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Biodiesel Production Using Residual Vegetable Oil and Activated by Geopolymer Matrixes with Magnetic Particles

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Abstract: The cooking oil, when reused in frying, undergoes a thermal degradation process that changes its physical and chemical characteristics. After repeated use of the oil, it becomes viscous and increasingly dark, it has high acidity and unpleasant odor, being inconvenient to use for new fried food because it gives unpleasant odor and taste to food and also harmful chemical characteristics to health. When these residues become unusable usually they are dumped into the sewage system and have a negative environmental impact, for example, in rainwater and sanitary sewage systems the oil mixes with the organic matter and obstructs grease boxes and pipes. Therefore, the recycling of residual vegetable oil is necessary and very useful, because it transforms the oil for other applications, for example, in this work the use of porous geopolymer with magnetic particles in the concentrations of 1%, 2%, and 3% were studied for act on the activation of the biodiesel reaction from the used cooking oil. The geopolymers with and without magnetic particles were studied using Fourrier Transform Infrared (FT-IR) and X-Ray Diffraction (XRD). The density, the kinematic viscosity, and yield of the biodiesel formation reaction were investigated. The results, calculated using analysis of variance (ANOVA) with a 95% confidence limit, indicate that all the biodiesel samples analyzed are in accordance with the kinematic viscosity value established by the Agência Nacional do Petróleo, Gás Natural e Biocombustíveis (ANP) but only samples with 0% and 1% magnetic particles have density according to the stipulated parameters.

Keywords: Residual vegetable oil, geopolymer, magnetite, biodiesel, recycling, cooking oil, magnetic particles, biofuel.

Adherence to the BJEDIS' scope: This work is related to the scope of BJEDIS as it presents statistical methods combined with analytical methods.

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1. INTRODUCTION

The inappropriate disposal of used oil causes serious environmental problems (1, 2); therefore, its transformation into biodiesel, through activation by geopolymers with magnetic particles, becomes an important recycling alternative.

The high demand for the consumption of fried foods aggravates the problems related to the disposal of residual vegetable oil, considering that its reuse becomes improper due to the chemical and physical changes suffered from the reheating. Among the many alterations, browning, high acidity, increase in viscosity, development of unpleasant odor and chemical changes such as hydrolysis and polymerization stand out (1, 3).

When vegetable oil is discharged into sewers, it can obstruct grease boxes and pipes. This oil, when it reaches rivers and oceans, forms a heterogeneous mixture with water that impairs underwater life and the availability of clean water (3). Only one liter of oil can contaminate approximately twenty thousand liters of water (4). When discarded in the soil, it makes it difficult for water to penetrate and releases methane gas as a product of its decomposition (5).

It is possible to observe that there is no consensus of experts on the ideal disposal of residual vegetable oil. So far, the most appropriate measure is the correct collection for recycling the waste to obtain resins used for the manufacture of paints and varnishes, detergent, soap, glycerin, engine lubricant, handmade candles, and biodiesel (3, 5–7). Biodiesel is non-toxic, renewable and, in addition to resolving the problem related to the disposal of residual oil, its use can reduce the high emissions from the burning of fossil fuel necessary to supply the current energy demand (8, 9).

Biodiesel consists of a mixture of straight-chain alkyl esters, obtained by transesterification of oil and fat triglycerides with the addition of short-chain alcohols, such as methanol and ethanol, and catalysts that can be homogeneous or heterogeneous (10–12).

Methyl transesterification with the use of basic homogeneous catalysts is currently the predominant process in the industry for obtaining biodiesel. Despite its disadvantages, such as the production of soaps due to the neutralization of nonesterified fatty acid or saponification of esters, in addition to the difficulty of purifying the final product (13, 14). Such factors indicate that it is essential to develop research to obtain more effective and efficient catalysts in the production of biodiesel. In this work, for example, the geopolymer with magnetite is evaluated as a catalyst in the production of biodiesel

In the 70 s, Joseph Davidovits applied the term “geopolymer” for the first time, referring to inorganic polymers identical to ceramics that are made up of chains of mineral molecules united by covalent bonds. Geopolymers can be synthesized from industrialized compounds, such as metakaolin, or by-products of industrial processes. Thus, geopolymers are an economically viable alternative for work (15–18).

The use of geopolymer with magnetic particles as a heterogeneous catalyst for the reaction to obtain biodiesel search advantages over homogeneous catalysts, such as the possibility of reusing the catalyst, facilitating the purification of biodiesel through a simple filtration procedure, thus amortizing the generation of effluents and reducing process costs (10, 14).

Biodiesel characteristics must be analyzed using analytical methods. Some methods of physical-chemical analysis of biodiesel are classic for the analysis of mineral diesel. The others are analytical methods for the determination of the quality of oils and fats. Frequent researches have been carried out in the search for reliable, fast, and low-cost analytical methods (10, 19).

To guarantee the reliability of the results, it is essential to use statistical methods combined with analytical methods. The analysis of variance (ANOVA) has proven very useful for experimental data analysis in chemistry (20, 21).

Research about the topic “Descarte inadequado de óleo residual no Brasil” (performed in March 2021 at the site https://scholar.google.com.br) presented approximately 3370 results in the set time interval from 2017 to 2021. This is an indication that there is a great concern about the destination of this residue.

In accordance with the proposal of the BJEDIS (Brazilian Journal of Experimental Design, Data Analysis and Inferential Statistics), the statistical analyses linked to the analytical methods allow inferring values of variance and confidence limit to determine if the biodiesel samples produced show significant differences in the analyzed variables (density, kinematic viscosity, and yield) when activated by geopolymeric composites.

2. MATERIALS AND METHODS

2.1. Preparation of Magnetic Particles

The magnetic particles were prepared from a 0.44 M iron salts solution and co-precipitated in dispersed silica solution in basic solution 4.33 M. Subsequently, the obtained magnetic particles were decanted with the aid of a magnet and the supernatant removed. The particles were washed three times with deionized water and dried in an oven at 50°C for two hours.

2.2. Synthesis of Geopolymers with and without Magnetic Particles

To carry out the synthesis of the geopolymer, a sodium silicate solution of molar ratio $SiO_2/Na_2O=1.6$ was prepared and the alkaline solution was stirred under 80 °C for five minutes. During stirring, metakaolin with molar ratio $Na_2O/Al_2O_3=1$ was added and, in the next step, the porosity agent 0.5% m/m H_2O_2 was added. Finally, the magnetite was added to obtain samples with 1%, 2%, and 3% magnetic particle content. A geopolymer sample without the addition of a magnetic particle was also synthesized. Stirring was continued for another ten minutes. The samples obtained were transferred to the oven to cure at 80 °C for two days.

2.3. Biodiesel Production

3.0 g of geopolymer was transferred to a beaker and 200.00mL of methanol was added. The beaker was heated to 45 °C. For the transesterification reaction, 160.00 mL of residual vegetable oil was used together with the geopolymer solution with methanol previously prepared. The reaction occurred in a flat bottom flask over two hours at a temperature of 45 °C. After the end of the transesterification reaction, the solution was left to stand for one hour in a separation funnel. As a result, the geopolymer was decanted and glycerin was subsequently separated and biodiesel was collected for characterization and comparison with the requirements established by the ANP (22).

2.4. Characterizations

2.4.1. X-Ray Diffraction

X-ray diffraction tests were performed on a Rigaku X-ray diffractometer, model Miniflex. The method used was FT, with an angular pitch of 0.05º, in the range of 2θ between 10° to 80°, with acquisition time at each point of 1.0 s.

2.4.2. X-Ray Fluorescence

The equipment used for the measurements was a PanAnalytical X-ray fluorescence spectrometer, model Axios Max with wavelength dispersion (WD) with rhodium tube at 4 kW, operated under standard routine conditions of the CETEM FRX laboratory, with voltage of 50 kV, current of 60 mA, goniometer step of 0.05 degrees 2θ and counting time of 0.25 s.

2.4.3. Magnetic Force

The tests were performed using an artisanal experimental setup. This configuration consists of a Shimadzu AY-220 analytical balance, an ICEL PS-4100 voltage source, an ICEL MD-6450 digital multimeter, a GlobalMag TLMP-Hall-02 gaussmeter, a handmade sample holder and an electromagnet. The system calibration was executed in the absence of magnetic material. Initially, using the ammeter and the gaussimeter, a current versus magnetic field calibration was performed. Subsequently, a current versus mass calibration was also performed. The results obtained were used to predict part of the error presented. The magnetic force tests were performed following the variation of the sample mass in the presence of the magnetic field produced by the electromagnet. Then, the apparent variation of the sample mass in the presence of the magnetic field was calculated by subtracting the sample mass in the presence of the magnetic field from the sample mass. The magnetic force (opposite to the gravitational force) was calculated with the aid of the equation:

$$Fmn = \frac{\Delta m \cdot g}{m_0} \quad (1)$$

Fmn is the magnetic force normalized by the initial mass of the sample, m0, Δm is the apparent variation in mass in the presence of the magnetic field and g is the acceleration of gravity.

The test was executed using triplicates to study the veracity of the results through statistical analysis.

2.4.4. Density

The density of biodiesel was measured in a 10.00 mL measuring cylinder. The volume that the biodiesel filled in the measuring cylinder was checked and the biodiesel sample was weighed. For the calculation the following equation was used:

$$d = \frac{m}{v} \tag{2}$$

d= density ($g{\cdot}mL^{-1}$)

m= fluid mass (g)

v= fluid volume (mL).

2.4.5. Kinematic Viscosity

The kinematic viscosity was calculated in centiStokes ($cm^2{\cdot}s^{-1}$). The biodiesel produced was added to a graduated burette and the height occupied by the fluid was measured, then the flow time (s) was investigated. Finally, the calculation was performed using the following equation:

$$cSt = \frac{cP}{d} \tag{3}$$

cSt= centiStokes ($cm^2{\cdot}s^{-1}$)

cP= centiPoise (P)

d= density ($g{\cdot}mL^{-1}$)

2.4.6. Yield

Calculations for biodiesel yield were performed with the support of the following equation:

$$\frac{vb * 100}{vt} \tag{4}$$

vb= volume of biodiesel formed

vt= total volume of reaction

3. RESULTS AND DISCUSSION

3.1. X-Ray Diffraction

The geopolymers were characterized using the X-Ray Diffraction technique in order to investigate a crystalline property of the material and the presence of the magnetic particle. The results obtained, shown in figure 1, showed that the peaks of the main components of the geopolymer formation were identified, these being the peak corresponding to sodalite ($Na_8(AlSiO_4)_6(NO_2)_2$) at angles of 17°, 29° , 37° and 41° and the peak corresponding to quartz (SiO_2) at an angle of 31°. It was also possible to identify the characteristic peak of maghemite at 35° (24, 25). These results indicate that there was the formation of the geopolymer and the obtaining of composites.

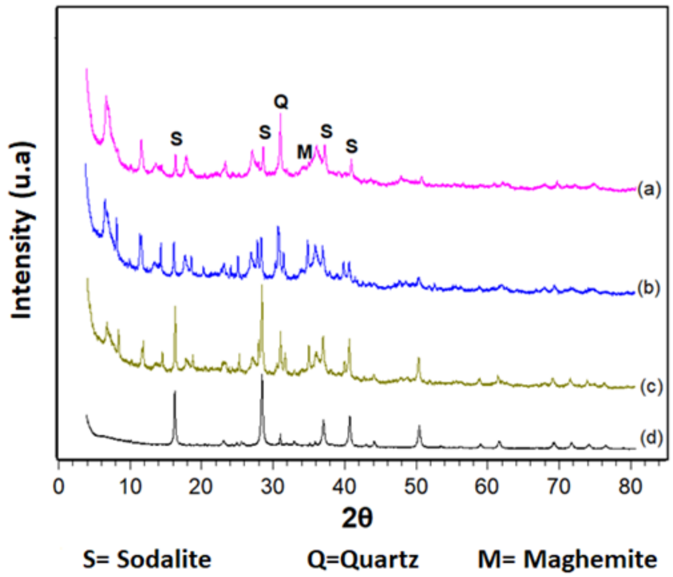

Figure 1. Diffractogram of porous geopolymer (a) 3% magnetic particles, (b) 2% magnetic particles, (c) 1% magnetic particles and (d) 0% magnetic particles.

3.2. X-Ray Fluorescence

X-Ray Fluorescence analysis was performed to verify the chemical composition of the geopolymers. The results obtained indicate that there was an increase in Na_2O due to the insertion of the activating agent NaOH in the matrix and the consumption of the reaction components Al_2O_3 and SiO_2 by the polymerization reaction. Also, it was possible to verify the increase of maghemite (Fe_2O_3) in the geopolymeric matrix (26), as can be seen in Table 1.

Table 1. Chemical composition of metakaolin and geopolymers with and without magnetic particles.

Materials	Chemical Composition				
	Na_2O	Al_2O_3	SiO_2	K_2O	Fe_2O_3
metakaolin	0.11	49.10	49.20	0.39	0.32
0 %	26.20	22.60	30.50	0.19	0.21
1%	28.00	21.30	29.50	0.16	0.35
2%	26.00	21.60	29.20	0.16	0.52
3%	26.90	21.70	29.80	0.17	0.69

3.3. Magnetic Force

Magnetic force analysis was carried out to examine the influence of the modification with silica on the magnetic properties of the particles and to verify the magnetic response of the composites. The results showed that the magnetite had a magnetic force of 2,804 ± 20 mN/g and the magnetite modified with silica 1,218 ± 43 mN/g, these values allow us to infer that the presence of silica in the particles decreased the magnetic properties by 43% (Figure 2 (b e c)). For composites, it was possible to observe that in 800G (Figure 2 (a)) as the amount of magnetic particle increased, the magnetic force also increased, with the values of 7.8± 0.3 mN/g for 1%, 11.1±0.1 mN/g for 2% and 12.3±1.4 mN/g for 3%. These results corroborate with the analysis of DR-X and FR-X, indicating the modification of composites with magnetic particles.

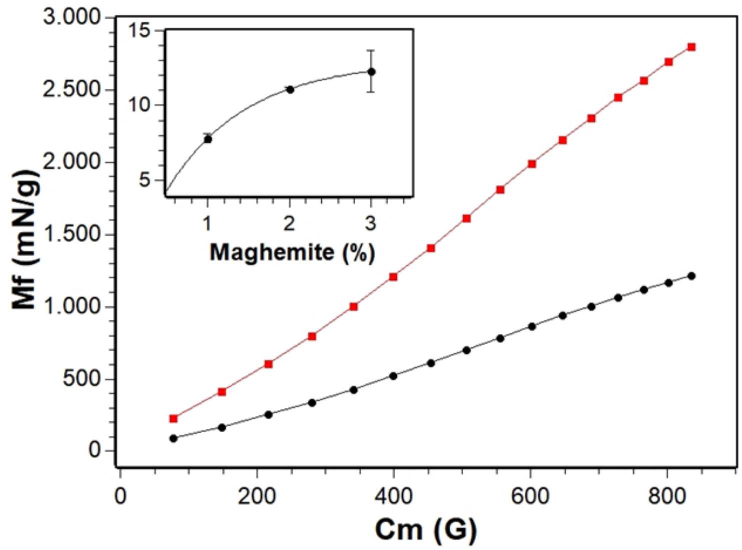

Figure 2. Magnetic force (a) geopolymer composites with 1%, 2% and 3% magnetic particles at 800G, (b) magnetite and (c) magnetite modified with silica.

3.4. Biodiesel Evaluation

After the production of the geopolymeric composites, it was followed by the production of biodiesel with the geopolymeric composite acting as an activator. Triplicate samples were used to perform the analysis and statistical calculations of density, kinematic viscosity, and yield through analysis of variance (ANOVA) with a 95% reliability limit, to evaluate the efficiency of the activation of the geopolymer in the conversion of oil biodiesel. The results can be found in table 2. The data obtained from the biodiesel analyzes were compared with the ANP parameters. According to the Agency, the density values for biodiesel should be in the range between 0.829 g/cm^3 and 0.834 g/cm^3, therefore, only the activated biodiesels with magnetite content of 0% and 1% are in the range according to the ANP. The presence of residues in biodiesel, such as ethanol, can influence the density value. Although the ASTM (American Society of Testing and Materials) standard does not consider density as a determining factor for assessing the quality of biodiesel (10). In terms of kinematic viscosity, according to the ANP, the values required for biodiesel should range from 2.3 cSt to 3.3 cSt. Based on this reference, all the biodiesel samples analyzed are under the parameters of kinematic viscosity estimated by the ANP (22).

Table 2. Values of density, kinematic viscosity, and yield of biodiesel activated with magnetic geopolymers.

Magnetite in geopolymer (%)	Density (g.cm^{-3})	Kinematic viscosity (cSt)	Yield (%) proportion 7:1
0	0.81±0.02	2.57±0.1	77.5±2
1	0.80±0.04	1.98±0.6	62.5±3
2	0.78±0.02	1.98±0.4	62.5±3
3	0.80±0.01	2.34±0.1	75.0±2

About the yield, the values obtained were compared with the result obtained by Meng and collaborators in 2008 (27), in which the proportion of methanol/oil was 7:1 and the conversion to biodiesel was 90%. In the present study, the yields were lower, which may be related to the poor dispersion of the geopolymer throughout the reaction, as it is dense and tends to decant, hindering the quality of the reaction.

4. CONCLUSIONS

From the results obtained, it was possible to conclude that the XRD and FRX analysis proved the formation of geopolymers and their chemical composition showed an increase in magnetic particles and polymerization. The magnetic force showed that there was a modification with silica in the magnetic particles, confirmed by the decrease in the magnetic force presented. The composites showed an increase in magnetic force with the increase of particles in the geopolymeric matrix. The activation of biodiesel using geopolymers with magnetic particles was efficient because all the biodiesel samples analyzed are following the parameters of kinematic viscosity estimated by ANP, however, only samples with 0% and 1% of magnetic particles have the density of according to the parameters stipulated by the ANP. The yield was low, so more studies are necessary to improve the dispersion of the geopolymer throughout the reaction.

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Sample CRediT author statement

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