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Production, Characterization, and Exergy Analysis of the Soybean Biodiesel Produced by Laboratory Scale

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Abstract

The world's hegemonic energy model is mostly based on the burning of fossil fuels. The heavy reliance on oil, coal, and natural gas for power generation as well as the pollution resulting from burning such fuels is a growing global concern. Biodiesel from the transesterification of vegetable oils is a promising alternative source for diesel engines. The ANP, National Agency of Petroleum, Natural Gas and Biofuels, regulates fuels in Brazil. In this work, soybean biodiesel was produced on a laboratory scale through an alkaline transesterification reaction. An analysis of the exegetic efficiency of the process, the physical-chemical characterization of biodiesel, and a brief discussion on the following parameters were carried out: higher and lower calorific value, water content, specific mass at 20°C, kinematic viscosity at 40°C, acidity index and oxidative stability, verifying the adequacy to the parameters of ANP Resolutions nº 45/2014 and nº 798/2019. Considering the exergetic efficiency of 63.42% and the physical-chemical characterization, despite the non-conformities in water content and oxidative stability, soybean biodiesel proved to be a satisfactory alternative for renewable fuel.

Keywords: Soybean oil; Biodiesel; Transesterification; Exergy

Introduction

In recent decades, the need for energy has been increasing due to increased industrialization and population growth. Thus, alternative fuels have stood out due to environmental issues, such as greenhouse gas emissions, and the scarcity of fossil fuels [1].

The production of energy is indispensable for the maintenance of daily human activities, and most of the fuel comes from non-renewable sources such as oil, gas, and coal. Such fuels imply a high level of pollution and a predictable depletion of their sources, which increases the concern with the search for alternative ways to generate energy [2-4].

Brazil is a country that has a privileged configuration of the energy matrix in terms of sustainability since around 44.7% of energy consumption is met by renewable sources, Figure 1 [5]. However, most cargo transportation in the country takes place by road, which implies a high

consumption of diesel oil [6]. Among the liquid biofuels produced in the country, the two that stand out the most are ethanol, obtained from sugarcane, and on a growing scale, biodiesel, which is produced from vegetable oils or animal fats and added to petroleum diesel in a 10% proportion.

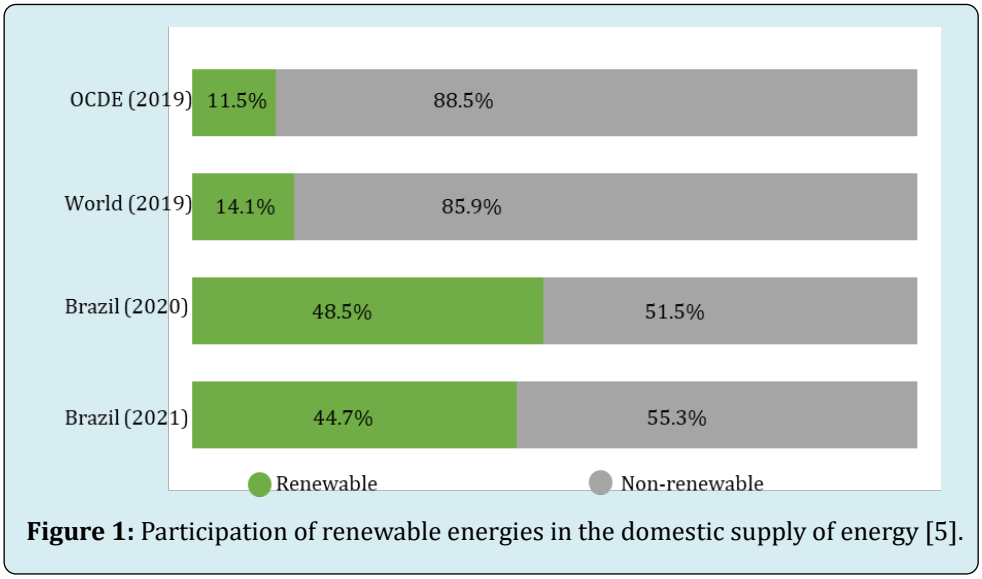

Figure 1: Participation of renewable energies in the domestic supply of energy [5].

Brazil was a world pioneer in the use of biofuels through the National Program for the Production and Use of Biodiesel (PNPB), an Interministerial Program of the Federal Government whose objective was to implement the production and use of biodiesel in a sustainable way, with a focus on including social and regional development, also aiming to reduce dependence on imports of the corresponding fossil derivative: diesel [7].

Biodiesel is a biodegradable fuel derived from several renewable sources [8,9], and can be produced from edible vegetable oils (first generation), such as soybean [10], palm [11], sunflower [12], rapeseed [13], linseed [14], coconut [15], canola [16]; inedible vegetable oils (second generation), such as karanja [17], pine nut [18], jojoba [19], neem [20], castor [21]; or from residual frying oils [22,23], and animal fats (Third generation) such as fish fat [24], chicken fat [25,26], tallow, white fat or lard [27-29]. In Brazil, about 90% of the raw material is concentrated in soy, which should reach a productivity of up to 9 billion liters by 2024 with the increase in the biodiesel content in the composition of automotive diesel [30].

The purity of the raw material used in biodiesel is essential in the production process, since some substances such as free fatty acids, gum, and phospholipids, among others, can compromise the quality of the biofuel depending on the production method. The storage of the product must be carried out in a such way as to prevent the degradation of biodiesel, commonly caused by temperature and luminosity variations, oxidation, and moisture absorption [31].

In the production of soybean biodiesel, alkaline transesterification is the most used chemical process, with glycerin as a by-product with wide reintegration in the production chain. Biodiesel can be commercialized after going through purification processes to adapt to the quality specification and is intended mainly for application in compression ignition engines, reaching the physicochemical standards established by the National Agency of Petroleum, Natural Gas, and Biofuels (ANP).

In Brazil, pure biodiesel (B100) was obligatorily added to petroleum diesel in progressive proportions. Addition to fossil diesel began in 2004, on an experimental basis, and between 2005 and 2007, at a 2% content, with voluntary commercialization [32]. The obligation came in Article 2 of Law nº 11,097/2005, which introduced biodiesel into the Brazilian energy matrix, instituting the initial addition of 2% of biodiesel to common diesel at fuel stations in the country. Currently, this percentage is already at 10% (Law nº 13.263/2016), with projections of an increase (ANP, 2018).

According to the ANP, some parameters must be evaluated before distributing biodiesel. ANP Resolution No. 45 of August 25, 2014, establishes the quality control parameters to be met for the commercialization of biodiesel in Brazil. Among these parameters, the specific mass stands out, which is normally greater than diesel, directly influencing the burning of biodiesel due to the air/fuel ratio (stoichiometric mixture)

so that the higher the density, the higher the consumption [33]; the kinematic viscosity, which influences the variation in the injection pressure, which may cause wear on the faces of the rings and cylinders of the engines, and oil lubricity must be observed [34]; the water content, as humidity has the potential to favor microbial activity, which degrades the fuel, generates sludge and saturates filter elements more quickly, in addition to impairing the operation of the injection pump or diesel engine injector nozzle, also favoring processes corrosives in fuel distribution chain equipment [35,36]; the acidity index, which evaluates the content of free acids in biodiesel, normally originating from free fatty acids that confer greater acidity, also resulting in corrosion [37]; and, finally, oxidative stability, which evaluates the degradation of biodiesel by oxidation, causing fuel crystallization and the formation of sludge and impurities that can clog and damage engine components [36].

Another important biofuel property, although not regulated by the ANP, is the calorific value, which is divided into Higher Heating Value (HHV) and Lower Heating Value (LHV). The HHV is defined as the amount of heat released by a specified quantity once it is combusted and the products have returned to a temperature of 25°C. It takes into account the latent heat of the vaporization of water in the combustion products and is useful in calculating heating values for fuels. The LHV is defined as the amount of heat released by combusting a specified quantity and returning the temperature of the combustion products to 150 °C. It assumes that the latent heat of the vaporization of water in the fuel and the reaction products is not recovered. As the temperature of the combustion gases is very high in endothermic engines, the LHV becomes a measure of greater importance for the fuel [33].

Based on the sustainability of the biodiesel production chain, evaluating the exergy flow of the process by accounting for waste, comparing different energy sources, and determining the exergy efficiency is very important in defining economic and environmental policies for the use of resources [38].

This work aims to evaluate the exergy efficiency of each stage of the process, characterizing physicochemically and analyzing the exergy in the production of soybean biodiesel on a laboratory scale**,** to contribute to the development of renewable energy sources; as well as make a brief discussion about the important parameters for the quality of soybean biodiesel as a fuel. The novelty of the work was to determine the total exergy of the soybean biodiesel production on a laboratory scale once it is more common to find studies on an industrial scale. In addition, the Lower Heating Value (LHV) and Higher Heating Value (HHV) were calculated using the mass composition of soybean biodiesel as a function of the carbon, hydrogen, and oxygen percentages.

Methods

Acidity Number

In a 100 mL erlenmeyer flask, 2.0 g of soybean oil, 25 mL of a solution of ethyl ether/ethyl alcohol (2:1), and two drops of 1% phenolphthalein alcoholic solution are added. The mixture is titrated with 0.1 mol/L sodium hydroxide (NaOH) solution. The acid number is determined according to equation (1), where V_{NaOH} is the volume of titrant consumed, f_{NaOH} is the correction factor for the solution of 0.1 mol/L of NaOH, and the mass of the oil in grams.

$$\text{Acidity number}(AN) = \frac{V_{NaOH}(mL) * f_{NaOH} * 5,61}{m(g)} \quad (1)$$

where AN is the acidity number of the sample, V_{NaOH} is the volume of the titrant (mL), f_{NaOH} is the correction factor for the solution and m is the mass of the sample (g).

Biodiesel Production – Alkaline Transesterification, Washing, and Drying

In a round bottom flask, 200.117 g of soybean oil, preheated to 60°C (15 minutes), were added to an alcohol/catalyst mixture containing methanol (MeOH) and potassium hydroxide (KOH) prepared according to the determined proportions by equations (2) and (3). The mixture is kept under stirring with a magnetic bar, with a condensation system, in a glycerin bath at 60°C for 60 minutes [39].

$$V_{MeOH} = \frac{mass_{oil}(g)}{density_{MeOH}} * 0,2 \quad (2)$$

$$M_{KOH} = mass_{oil} * \left(\frac{Purity_{KOH}(\%)}{100} + \frac{AN}{1000} \right) * \frac{1}{0,85} \quad (3)$$

where V_{MeOH} is the volume of methanol (mL), $mass_{oil}$ is the mass of oil (g), $density_{MeOH}$ is the density of methanol (g/mL), M_{KOH} is the molarity of KOH (mol/L), $purity_{KOH}$ is the purity percentage of KOH, and AN is the acidity number of the sample.

After the reaction time, the biodiesel is transferred to a 500 mL separation funnel, with three washes being carried out at intervals of 30 minutes, two with 20.0 g of distilled water at 30 °C and one with 20.0 g of distilled water at 90 °C. Biodiesel was dried in a 500 mL beaker under constant stirring (200 rpm) at a temperature of 105-110 °C for 30 minutes.

Density at 20°C

The determination of the density was carried out in a digital densimeter of the Anton Paar, model 4500, with an oscillating U-tube system, following the NBR 14065 standard.

Kinematic Viscosity at 40°C

The determination of the kinematic viscosity was carried out in a capillary viscometer in a thermostatic bath at 40 °C after thermal equilibrium for 15 minutes, according to EN ISO 3104.

Water content

The water content was determined with 0.2 g of biodiesel in the Karl Fischer titrator, Metrohm Coulometric Model, according to ASTM D6304.

Accelerated Oxidative Stability

The oxidative stability tests were carried out in Rancimat equipment, Metrohm, Model 843, following EN 14112 standard. A sample of 3.0 g was used, with a temperature of 110 °C and airflow of 10 L/min. The experiments were performed in triplicate.

Heating Value

The Higher Heating Value (HHV) and Lower Heating Value (LHV) were estimated through the mass composition of soybean biodiesel as a function of the percentages of carbon, hydrogen, and oxygen. The values obtained in the literature were used to determine the exergy.

Exergy Efficiency Analysis

The determination of the total exergy of the process was calculated as proposed by Kotas [40], where the total exergy of the process (e_{Total}) is obtained by the sum of the potential (e_P), kinetic (e_K), physical (e_F) and chemistry of biomass compounds (e_{CH}), according to equation (4).

$$e_{Total} = e_P + e_K + e_F + e_{CH} \quad (4)$$

where e_{Total} is the total exergy of the process (kJ/kg), e_P is the potential exergy (kJ/kg), e_P is the kinetic exergy (kJ/kg), e_F is the physical exergy (kJ/kg), and e_{CH} is the chemical exergy (kJ/kg).

The chemical exergy (e_{CH}) of the biomass compounds present in the soybean oil and biodiesel was determined by equation (5), being the multiply between the Lower Heating Value (LHV) and the coefficient β, obtained from the mass ratios of H, C, and O in the equations (6) and (7), for liquid biomass, such as biodiesel, glycerin, free fatty acids and triglycerides [41,42].

$$e_{CH} = \beta * LHV \quad (5)$$

$$LHV = HHV - 0.0894 * 2442.3 * H \quad (6)$$

$$\beta = 1.0374 + 0.0159 * \frac{H}{C} + 0.0567 * \frac{O}{C} \quad (7)$$

where β is the coefficient, LHV is the Lower Heating Value (kJ/kg), HHV is the Higher Heating Value (kJ/kg), and C, H, and O represents carbon, hydrogen, and oxygen, respectively.

Finally, the exergy efficiency of the process is obtained by the ratio between the output exergy (e_{out}) by the input exergy (e_{in}), according to Arredondo [42] and Mamede, et al. [39].

$$\mu_{ex} = \frac{e_{out}}{e_{in}} x100 \quad (8)$$

where is the exergy efficiency, e_{out} is the output exergy (kJ), and e_{in} is the input exergy (kJ).

Results and Discussion

The values of density, kinematic viscosity, water content, and oxidative stability obtained experimentally were compared with the standards established by the ANP in Resolutions nº 45 of 08/25/2014 and nº 798 of 08/01/2019, as shown in Table 1.

Parameter	ANP	This study
Density at 20°C (kg/m^3)	850-900	882.0 ± 0.6
Kinematic Viscosity at 40°C (mm^2/s)	3.0 - 6.0	4.3
Acidity number (mg KOH/g)	≤ 0.50	0.14
Water content, max. (mg/kg)	≤ 200.0	259.0±20.3
Oxidation stability (h)	≥ 12.0	3.70 ± 0.06

Table 1: Results for the soybean biodiesel characterization.

The low time of oxidative stability observed is consistent with the values found in several studies Silva de Sousa, et al. [43-48], and the use of antioxidants should be employed to prevent biodiesel degradation, meeting the requirements established by the ANP.

The water content present in biodiesel did not meet the limit established by the standard, however, the resolution establishes a tolerance limit of up to + 50 mg/kg for the producer and +150 mg/kg for the distributor.

The Higher Heating Value (HHV) was obtained using data available in the literature Huang J, et al. [49-57], shown in, from which the average value was obtained to Table 2 to determine the Lower Heating Value (LHV).

Soybean Oil	HHV (kJ/kg)	Soybean Biodiesel	HHV (kJ/kg)
Huang, et al. [49]	39870	Zheng, et al. [53]	39520
Elkelawy, et al. [50]	37600	Kratzeisen, et al. [54]	39685
Efe, et al. [51]	39530	Çelikten, et al. [55]	37620
Vellaiyan, et al. [58]	37900	Bhandari, et al. [56]	37100
Hou, et al. [52]	37000	Li, et al. [57]	39700
Average	38380	Average	38725

Table 2: Higher Heating Value (HHV) of the Soybean Oil and Soybean Biodiesel [49-58].

The mass percentage compositions of soybean oil and soybean biodiesel were obtained using data available in the literature Mamede AA, et al. [39,59-65], with the average value of the C, H, and O contents being determined Table 3.

Soybean oil	Percentage (%)			Soybean Biodiesel	Percentage (%)		
	C	H	O		C	H	O
[39]	77.83	11.5	10.67	[39]	79.3	13.2	7.68
[59]	76.71	10.64	12.65	[63]	80.94	11.44	7.49
[60]	76.64	10.95	12.11	[64]	76.05	12.74	10.76
[61]	76.2	11.6	10.4	[65]	82.22	11.96	5.74
[62]	77.15	11.89	10.96	[66]	76.96	11.85	9.41
Average	76.91	11.32	11.36	Average	79.09	12.24	8.22

Table 3: Mass percentage of C, H, and O for the soybean oil and soybean biodiesel.

The LHV is obtained from the mass composition data and the HHV of each matrix, being used in Equation 6. The β coefficient for liquid biomass substances is calculated by equation (7) with the average C, H, and O percentages obtained in Table 4.

Samples	HHV (kJ/kg)	LHV (kJ/kg)	H/C	O/C	β	e_{CH} (kJ/kg)
Soybean oil	38,380	35,909	0.1471	0.1477	1.0481	37,637
Soybean biodiesel	38,725	36,053	0.1547	0.1039	1.0457	37,702

Table 4: Results for the soybean oil and soybean biodiesel

Exergy analysis of biodiesel production is performed using exergy efficiency, which is the ratio between energy output and energy input, according to Equation 8. The input exergy (e_{in}) is obtained by the sum of the chemical exergy of the reagents (soybean oil, methanol, and potassium hydroxide (Table 5)) and the exergy of the equipment (Table 6), with the output exergy (e_{out}) being the sum of product exergy (biodiesel and glycerol (Table 7)). The electrical energy coefficient is considered 1 so that 1 kJ of electrical energy corresponds to an exergy flow of 1 kJ [39,42].

Reagents	e_{CH} (kJ/kg)	Quantity (g)	Exergy of the reagents (kJ)
Soybean oil	37,637	200,117	7,531.80
Methanol (CH_3 OH) [67]	22,420	40.02	897.2
Potassium hydroxide (KOH) [38]	1911	1,474	2.8
Total	34,898.50	241,611	8,431.90

Table 5: Exergy of the reagents.

Equipment	Consumption (W)	time (s)	Exergy (kJ)
Magnetic stirrer with heating – preheating	650	900	585
Magnetic stirrer with heating – transesterification	650	3,600	2,340
Magnetic stirrer with heating – drying	650	1,800	1,170
Total	650	6,300	4,095

Table 6: Exergy of the equipment.

Products	e_{CH} (kJ/kg)	Quantity (g)	Exergy of the products (kJ)
Soybean biodiesel	37,671	196.027	7,384.50
Glycerol [38]	22,953	24,385	559.7
Total	36,042.70	220,412	7,944.20

Table 7: Exergy of the products.

For the production of biodiesel, the exergy input was 12,527 kJ with an output of 7,944 kJ, resulting in a consumption of 4,583 kJ. The exergetic efficiency of the process is 63.42%, higher than that presented by Caliskan, et al. [68], close to that obtained by Silva, et al. [69] and a little lower than that obtained by Mamede, et al. [39]. In this way, one can observe the importance of studying the exergy efficiency for optimizing the production process and studying economic viability.

Conclusion

The production of soybean biodiesel was carried out in a batch reactor, on a laboratory scale, with an exergy efficiency of 63.42%. The difference between input and output exergy can be attributed to process consumption due to heat losses, non-reactive residues from reactions, and the irreversibility of the systems. According to the work, the importance of exergy analysis can be observed to evaluate the feasibility of applying biodiesel production, which allows identifying and discussing the aspects that cause the loss of useful energy, allowing the parameters to be optimized such as temperature, agitation, time and stoichiometric ratio seeking to improve the use of energy. The findings of this work provide crucial information to researchers, decision-makers, and technical operators of the biodiesel industry.

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