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Use of sugarcane bagasse ashes from the sugar and alcohol industry in the biodiesel production process from waste oil

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ABSTRACT
The ashes of sugarcane bagasse from the sugar and alcohol industry are considered an abundant and underutilized by-product. Its use to reduce acidity levels of residual oils in the biodiesel production chain can be a viable alternative. The ashes used in this work were donated by an industry in the region of Uberaba (MG). The material obtained, after being submitted to pre-treatment, was added to the residual oil in different amounts and different temperature and agitation conditions. The results were analyzed using an experimental design, with the aim of determining the best reaction parameters for biodiesel production, taking into account the specification of significant variables and their interactions. The homogeneous production of biodiesel occurred from the use of potassium hydroxide (KOH). For the characterization of the fuel, the reaction parameters were evaluated: acidity (AI) and iodine index, density and relative viscosity. The best ash pre-treatment conditions were the use of 3.6% of adsorbent mass in relation to AI, reaction time interval of 1.6 hours and 1 rpm of agitation. The reduction of the AI obtained in the treated residual oil allowed the evaluation of ash as an effective adsorbent. The characteristics of the final product were within the standards for the production of biodiesel.

Keywords: Adsorption, biodiesel, biofuel, biomass, cane ash, adsorbent materials, alternative methods, residue.

1 INTRODUCTION

Since the Industrial Revolution, the use of fossil fuels has become essential in industrial and transportation activities. The increased use of these fuels leads to environmental pollution caused by the accumulation of harmful atmospheric waste and arising from the process of separation of petroleum fractions, which generates greenhouse gases.

Currently, Brazil occupies the fourth position in the ranking of emission of polluting gases (BBC News Brasil, 2021). According to the Organization for Economic Cooperation and Development (OECD) and the International Energy Agency (IEA), government investment in oil between 2020 and 2021 nearly doubled. According to the EPBR agency, the reduction of the ICMS of fuels in 2022 caused an increase in gasoline consumption and, consequently, in carbon dioxide (CO_2) emissions, which reached 27.3 million tons. In this context, biodiesel stands out favorably among other energy

sources, because it is renewable (FERREIRA et al., 2020). Biodiesel is the generic name assigned to fuels and additives from renewable sources.

Compared to petroleum-derived diesel oil, it can reduce CO_2 emissions by 78% (ABREU et al., 2021). According to Maier et al. (2022), the explanation is due to the fact that the presence of CO_2 in the chain of production and consumption of renewable fuels, that is, its absorption and disposal in the atmosphere occur proportionally.

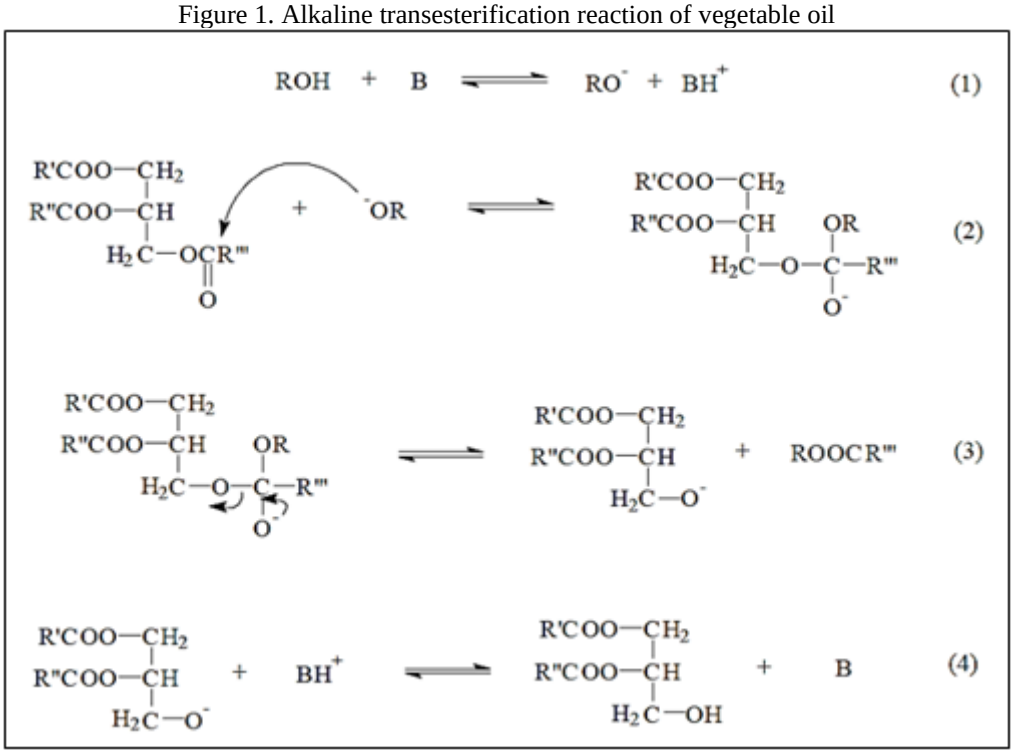
The most widely used method to obtain biodiesel is homogeneous transesterification, in which there is interaction of a short-chain alcohol with the triacylglycerides (TAG) present in vegetable oils or animal fats.

Homogeneous transesterification is a reversible process that uses an alkaline catalyst to enable the generation of biodiesel and glycerol. In general, alcohol is added in excess to shift the balance in the direction of the products and ensure a higher yield of the process.

The excess added alcohol is removed in the distillation step and can be reused after dehydration (VIEIRA, et al., 2017). Figure 1 shows the alkaline transesterification reaction in stages.
According to Souza (2022), transesterification by alkaline homogeneous catalysis is preferred because it presents advantageous characteristics, such as having a good reaction yield at room temperature, being kinetically favorable and presenting a lower risk of corrosion by the catalyst.

However, some reaction limitations are observed, since the raw material must have low acidity and humidity.

Figure 1. Alkaline transesterification reaction of vegetable oil



Source: Bortoletto, 2020

In this process, the main raw materials used for the national production of biodiesel are: soybean, corn, sunflower, peanuts, cotton, canola, castor bean, babassu, palm (oil palm) and macaúba, among other oilseeds existing in the country, which are used as a source of food in daily life (BARROS, T. D. et al, 2021).

According to Duarte et al. (2022), the use of raw materials from food in the production of second-generation biodiesel results in an increase in the cost of food, since there is a reduction in the amount available for food.

Thus, the use of alternative raw materials, such as residual oil, is attractive and can be the target of research and applications.

The transesterification of frying oils in the production of biodiesel is advantageous from an economic, environmental and social point of view, and this type of raw material has a lower cost than virgin oils, in addition to enabling the recycling of large volumes of residual oils and offering greater energy security, without harming the fraction available to the food chain (BONASSA, 2017).

However, the major disadvantage of using raw materials with high levels of free fatty acids and without a previous treatment, is the occurrence of saponification reactions, which lead to a considerable reduction in reaction yield, making the process unfeasible (LIMA, 2020).

Thus, it is necessary to perform treatments, such as evaporation by drag (MARINS et al., 2015), neutralization and esterification (ROVERE et al., 2020), capable of transforming it into a reagent suitable for the process.

Adsorption is an alternative that has been shown to be efficient in the treatment of industrial solid waste. It is a physicochemical phenomenon where the component of a gas or liquid phase is transferred to a solid phase surface.

The phase whose interface will adsorb the product is called adsorbent or substrate and adsorbate corresponds to the phase that will be adsorbed. Although this process involves phases resulting from solid-liquid, solid-gas, liquid-gas and liquid-liquid systems, most studies focus on the first two systems (NASCIMENTO et al., 2018).

There are several economically viable materials that can be used in the adsorption process, such as activated carbon, silica, clay, green banana peel, sugarcane bagasse, cob and corn and coconut mesocarp (OLIVEIRA et al., 2022), among others.

The ashes from sugarcane bagasse contain about 60% silicon dioxide (SiO_2) in its composition. The predominance of this oxide is responsible for the structural arrangement of alkaline and porous particles, and evidences its applicability in acidic materials, and can then act as an adsorbent material.

Changes may occur in the chemical composition of sugarcane bagasse ash, according to the conditions it undergoes. The ash composition of sugarcane bagasse produced at 600ºC is shown in Figure 2.

Figure 2 – Composition of bagasse ash produced at 600ºC

Compound	Content (% by mass)
SiO2	60,96
Al2O3	0,09
Fe2O3	0,09
Tall	5,97
Na2O	0,70
K2O	9,02
Too	0,48
MgO	8,65
P2O5	8,34
Residual carbon mass	5,70

Source: Química Nova, 2009

Brazil is the world's largest producer of sugarcane, whose 2021/22 harvest totaled 585.2 million tons, according to the National Supply Company (CONAB).

Due to the exorbitant production, the residues arising from the sugar and ethanol production processes are cumulative and have a high potential for environmental degradation.

An estimate made by EMPRAPA (2022) reported that for each ton of sugarcane processed, 280 kg of bagasse are generated. The ash is obtained at the end of the production process, in a ratio of 25 kg of ash for each ton of sugarcane processed, approximately.

In order to provide a destination for this cumulative, easily accessible and low-cost waste and to reuse residual oils and fats, this work aimed to reduce the IA of residual oils through the application of sugarcane bagasse ash, from sugarcane industries in the Triângulo Mineiro region, and to enable the process of biodiesel production by homogeneous route.

2 METHODOLOGIES

A Central Composite Planning (PCC) was constructed with three independent variables using the software STATISTICA ® 10 - license of the Professional Master's Program in Technological Innovation of UFTM (PMPIT/UFTM). The time interval, rotation speed and adsorbent mass were the parameters selected for the study (Table 1).

Table 1 – Central Composite Planning (CCP)

	x1	x2	x3	T(h)	MB (%m/m)	VA (rpm)	mg/g oil
1	-100.000	-100.000	-100.000	1.6	3.6	1	6.56
2	-100.000	100.000	-100.000	1.6	6.4	1	6.87
3	100.000	-100.000	-100.000	4.4	3.6	1	7.46
4	100.000	100.000	-100.000	4.4	6.4	1	6.77
5	-100.000	-100.000	100.000	1.6	3.6	5	7.97
6	-100.000	100.000	100.000	1.6	6.4	5	8.15
7	100.000	-100.000	100.000	4.4	3.6	5	10.44
8	100.000	100.000	100.000	4.4	6.4	5	14.04
9	0.00000	0.00000	-141.421	3	5	0	6.74
10	0.00000	0.00000	141.421	3	5	6	7.38
11	-141.421	0.00000	0.00000	1	5	3	6.85
12	141.421	0.00000	0.00000	5	5	3	7.10

13	0.00000	-141.421	0.00000	3	3	3	6.85
14	0.00000	141.421	0.00000	3	7	3	7.51
15	0.00000	0.00000	0.00000	3	5	3	7.69
16	0.00000	0.00000	0.00000	3	5	3	6.99
17	0.00000	0.00000	0.00000	3	5	3	7.42
18	0.00000	0.00000	0.00000	3	5	3	7.42

Source: From the Authors, 2023

The ashes of sugarcane bagasse were donated by sugar-alcohol industries in the Triângulo Mineiro region.

They were exposed to the sun for 3 days in order to remove excess moisture. Submitted to pre-treatment, the ashes were mixed with 5 g of potassium hydroxide (KOH) 1:0.25 [m cane / mKOH], in a mortar.

The mixture was sent to the oven at 110ºC for a period of 48 hours. Then, pyrolysis was performed in a muffle furnace, with a temperature ramp at 5ºC/min, until reaching 450º C, remaining at this temperature for 1 hour.

The 0.10 mol L-1 HCl solution and distilled water were used to wash the residual material until the neutral pH was reached. The residue was dried in an oven at 105ºC for 24 hours (BAVARESCO, A. 2017)

The determination of the IA was performed by applying the official method Cd 3d-63 applied to the analysis of Acidity Value of Oils and Fats (American Oil Chemist's Society).

In an erlemneyer, the mass of 1 g of the oil sample was weighed and 5 mL of ethyl alcohol, 5 ml of ethyl ether and 3 drops of the phenolphthalein indicator were added.

The calculation of the AI was performed according to Equation 1. The AI reduction tests were based on the PCC, in order to identify the best experimental condition (COSTA FILHO, E. H. 2008).

$$IA = \frac{V * C_{NaOH} * Mm}{m} \quad (1)$$

Where:
AI = acidity index (mg NaOH/g oil);

V = volume of NaOH of the titration (mL);

CNaOH = Concentration of the indicator solution (mol L-1);

M = molar mass of NaOH (mol L-1);

m = mass of oil (g).

Methanol was the reagent used in the synthesis of biodiesel, from alkaline transesterification, whose procedure was performed considering the residual oil sample, which was treated with the ash of sugarcane bagasse, and the virgin oil sample.

In a beaker was added the mass of 1.5 g KOH in 35 g of methanol. Then, in an erlenmeyer, the solution was incorporated into 100 g of oil and kept in constant agitation at 50º C for 20 minutes.

After this time interval, the mixture was transferred to a separation funnel. This procedure was performed considering the three sampling conditions.

To evaluate the quality of the synthesized biodiesel, its characterization was performed. The first method consisted of the evaluation of the IA through the official method Cd 3d-63 for the analysis of acidity value of oils and fats (American Oil Chemist's Society) (OLIVEIRA, K. 2022).

Pycnometry was the technique used to perform the final product density test according to the methodology described by Neto et al. (2018).

To perform the Iodine Index test, the mass of 0.25 g of sample was weighed in different erlenmeyers.

The volume of 10 mL of chloroform and 10 mL of the WIJS solution (LEONARDI, J. G. et al. 2018) were added to the samples, whose vials were kept under constant agitation, until the homogenization of the solution.

Then, the system was left at rest for 30 minutes and without the presence of light. After this time interval, 5 mL of 15% (m/v) KI (potassium iodide) solution were added under agitation until complete homogenization of the solution.

By adding 100 mL of distilled water, the mixture was titrated with 0.10% (m/v) $Na_2S_2O_3$ (sodium thiosulfate) solution until the yellowish color disappeared. Subsequently, 2 mL of the 1% starch indicator solution were added, and a new titration was performed until the purple color dissipated.

The Relative Viscosity test was performed based on its flow time (RINALDI, R. et al. 2007). A 10 mL graduated pipette was first filled with distilled water and the flow time was measured in duplicate.

Subsequently, the same process was done with the biodiesel samples. Next, the ratio between the time intervals of flow of the samples in relation to the average time of water flow was calculated.

3 RESULTS AND DISCUSSION

REDUCTION OF THE ACIDITY INDEX (IA)

From the PCC, it was identified that condition 1 resulted in a greater decrease in the AI of the residual oil, by the application of 3.6% of the mass of residue treated for 1.6 hours and with a stirring

speed of 1 rpm. As shown in Table 2, Analysis of Variance (ANOVA) for all significant process variables was performed.

It was contacted that AI had influence of the parameters time and speed, when applied together or separately.

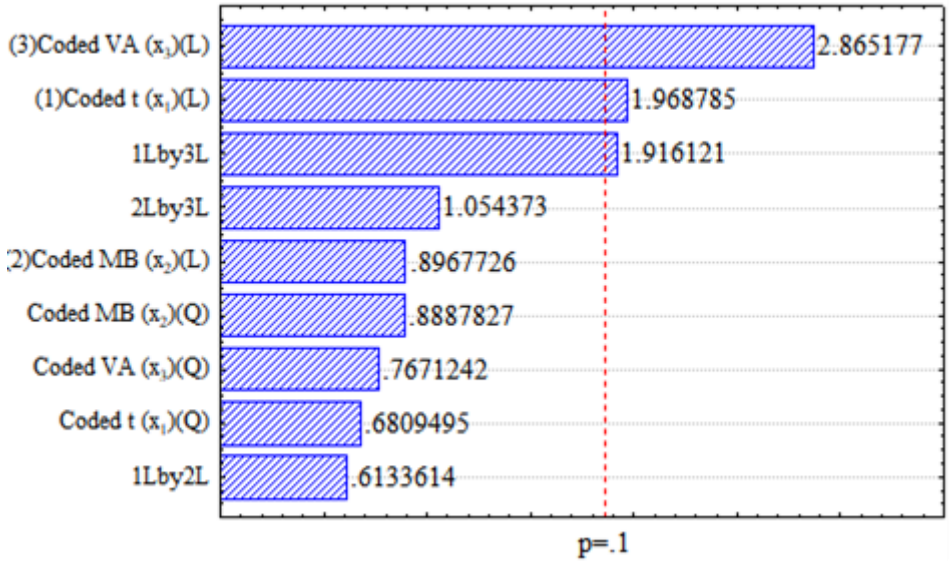
Table 2 – Analysis of Variance of the IA reduction of sugarcane bagasse ash

Variable	SQ	GL	MQ	F-value	Prob. > F	Significance (90% confidence)
Xt	**7.54231**	1	**7.54231**	3.876113	0.084502	Significant
xT2	**0.90227**	1	**0.90227**	0.463692	0.515126	Not significant
xMB	**1.56485**	1	**1.56485**	0.804201	0.396024	Not significant
xMB2	1.53709	1	1.53709	0.789935	0.400054	Not significant
xVA	**15.97389**	1	**15.97389**	8.209239	0.020984	Significant
xVA2	1.14509	1	1.14509	0.588480	0.465042	Not significant
xTxMB	0.73205	1	0.73205	0.376212	0.556670	Not significant
xTxVA	7.14420	1	7.14420	3.671519	0.0916662	Significant

Source: Statistics, 2022

A Pareto chart was elaborated to evaluate the influence of the residue mass, the time interval and the agitation speed on the reduction of the IA with regard to the reduction of the IA (Figure 2). It was observed that the agitation speed and the time interval presented a statistically significant effect, with a confidence level of 90%.

Figure 2 – Pareto graph for the reduction of the acidity index from the ashes of sugarcane bagasse



Source: Statistica, 2022.

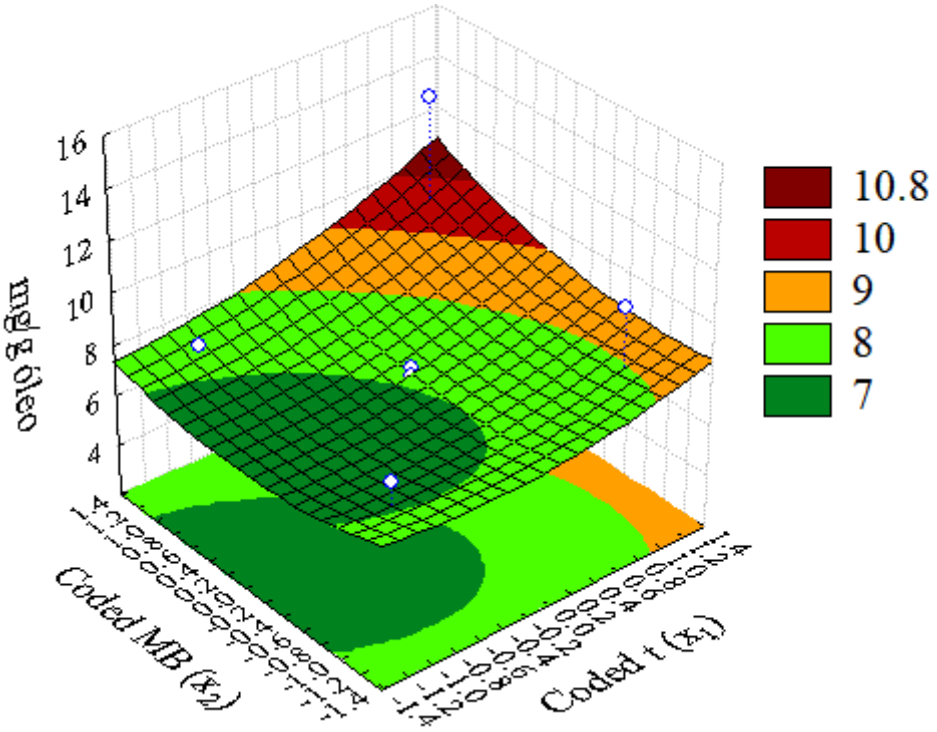
Subsequently, the response surface graphs were elaborated (Figure 3), which allowed to evaluate the joint influence of the adsorbent mass and the time interval (a); the agitation speed and the time interval (b); the agitation speed and the adsorbent mass (c), for the reduction of the IA.

In this study it was possible to notice a higher efficiency for the minimum conditions of agitation speed and adsorbent mass, inferring that shorter time intervals induce a better reduction of

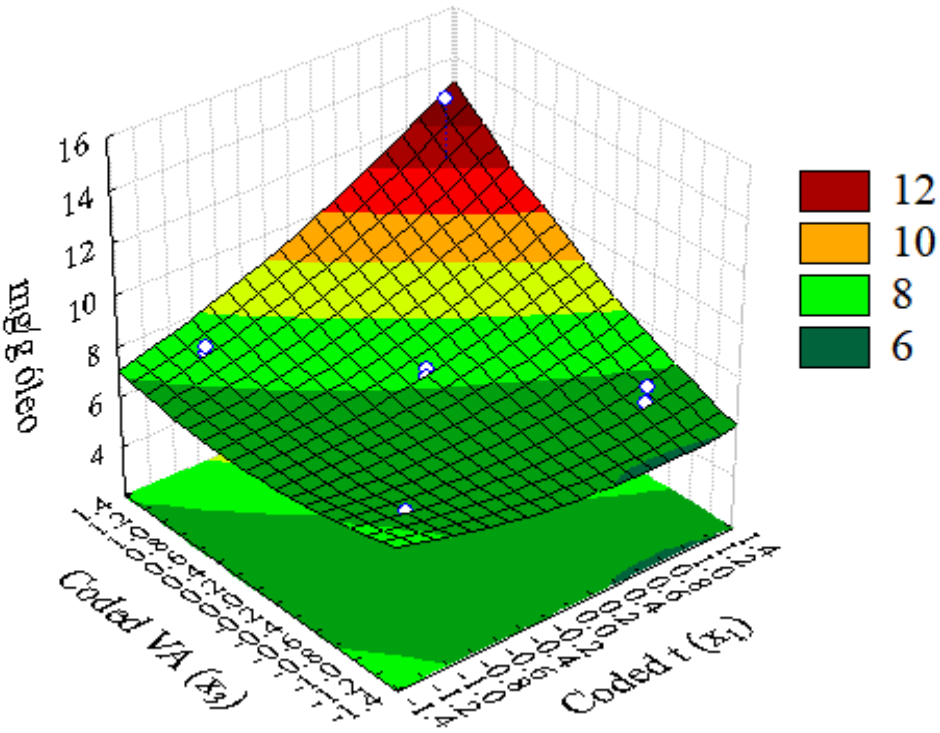
fatty acid concentrations. This statement can be substantiated by condition 1, whose values provided greater reductions in AI.

Figure 3 – Response surface of the influence of adsorbent mass, agitation speed and time

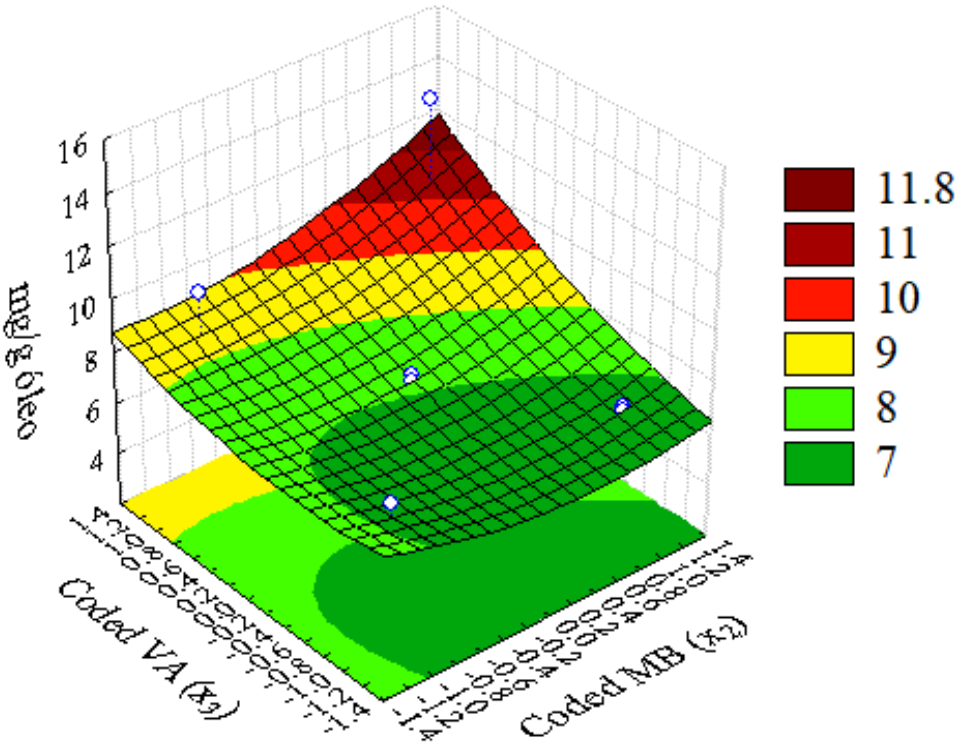
(A)



(B)



(C)



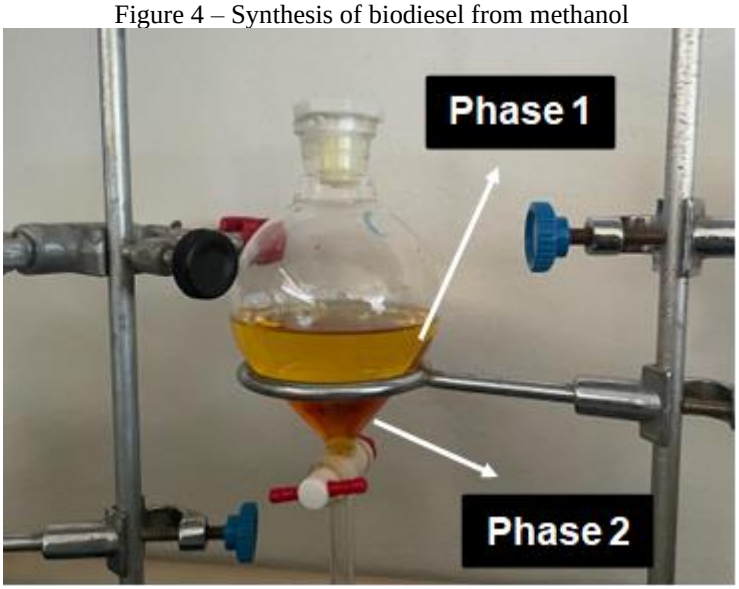
Source: STATISTICA, 2022

4 BIODIESEL SYNTHESIS

The methodology applied for the synthesis of biodiesel presented satisfactory results, and it was possible to observe the occurrence of two phases inside the separation funnel (Figure 4).

The use of the methyl route allowed the quantification of the final volume and the performance of the biofuel quality tests. As a comparison, the synthesis of biodiesel was also made from samples of virgin soybean oil. The results of the characterization and quality tests are shown in Table 3.

Figure 4 – Synthesis of biodiesel from methanol



Source: From the Authors, 2023

Table 3 – Characterization of biodiesel

	Sample with treatment	Virgin oil
Final volume (mL)	75	115
Acidity Index (AI)	0,405	0,059
Relative density (g/cm³)	0,875	0,829
Iodine Index	48,945	47,739
Relative viscosity (g/cm*s)	1,383	1,172

Source: From the Authors, 2023

The lower volume of biodiesel from the residual oil, in relation to the biofuel produced by the virgin oil, can be justified due to the amount of free fatty acids, which provided a loss of efficiency of the process. In addition, there was retention of a portion of the oil sample in the pre-treatment stage, which slightly influenced the final volume of the product.

5 CHARACTERIZATIONS OF BIODIESEL

The IA of the treated residual oil corresponded to 4046 mg KOH/g. This value is in accordance with method 14448, which establishes a maximum limit of 0.5 mg KOH/g (ANP Nº 798, 2019).

Thus, the efficacy of the treatment with the ash of sugarcane bagasse was verified. According to Oliveira et al. (2022), it was verified that the ashes of sugarcane bagasse promoted a reduction of 52% of the acidity of the residual oil in less time than the activated carbon (49%), which proved its efficiency as an adsorbent, analogous to what was presented in this work.

Some physicochemical properties of biodiesel taken from ANP 798 (National Agency of Petroleum, Natural Gas and Biofuel, 2019) can be observed in Table 4.

Regarding density, the biodiesel produced from the residual oil treated with sugarcane ash presented results consistent with the established limits, whose value was 0.875 g/cm³.

For confirmation, a recent study estimated the specific masses of poultry oil at 0.87 g/cm³, which was stated to be a typical value for methyl biodies, because the property does not depend on the nature of the raw material (SILVA, C. et al., p. 166, 2022).

Table 4 – Physicochemical properties of biodiesel

CHARACTERISTIC	UNIT	BOUND	METHOD
			ABNT NBR
Aspect	-	LII (1) (2)	-
Specific mass at 20°C	kg/m³	850 a 900	7148 14065
Acid number, max.	mg KOH/g	0,50	14448 -
Iodine Index	g/100g	Jot	-
Oxidation stability at 110ºC, min. (11) **(Text given by ANP Resolution No. 798 OF 08/01/2019).**	Hour	12	

Source: ANP 798, 2019

The Iodine Index evaluates the presence of total unsaturations of the sample and, consequently, the susceptibility to oxidation.

However, according to the technical note SEI/ANP 1607120, the method neglects the positions of the unsaturated, not being able to measure the real stage of degradation of the biodiesel produced.

Thus, there is no maximum value stipulated by the ANP for this condition and, despite the similarity between the results obtained in this study, this provision was not seen as a problem.

The relative viscosity was evaluated from the ratio of the average flow velocity of each sample in relation to that of the water (RINALDI, R. et al., 2007). Table 5 presents the relative viscosity values found for each condition.

Table 5 – Result obtained from the analysis of the relative viscosity of the biodiesel samples

	Average flow time(s)	**Relative Viscosity (g/cm*s)**
Water	11,76	1
Sample with treatment	14,53	1,3827
Sample of virgin oil	14,96	1,1719

Source: From the Author, 2023

In its evaluation, as expected, it was noticed that the sample from the virgin oil flows more quickly when compared to the sample of treated residual oil, however, the value still remained close to one.

The work of RINALDI, R. et al. (2007) found a relative viscosity of 1.5 [$g.cm^{-1}.s^{-1}$] for biodiesel obtained through the transesterification of soybean oil with methanol, which is a value similar to that found in this work.

Finally, it was observed that the flow time had a similar behavior in both conditions, its slight variation can be justified by the presence of mono-diglycerides present in the final product.

6 CONCLUSIONS

Sugarcane bagasse ash has basic active sites capable of adsorbing fatty acids in solution. In this study, the ash exerted its function as an adsorbent effectively, reducing the AI of the residual oil.

The samples showed the influence of the agitation speed and the reaction time interval, when applied together and in isolation.

The minimum agitation conditions and time intervals provided favorable results in the reduction of AI.

When performing the homogeneous synthesis of biodiesel, the sample submitted to pre-treatment with the ashes of sugarcane bagasse, as well as that of virgin soybean oil, showed results in agreement with the standards required by the ANP.

From the quality tests, it was confirmed that the ashes from sugarcane bagasse were essential to produce an appropriate biodiesel and within the quality standards.

The application of sugarcane bagasse ash as adsorbent material for the treatment of residual oils demonstrated a profile of sustainability and reduction of production costs.

This process can be a viable alternative for the production of biodiesel.

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