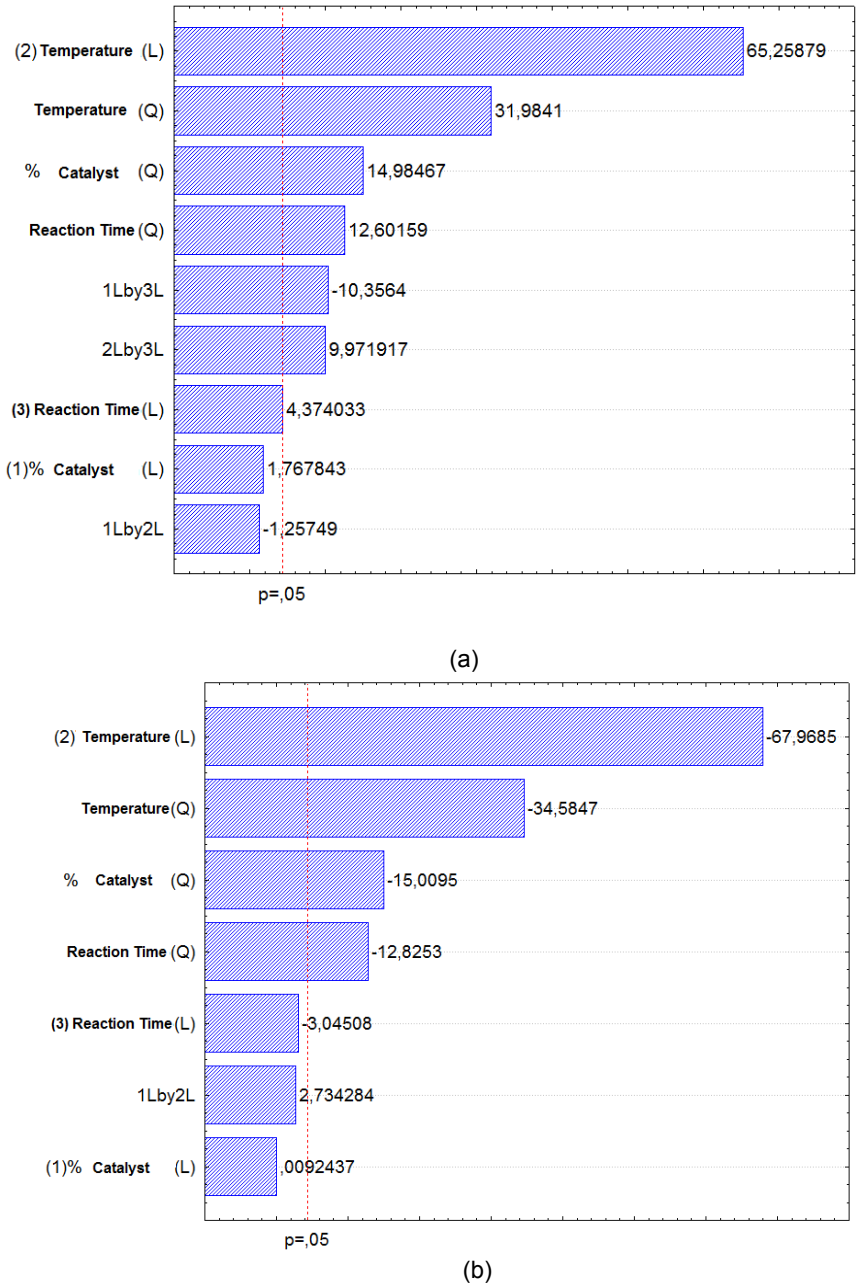


Figure 2: Response of Pareto chart of standardized effects: (a) biodiesel conversion; (b) glycerin yield.

In other hand, maximum glycerin production could be obtained in temperature ranges about 50-70 ° C and 1 w% of catalyst. Moreover, Figure 3(d) shows that glycerin yield increase in a relation of catalyst of 0.6-1.0 wt% and reaction time about 60-70 min.

Linear and quadratic terms of temperature (T) and time (t), quadratic term of catalyst (C) percent and two-factor interaction of catalyst/time and temperature/time are significant effects for biodiesel yield model ($\%C_{biodiesel}$). In addition, linear and quadratic terms of temperature (T), quadratic terms of catalyst (C) percent and reaction time, two-factor interaction of catalyst/time and time/temperature are significant effects for glycerin yield model ($\%C_{glycerin}$). Table 3 shows the final mathematical models in terms of coded factors by statistical design of experiment with a confidence interval of 95 %. Adjusted R-squared (R^2) for the biodiesel and glycerine models shows in the table below was 0.79713 and 0.79052, respectively.

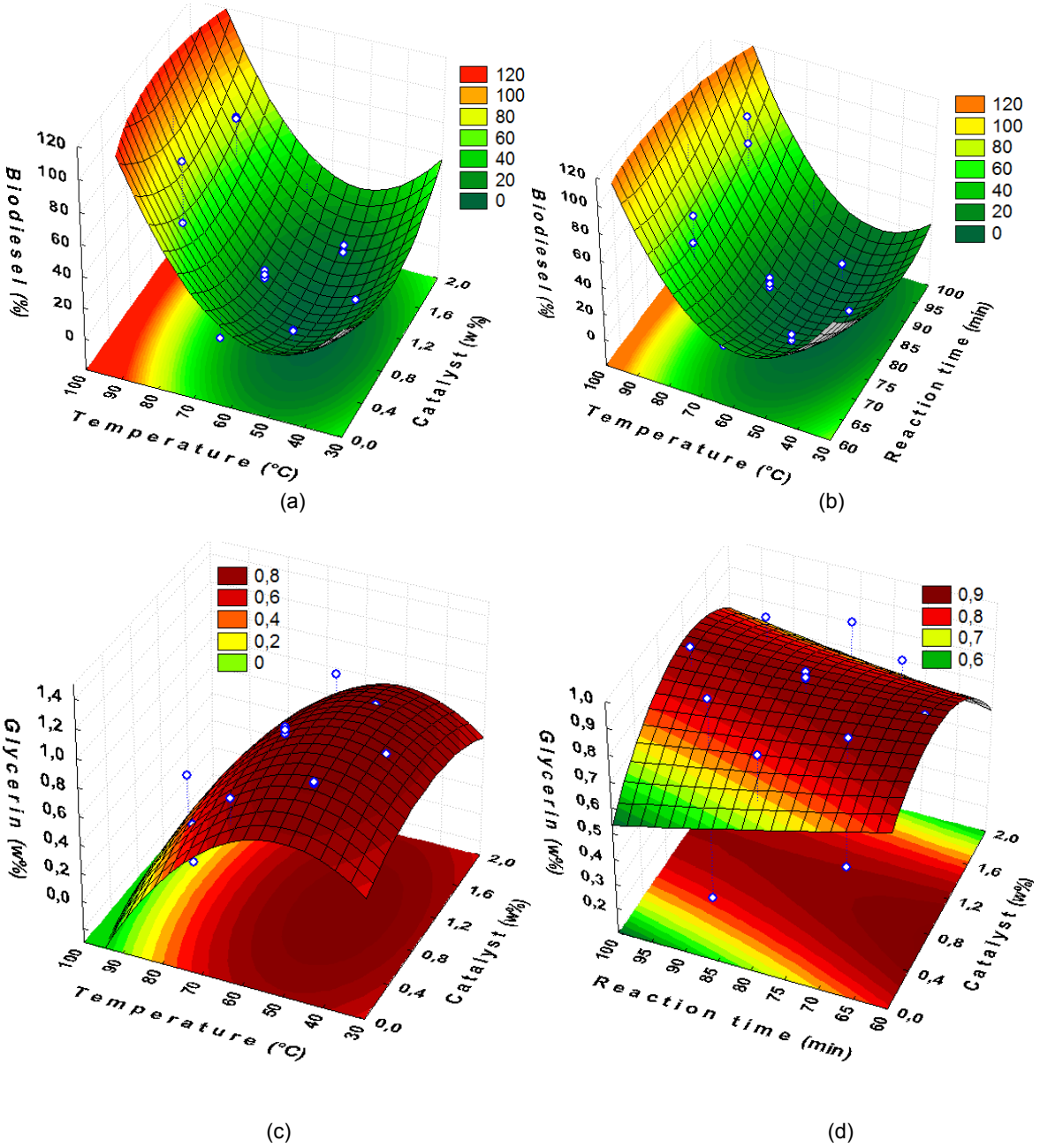


Figure 3: Response surface for biodiesel and glycerin (wt %) of waste frying oil transesterification reaction: (a) biodiesel yield: temperature vs catalyst; (b) biodiesel yield: temperature vs reaction time; (c) glycerin yield: temperature vs catalyst; (d) glycerin yield: reaction time vs catalyst.

Table 3: Mathematical models for biodiesel and glycerin yield (wt%) waste frying oil transesterification reaction

Response term	Mathematical model	Equation number
Biodiesel (wt%)	$\%C_{biodiesel} = 673{,}0705 + 29{,}4030C^2 - 9{,}5136T + 0{,}0651T^2 - 10{,}5139t + 0{,}0570t^2 - 0{,}72 + 0{,}0351T \cdot t$	(1)
Glycerin (wt%)	$\%C_{glicerina} = -67{,}8249 - 20{,}2296C^2 + 6{,}5105T - 0{,}0547T^2 + 0{,}0011t^2 + 0{,}5091C \cdot t - 0{,}0126T \cdot t$	(2)

4. Conclusions

The study of the transesterification reaction with base catalysis was performed by 17 experiments with different operational variables (temperature, catalyst percent and reaction time). The highest biodiesel production was obtained using catalyst percent, temperature and reaction time of 0.5%, 80°C and 90 min, respectively. By statistical experimental design was possible to determine a mathematical model to predict the best operational conditions to maximize biodiesel production. In this analysis, higher production of glycerine (about 1.2 wt%) could be obtain in a temperature and catalyst percent of 50 °C and 1 wt%, respectively. Finally, the developed mathematical models are accurate as the percentages of error in prediction are in good agreement.

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