v. 2, n. 25, 2022

Journal of
Engineering
Research

PRODUCTION OF ETHYL BIODIESEL WITH NAOH CATALYSIS FROM RESIDUAL FRYING OIL FROM SELECTIVE COLLECTION IN FOZ DO IGUAÇU/PR

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Abstract: The technical and economic feasibility of biodiesel production by ethyl transesterification with homogeneous basic sodium hydroxide catalysis was analyzed, from residual frying oil (ORF) collected by selective collection in Foz do Iguaçu/PR - Brazil. The following mean values were found in the ORF: free fatty acids of 0.52%; specific mass of 912.53 kg/m^3; and kinematic viscosity of 34.25 mm^2/s. The average yield of the reaction was 87.87%. Biodiesel presented the following average values, meeting the ANP specifications: acidity index of 0.21 mgKOH/g; specific mass of 851.83 kg/m^3; and kinematic viscosity of 5.1 mm^2/s. The TGA and DTG analyzes indicated a certain homogeneity in the ORF and, for biodiesel, evidence of total conversion of triglycerides into ethyl esters. The investment analysis also showed a favorable result: positive net present value; internal rate of return of 7.01%; profitability index of 1.10; and payback time of 8.86 years.
Keywords: Biofuel. Ethyl biodiesel. Basic catalysis. Residual frying oil. Selective collect.

INTRODUCTION

The process most used today to obtain biodiesel is the transesterification of vegetable oil or animal fat (mainly soybean oil in Brazil), carried out in the presence of acid, basic or enzymatic catalysts, whether homogeneous or heterogeneous, using a short chain alcohol, such as methanol, ethanol, propanol or butanol (SALTARIN, 2013).

Corrosivity, reaction conditions, cost and availability must be considered when choosing a catalyst. In these aspects, alkaline catalysts such as potassium hydroxide (KOH) and sodium hydroxide (NaOH) stand out, which are the most used, due to their greater availability and low cost (ENCINAR et al., 2007; MATH et al. 2007; SALTARIN, 2013; SILVA, 2011). Sodium hydroxide, also known with the name: soda, is freely traded with a high purity content in Brazil, which facilitates its access.

In the country, ethanol is obtained from renewable biomass (sugarcane), and is cheaper and more abundant than methanol and other short-chain alcohols, which makes its use quite promising. On the other hand, methanol is more toxic compared to ethanol and is usually imported and obtained from petroleum derivatives. Tests indicate that when ethanol from sugarcane is used to obtain pure biodiesel (B100), the reduction in greenhouse gas emissions can reach 90% (CHRISTOFF, 2006; OLIVEIRA and ROSA, 2003).

Residual frying oil (ORF) as a raw material reduces biodiesel production costs to about 60%, reducing the use of human and animal food sources (edible oils) and the use of arable land (GUPTA et al, 2015). According to Diya'uddeen et al. (2012) it is possible to obtain an optimal yield of biodiesel (about 99.3%) using ORF as a raw material, although it usually presents a high degree of degradation, as it is heated to temperatures close to 200°C, in the presence of of oxygen and water, for a varied time before being discarded.

The disposal of the ORF via drainage (urban sewage) or sanitary landfill can cause water and soil pollution and disturbances in ecosystems (YAAKOB et al., 2013). Therefore, aiming at the use of a potentially polluting waste (ORF) already collected in the city by the Cooperativa dos Agentes Ambientais de Foz do Iguaçu/PR - Brazil - COAAFI, this article analyzed the technical and economic feasibility of producing biodiesel by ethyl transesterification with NaOH by the cooperative itself.

METHODOLOGY

The oil collected from households in the municipality, by COAAFI, is stored in the sorting centers in 60-liter drums. For the analysis of the oil, four collections were carried out, from containers that were already full, with intervals of 15 days between them, during the months of August and September 2019. The pre-treatment of the ORF was carried out as suggested by Christoff (2006), with heating to remove moisture, and vacuum filtering to remove residues from frying.

TRANSESTERIFICATION REACTION

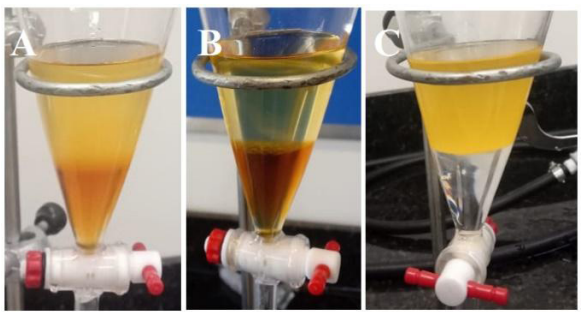
The 4 ORF collections were reacted in triplicate. The determination of the mass of the reagents and the reaction procedure to obtain the Ethyl Biodiesel with NaOH catalysis (BENaOH) are based on the studies by Christoff (2006). In this one, an excess of alcohol in the proportion of 1:12 in moles (ORF:alcohol) was used and the amount of catalyst added corresponded to 0.5% of the mass of ORF used, plus an additional amount of NaOH determined from the % AGL of the oil.

After the reaction time, the mixture was taken to a rotary evaporator to evaporate excess alcohol. Evaporation was conducted for 10 min under the following conditions: 45 RPM rotation; cold water flow of about 30 mL/s; water bath at 60°C; and vacuum pump pressure at -900 mbar.

At the end of the evaporation, the mixture was transferred to a decantation flask, where the spontaneous separation of its phases took place. Figure 1 – A illustrates how the samples were taken from the rotary evaporator, and Figure 1 – B shows the phase separation after a period of rest (between 4 and 8h at room temperature).

After decanting, the glycerol was removed and then the biodiesel underwent a consecutive wet washing process to remove excess alcohol, catalyst, residual glycerol and soaps formed during the reaction. Washing was carried out in the separating funnel, with the addition of 10 mL of distilled water heated to 80°C to the biodiesel, followed by light manual agitation to allow better contact between the phases (CHRISTOFF, 2006).

The mixture was allowed to stand at room temperature until it cooled and the separation clearly occurred. Then, the denser phase was drained off. This procedure was repeated 3 times until the pH approached neutral, and the aqueous phase was colorless as shown in Figure 1 – C.



A – Mixture out of the evaporator. B – Phase separation after 4-8h: ethyl ester (upper phase); glycerol (lower phase). C – Phase separation from the last wash: ethyl ester (upper phase); water (lower stage).

Figure 1 - Decanting and washing process.

Source: Self elaboration.

After washing, the biodiesel was transferred to a 100 mL beaker and placed in an oven at a constant temperature of 90°C for 2 h to remove the alcohol and/or water still present. Then, with the biodiesel at room temperature, the sample was weighed to determine the mass yield (η) using Equation (1).

$$\eta = \left(\frac{m_{BENaOH}}{m_{ORF}}\right).100\% \qquad (1)$$

Where:

η - Yield of the transesterification reaction (%);

m_{BENaOH} - Mass of biodiesel obtained (g);

m_{ORF} - ORF mass (g).

CHARACTERIZATION OF RESIDUAL OIL AND BIODIESEL OBTAINED

The percentage of free fatty acids (FFA%) was analyzed according to the methodology of Christoff (2006). Similar to the AGL% of the ORF, the acid value (AI) of the BENaOH obtained was determined by titration, according to the ASTM D-664 method indicated by the ANP (2021).

In the ORF and BENaOH were analyzed: the specific mass (MS) at 20°C, determined by the volumetric method used by Dib (2010); and the kinematic viscosity at 40°C, obtained by a Brookfield viscometer, model DV3T Extra Rheometer, equipped with a Thermosel for heating the samples.

Thermogravimetry (TGA) and Derived Thermogravimetry (DTG) analyzes were also performed on the Perkin Elmer model STA 8000 analyzer, using 10 mg of sample per analysis on an alumina support, with a temperature range of 30 to 600 °C, heating of 10 °C/min and flow of inert gas (N2) of 50 mL/min.

RESULTS AND DISCUSSIONS

CHARACTERIZATION OF RESIDUAL OIL

The AGL% is a parameter that directly affects the cost of oil pretreatment and the yield of the transesterification reaction. Thus, the ideal is for ORF to have a low FFA content (<0.5%), similar to soybean oil (AGUIEIRAS et al., 2014). The average value of FFA% obtained for the analyzed collections was 0.52%, close to the value considered ideal.

The average DM at a temperature of 20°C, determined for the ORF (912.53 kg/m^3) was lower than the limit established by ANVISA (1999) for refined soybean oil (between 919 and 925 kg/m3). This is probably due to the degradation of the oil in the frying process, which can affect the yield of the transesterification reaction, favoring saponification. The average viscosity of the ORF collected was 34.25 mm^2/s, slightly higher than the ideal value for the traditional transesterification reaction, which is 34 mm^2/s for soybean oil (QUEIROZ, 2011).

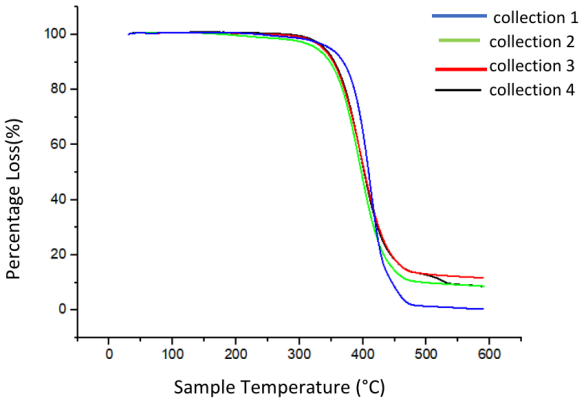
Table 1 presents the mean results of the ORF analyses, their standard deviations (SD), and the values considered as reference. The high DP can be attributed to the way of disposal and storage of ORF, which uses used containers still containing other substances, such as cleaning products.

Specifications	Mean values obtained (ORF)	Reference Values (Soybean Oil)
AGL (%)	0,52 ± 0,37	<0,5% (AGUIEIRAS et al., 2014).
Specific mass at 20 °C (kg/m^3)	912,53 ± 0,85	Entre 919 e 925 kg/m3 (ANVISA, 1999)
Kinematic viscosity at 40 °C (mm^2/s)	34,25 ± 2,75	34 mm^2/s (QUEIROZ, 2011)

Table 1 - Analysis of the COAAFI ORF.

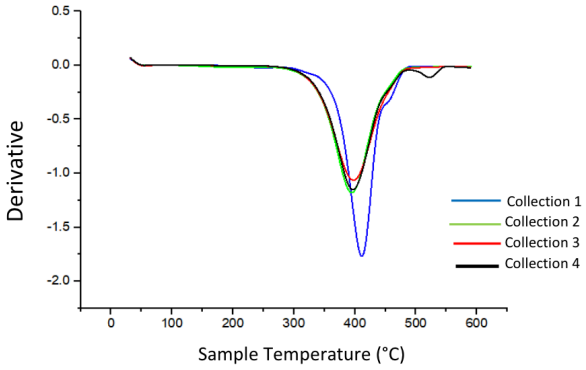
Source: Own elaboration.

Graphs 1 and 2 present the TGA and DTG analysis curves of the ORF samples, respectively. It is possible to observe the tendency of mass loss in a single step, at temperatures between 350°C and 495°C, referring to the gradual decomposition of the material. Occurring, on average, about 90% of the loss, which indicates a certain homogeneity of the substance (DANTAS, 2006).



Graph 1 - Analysis of the ORF TGA.

Source: Own elaboration.



Graph 2- Analysis of the ORF DTG.

Source: Own elaboration.

ORF TRANSESTERIFICATION REACTION

The η of the transesterification reaction for the ORF samples was 87.87%, higher than the value found by Christoff (2006) of 85%. This result can be attributed to the greater degradation of the oil used in their work, collected directly from commercial establishments (restaurants, cafeterias and hotels). The mean value of AI after purification and phase separation was 0.21±0.07 mgKOH/g. This result is within the standards established by the ANP (2021), which is a maximum of 0.50 mgKOH/g.

The DM at 20°C of BENaOH had its average value of 851.83±48 kg/m³, within the range established by the ANP (2021) (between 850 and 900 kg/m³). In the present study, the viscosity of the raw material was also considerably reduced, reaching an average value of 5.1±2.46 mm²/s, which is in the range of 3.0 to 6.0 mm²/s specified by the ANP (2021). The high standard deviation values are due to the heterogeneity of the samples. The AI, MS and viscosity of the esters obtained in the transesterification reactions are shown in Table 2.

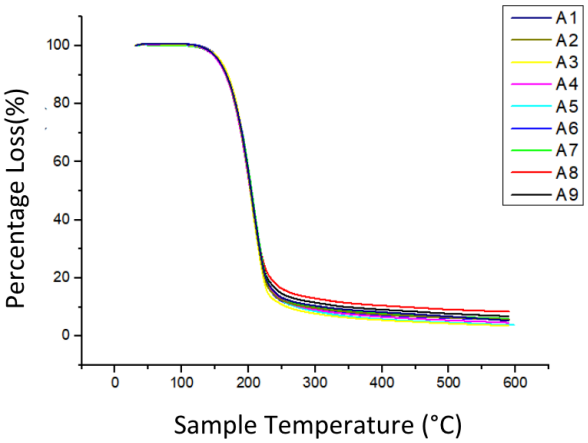
Specifications	Average values obtained	Parameters (ANP, 2021).
Acidity level	0,21±0,07 mgKOH/g	Max. 0,50 mgKOH/g
Specific mass at 20 °C	851,83±48 kg/m³	Between 850 and 900 kg/m³
Kinematic viscosity at 40°C	5,1±2,46 mm²/s	Between 3.0 and 6.0 mm²/s

Table 2- COAAFI ORF BENaOH Specifications.

Source: Own elaboration.

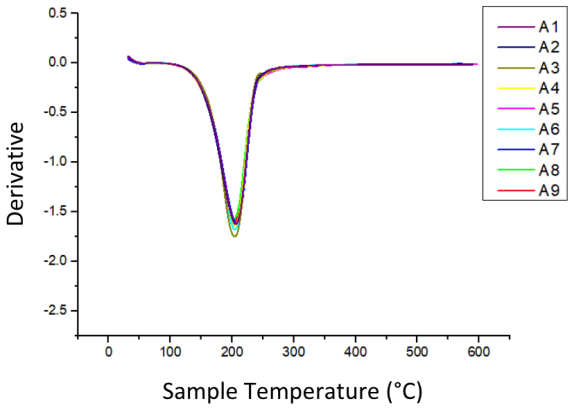
From the thermogravimetric tests carried out with the samples, it was possible to obtain the curve shown in Graph 3. The TGA/DTG analyzes of BENaOH showed mass losses in a single step, with the initial temperature of approximately 160°C, the peak at 200° C, and with about 98% loss up to 400°C.

Thus, it is possible to infer that the transesterification and purification of biodiesel may have been complete, as no decomposition steps resulting from alcohol, free fatty acids, triglycerides, glycerin, or larger molecules were identified, as can be seen from the DTG curves. of the ester samples, shown in Graph 4. These results are in agreement with results obtained by other authors in similar analyzes (SILVA, 2013; DANTAS, 2006).



Graph 3- BENaOH TGA Analysis.

Source: Own elaboration.



Graph 4- BENaOH DTG analyzes.

Source: Own elaboration.

Only for collection 3, it was not possible to separate the ethyl ester and glycerol produced, making it impossible to analyze the biodiesel obtained. It is likely that on the date of collection, the drum was recently fed with oil contaminated by some substance that impaired the reaction process.

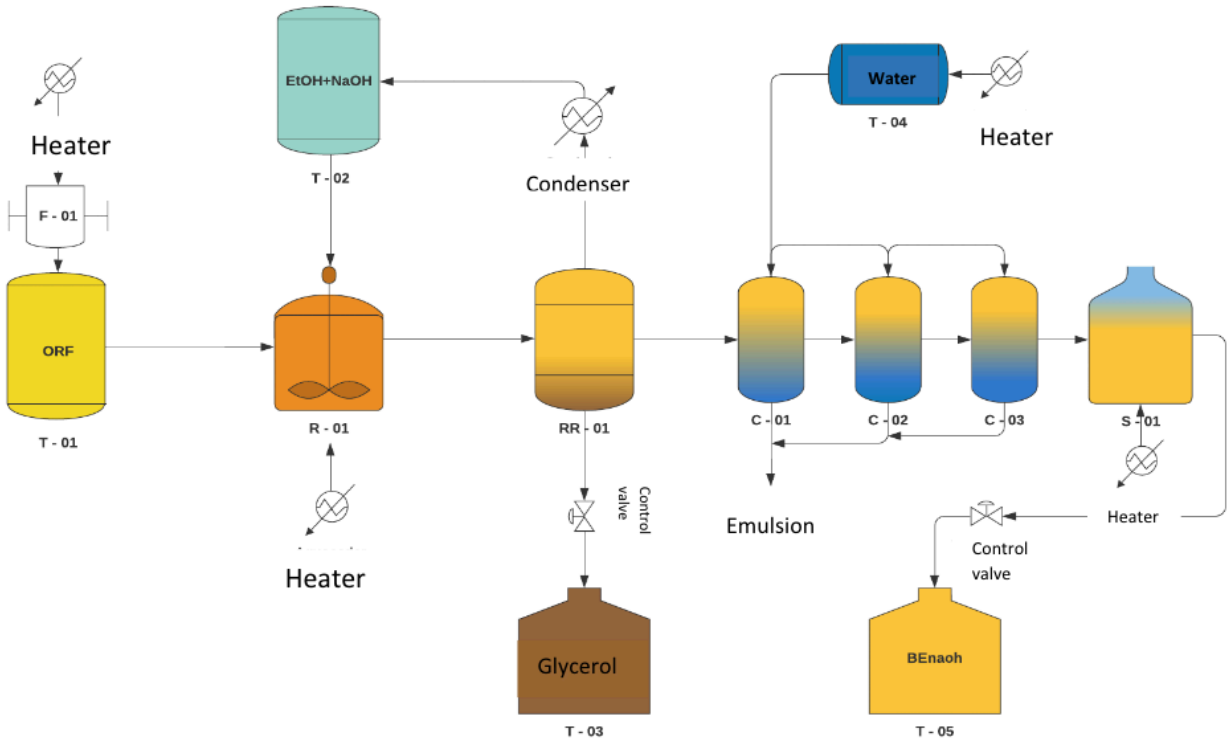
INVESTMENT ANALYSIS

From the preliminary analyzes carried out, it is possible to perceive the energy potential that the ORF collected by COAAFI has for the production of biodiesel. Therefore, it was possible to propose a process, based on studies carried out in the laboratory, which could be implemented by the municipal government in the cooperative, for the use of fuel by selective collection trucks. Diagram 1 presents the proposed process.

COAAFI has 6 sorting centers, and according to the cooperative, each center received, until 2019, on average, 60 liters of residual oil in approximately 10 days. Thus, all screening centers collected together, about 1080 L of ORF per month. At the time, selective collection covered half of the population of the municipality. According to the city hall, the forecast is that by the end of 2022 they will reach 100% of urban residences, which may represent a good increase in ORF collection as well. Assuming that this increase is 35% and knowing that the average efficiency of this study was 87.87%, it is possible to estimate a monthly production of approximately 1,298.94 L of BENaOH.

Based on the production process proposed here and through market research, it was possible to predict the investment analysis of the project, estimating the most used indicators as acceptance criteria: Net Present Value (NPV); Internal Rate of Return (IRR); Profitability Index (LI); Payback Time. In order to do so, plant designs that could adapt to the production process and the technological route proposed in this study were analyzed, at the lowest possible cost.

The selected Plant was the Biodiesel Plant - UB 200 of the company Biotechnos, considering that it can be adapted to the parameters mentioned in this study, and maintains its production capacity of 200 liters of biodiesel every 8 hours. To compose the cash flow outflows, in addition to the investment in the plant, the following were considered: raw material; labor; and overhead. The raw material refers to anhydrous ethanol and sodium hydroxide PA, which were estimated per month, considering, respectively, the official quotation plus ICMS and the best proposal among the evaluated budgets.



Identification of the following diagram:

F – 01: ORF filter;

T – 01: Filtered ORF storage tank;

T – 02: Catalytic solution tank (Ethanol and NaOH);

R – 01: Mixing reactor, under heating;

RR – 01: Reaction tank, for distillation and decantation;

T – 03: Residual glycerol storage tank;

T – 04: Wash water storage tank, under heating;

C – 01, C – 02 and C – 03: Columns for washing the ester obtained;

S – 01: Fuel dryer;

T – 05: BENaOH storage tank.

Diagram 1. Ethyl Biodiesel Production Process from Residual Frying Oil.

Source: Own elaboration.

In terms of labor, it was considered an hourly remuneration for an employee of the cooperative itself, temporarily relocated to operate the plant, which is completely automated, the value already includes taxes. In general expenses, preventive maintenance, periodic laboratory analysis, storage (storage of biodiesel in tanks) and the increase in water and energy expenses were estimated. Structural modifications were not considered for the installation of the plant because, according to the cooperative, the COAAFI sheds were recently renovated and can house various types of industrial equipment.

The inputs are made up of the avoided cost of buying diesel for the cooperative's trucks, and the sale of the crude glycerin that will be produced in the process, about 208.3 L. The value of crude glycerin (80%) varies widely in the market, but the average price of BRL 1,600/ton was considered (ALMEIDA, 2021). Table 3 summarizes the cash flow estimate for the investment analysis.

Investment	Initial value	Source
Biodiesel Plant Plant	R$ 138.000,00	[1]BIOTECHNOS, 2017.
Appetizer	**Monthly Values**	**Source**
Avoided cost of diesel	R$ 5.520,49	PRICE OF FUELS, 2021.
Crude Glycerol	R$ 249,96	ALMEIDA, 2021.
Total	**R$ 5.770,45**	
Outputs	**Monthly Values**	**Source**
Accounting Depreciation	R$ 1.150,00	BRAZIL, 2017.
Manpower	R$ 1.108,69	BRAZIL, 2021.
Feedstock	R$ 1.073,63	CEPEA, 2021; CHEMICAL UNION, 2021.
General expenses	R$ 800,00	VEDANA, 2011.
Total	**R$ 4.132,32**	

[1]The plant is the same as in the referenced catalog, but with the value adjusted by the supplier in the budget in March 2021.

Table 3- Cash flow.

Source: Own elaboration.

It was possible to estimate the design indicators presented in Table 1 by posting these inputs and outputs for the period of 10 years (half of the plant's useful life, according to the manufacturer), and considering a Minimum Attractive Rate (TMA) of 5% per year. The positive NPV, the IRR greater than the TMA and the IL above one with a Payback Time of 8.86 years, indicate the attractiveness and acceptance of the project.

Indicator	Value
Net Present Value (NPV)	R$ 13.790,43
Internal Rate of Return (IRR)	7,01%
Profitability Index (LI)	1,10
Payback Time	8,86

Table 1- Project Indicators.

Source: Own elaboration.

FINAL CONSIDERATIONS

In this study, samples of raw material were collected from COAAFI. The pre-treatment of the oil and the synthesis and purification of biodiesel were carried out. The ORF samples and the BENaOH obtained from each collection were characterized. Although one of the collections did not show a satisfactory result, probably due to inadequate storage, and despite the difficulties inherent in the transesterification reaction with ethyl alcohol and NaOH, it was possible to obtain indications of the technical feasibility of the reaction and the quality of the fuel produced.

Other analyzes still need to be done, such as moisture content, oxidative stability and flash point. But, as a preliminary study of the raw material collected from homes in Foz do Iguaçu/PR, the results proved to be relevant and the project is attractive. Some of the main specifications of the fuel obtained are within those recommended by the ANP (2021) as shown in Table 2, and the investment indicators recommend the acceptance of the

project. Therefore, it is expected that this research will help to compose the studies for the feasibility of the use of biodiesel obtained in a sustainable way.

THANKS

The authors thank PRPPG for the master's scholarship granted (BID Number 54/2020/ PRPPG) and for the financial support (Notice number 137/2018/PRPPG- Triple Frontier Agenda I and Notice Number Researcher Integration).

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